Dairy Powders and Concentrated Products

EDITED BY

A. Y. TAMIME

SOCIETY OF DAIRY TECHNOLOGY

WILEY-BLACKWELL

SDT

Dairy Powders and Concentrated Products

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Ayr, UK

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The Society of Dairy Technology (SDT) has joined with Wiley-Blackwell to produce a series of technical dairy-related handbooks providing an invaluable resource for all those involved in the dairy industry, from practitioners to technologists, working in both traditional and modern large-scale dairy operations.

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This edition first published 2009

Wiley-Blackwell is an imprint of John Wiley & Sons, formed by the merger of Wiley's global Scientific, Technical and Medical business with Blackwell Publishing.

Registered office
John Wiley & Sons Ltd, The Atrium, Southern Gate, Chichester, West Sussex, PO19 8SQ, United Kingdom

Editorial office
John Wiley & Sons Ltd, The Atrium, Southern Gate, Chichester, West Sussex, PO19 8SQ, United Kingdom

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Library of Congress Cataloging-in-Publication Data:

Dairy powders and concentrated milk products / edited by Adnan Tamime. – 1st ed.
p. cm.
Includes bibliographical references and index.
ISBN 978-1-4051-5764-3 (hardback : alk. paper) 1. Concentrated milk. 2. Dried milk. I. Tamime, A. Y.
SF259.D323 2009
637′.142 – dc22

2008045264

A catalogue record for this book is available from the British Library.

Typeset in 10/12.5 Times-Roman by Laserwords Private Limited, Chennai, India
Printed and bound in Singapore by Fabulous Printers Pte Ltd

1 2009

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Preface to the Technical Series

For more than 60 years, the Society of Dairy Technology (SDT) has sought to provide education and training in the dairy field, disseminating knowledge and fostering personal development through symposia, conferences, residential courses, publications and its journal, the *International Journal of Dairy Technology* (previously known as the *Journal of the Society of Dairy Technology*).

In recent years, there have been significant advances in our understanding of milk systems, probably the most complex natural food available to man. Improvements in process technology have been accompanied by massive changes in the scale of many milk processing operations, and the manufacture of a wide range of dairy and other related products.

The Society has now embarked on a project with Blackwell Publishing to produce a Technical Series of dairy-related books to provide an invaluable source of information for practising dairy scientists and technologists, covering the range from small enterprises to modern large-scale operation. This latest volume in the series, *Dairy Powders and Concentrated Products,* under the editorship of Dr A.Y. Tamime, provides a timely and comprehensive update on the principles and practices involved in producing these concentrated milk and milk fractions. Though the final products are often shelf stable, the milder methods now used to aid the retention of the nutritional and functional properties have led to a further increase in hygiene standards within the industry. While some products, for instance infant formulae, provide a complete food, a new sector has developed within the dairy industry to provide specialised ingredients to the food industry. This book provides a valuable review of the progress being made in the provision of these products.

Andrew Wilbey
Chairman of the Publications Committee, SDT
September 2008

Preface

Given the recent developments in dairy technology, it has become apparent that the revision of the Society of Dairy Technology publication (Milk and Whey Powders – published in 1980) is overdue. Although there have been some technological developments in the manufacture of these products, including concentrated and sweetened condensed milk, over the past couple of decades, the total world production figures in 2005 ($\times$1000 tonnes; as reported by the International Dairy Federation of the main dairy-producing countries) of condensed products and dairy powders are 1777.6 and 3025.8, respectively. The economic importance of these products to dairy-producing countries is very significant, and there is a large demand for them in countries where milk production is low or non-existent. In these markets, dairy products are made locally to meet the demand of consumers from recombined powders, anhydrous milk fat and concentrated dairy ingredients (evaporated and sweetened condensed milk).

Dairy Powders and Concentrated Products is the latest book in the Technical Series of The Society of Dairy Technology. Numerous scientific data are available in journals and books that have been published since the early 1990s, and the primary aim of this text is to detail in one publication the manufacturing methods, scientific aspects and properties of milk powders (full-fat, skimmed and high-protein powders made from milk retentates), whey powders including whey powder concentrates, lactose, caseinates, sweetened condensed milk, evaporated milk and infant baby feed. The book also covers the international standards relating to these products for trading purposes, as well as the hazards such as explosion and fire that may occur during the manufacture of dairy powders.

The authors, who are all specialists in these products, have been chosen from around the world. The book will be of interest to dairy scientists, students, researchers and dairy operatives around the world and will become an important volume in the Technical Series of Society of Dairy Technology.

A.Y. Tamime
Technical Series Editor
September 2008

This book is dedicated to the memory of Dr Richard Robinson, who generously devoted much time and effort to checking the text of the volumes in the SDT technical series prior to publication.

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1 Chemistry of Milk – Role of Constituents in Evaporation and Drying

H.C. Deeth and J. Hartanto

1.1 Introduction

This chapter discusses the relevance of major milk components to concentrated and dried products, the chemical composition of the various products and some of the quality issues of the products associated with the various components. Knowledge of the chemical composition of these products is essential for understanding their manufacture, applications, nutritional attributes, essential chemical differences and functional properties, as well as the changes that occur during their manufacture and storage. Several comprehensive reviews of the chemical composition of milk are available in dairy chemistry texts and other publications (e.g. Walstra & Jenness, 1984; Wong *et al*., 1988; Fox & McSweeney, 1998; Varnam & Sutherland, 2001; Anonymous, 2003; Walstra *et al*., 2006).

Many factors affect the composition of milk. These include the species and breed of animal from which the milk is derived, the stage of lactation, the season and the nutritional status and health of the animal. In addition, changes to the milk occur after it is harvested and before it is processed, which may affect its processibility. Therefore, it is impossible to provide accurate compositional data. In Table 1.1, 'textbook values' of the major constituents, water, fat, protein, carbohydrate (lactose) and minerals or ash are given for whole milk and skimmed milk, that is, milk from which fat has been removed. Table 1.1 also gives compositional data for a range of concentrated and dried milk products selected from a range of sources. As for the composition of milk, several factors affect the composition of these products also. These include the factors that affect the unprocessed milk and also many processing and storage variables. Therefore, the data in Table 1.1 should be used as a guide only to the composition of particular products. Figure 1.1 shows a graphical comparison of the proximate compositions of the major dried products. For the sake of this illustration, the water content of the powders is assumed to be zero. In practice, however, the water content is approximately 3–5 g 100 g^{-1}.

Table 1.1 and Figure 1.1 illustrate a wide range of compositions of the concentrated and dried milk products. In the following sections, these aspects are discussed in relation to the composition and quality aspects of the concentrated and dried products.

1.2 Chemical components of liquid, concentrated and dried milk products

1.2.1 *Protein*

Both the protein content and protein composition are important in milk concentrates and powders, with some products being characterised by their protein content. For example,

Table 1.1 Proximate composition (g 100 g^{-1}) of liquid, concentrated and dried milk products.

Product	Water	Fat	Protein	Carbohydrate	Ash/minerals
Liquid milks					
Whole milk	87	3.7	3.3	4.8	0.7
Skimmed milk	90	<0.1	3.4	4.9	0.75
Concentrated milks					
Evaporated whole milk					
American standard	72.7–74.7	7.5–8.0	6.5–7.1	9–10	1.3–1.6
British standard	67–69	9–10	8–9	11.0–12.5	1.9–2.1
Evaporated skimmed milk	79.5	0.3	7.6	11	1.6
Sweetened condensed milk	27	9	8	55	1.8
Sweetened condensed skimmed milk	28	0.3	10	59	2.3
Milk powders					
Whole milk powder	2–4	25–28	25–27	37–38	6–7
Skimmed milk powder	3–5	0.7–1.3	35–37	49–52	7.5–8.0
Buttermilk powder	2.8–3.8	3–6	33–36	47–49	7–8
Cream powder	2.6–3.0	55–70	12–15	13–24	2.0–3.5
Milk and whey protein powders					
MPC 42	3.5	1.0	42	46.0	7.5
MPC 70	4.2	1.4	70	16.2	8.2
MPC 75	5.0	1.5	75	10.9	7.6
MPC 80	3.9	1.8	80	4.1	7.4
MPC 85	4.9	1.6	85	1.0	7.1
High milk protein powder	5.3	2.3	88	0.7	7
Caseinate (Ca, K, Na)	3–5	0.9–1.5	89–95	0.2	3.3–5
Casein (acid)	9.5	0.8	97	0.1	1.8
Casein (rennet)	9.5	0.8	90.5	0.1	8.5
Low-protein WPC	4.6	2–4	34–36	44–53	7–8
Medium-protein WPC	4.3	5	53	35	7
High-protein WPC	3–4	4–6	59–65	21–22	3.5–4
Very high-protein WPC	4–5	0.3–7.0	72–81	2–13	2.5–6.5
Whey protein isolate	2.5–6	0.1–0.7	89–93	0.1–0.8	1.4–3.8
Fractionated whey proteins					
α-fraction	4.5	1.0	81.5	7	3.4
β-fraction	4.5	0.4	87	0.5	3.0

Table 1.1 Continued.

Product	Water	Fat	Protein	Carbohydrate	Ash/minerals
Milk/whey protein hydrolysate	4	5	81.5	3	4.5
Whey powders					
Whey powder (acid)	≤3.5	0.8	9–12	65–69	11–12
Whey powder (sweet)	3–6	0.8–1.5	12–13	70–73	7.5–8.5
Whey powder (demineralised)	≤3	≤1.5	≥11	78–82	≤4
Whey powder (demineralised)	≤3	≤1.5	≥11	80–84	≤1.5
Whey powder (deproteinised)	3	0.2–1	2.5	80–85.5	8.5–10
Whey powder (lactose-reduced)	2–3	1–4	18–25	40–60	11–27
Miscellaneous products					
Lactose (food grade)	0.5	0.1	0.1	99	0.1–0.3
Infant formula	2–3	26–39	10–18	40–60	8

MPC = milk protein concentrate; WPC = whey protein concentrate.

Data compiled from Hargrove & Alford (1974), Posati & Orr (1976), Walstra & Jenness (1984), Morr (1984), Bassette & Acosta (1988), Jensen (1990), Morr & Foegeding (1990), Morr & Ha (1993), Caric (1993), Haylock (1995), Huffman (1996), Early (1998), Australian Dairy Corporation (1999), Pintado *et al.* (1999), Holt *et al.* (1999), O'Malley *et al.* (2000), Mistry (2002), Fox (2002, 2003), Mleko *et al.* (2003), Thomas *et al.* (2004), Kim *et al.* (2005), FSANZ (2006), Walstra *et al.* (2006), Millqvist-Fureby & Smith (2007) and Sinha *et al.* (2007).

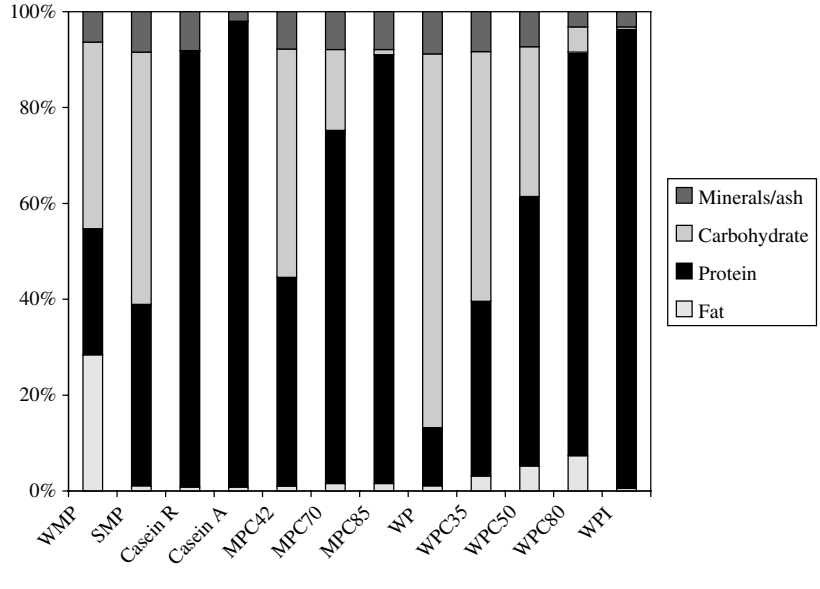

Fig. 1.1 Proximate composition of major milk-derived powders.
WMP = whole milk powder; SMP = skimmed milk powder; MPC = milk protein concentrate; WP = whey powder; WPC = whey protein concentrate; WPI = whey protein isolate; numbers following abbreviations denote approximate protein percentages.

milk protein concentrates (MPC) and whey protein concentrates (WPC) are marketed on the basis of their protein content, for example, WPC80 contains 80 g 100 g^{-1} protein powder. In most cases, the nominal protein content is a crude protein figure, not a true protein figure. The non-protein nitrogen components, such as urea, represent the difference between these two values.

The proteins in milk consist of two broad types, the caseins that are insoluble at pH 4.6 and the whey proteins that are soluble at this pH. About 80 g 100 g^{-1} of the protein is casein and the remainder is whey proteins. Hence, the casein: whey protein ratio in milk is ~4:1. A third minor class is the membrane proteins that form part of both the milk fat globule membrane and the skimmed milk membrane material. The membrane proteins have only a minor role in the properties of most concentrates and powders.

Table 1.1 and Figure 1.1 also show the difference in the protein contents of different powders. Four types of powder stand out as having a high protein content – casein (both acid and rennet), high-protein MPC such as MPC85, high protein WPC such as WPC80 and whey protein isolate. However, the type of protein differs considerably, with caseins being almost entirely casein, MPC containing both casein and whey protein in the same proportion as the original milk and the whey protein products containing mostly whey protein with only a minor amount of casein. Fractionated whey proteins, such as the alpha and beta fractions contain predominantly the whey proteins α-lactalbumin and β-lactoglobulin, respectively.

In Table 1.1 and Figure 1.1, the compositions of two different caseins are shown. This is a good example of a product with the same name produced by different methods having different compositions. Rennet casein produced by coagulation of casein by the action of chymosin (in rennet) is depleted in the glycomacropeptide or casein-derived peptide of κ-casein that remains in the whey, while acid casein, produced by the acid precipitation of casein, contains the complete caseins. This also means that the corresponding rennet and acid wheys differ also with rennet whey containing a substantial amount of the glycomacropeptide (~15 g 100 g^{-1} of the protein), which is not present in acid whey.

In milk, most of the casein exists in the form of casein micelles that contain the four major caseins, α_{s1}-, α_{s2}-, β- and κ-caseins in the ratio of approximately 40:10:35:12. In addition, about 6 g 100 g^{-1} of the solid material in the micelle is colloidal calcium phosphate that acts as 'glue' to help maintain the integrity of the micelle. If the calcium phosphate is removed from the micelle, for example by acidification, the micelles are disrupted and the casein coagulates into curd. Therefore, the form in which the caseins exist in milk products is determined by the processing procedures used. For example, caseins that are produced by acid precipitation are largely in non-micellar form, while the casein in skimmed milk powder (SMP) or MPC is largely 'micellar' (Mulvihill & Ennis, 2003). However, it should be noted that though micelles in milk contain 4–5 g water g^{-1}, the dried micelles in powders contain little water and, hence, are quite different from native micelles.

The micelles in milk range in size from 30 to 300 nm diameter (Varnam & Sutherland, 2001). However, after heat treatment they increase in size. Martin *et al*. (2007) found that the size of the micelles increased on average by ~3, 6 and 39 nm after low-heat (79°C for <5 s), medium-heat (90°C for 30 s) and high-heat (120°C for 4 min) treatment of skimmed milk. This increase is due to the attachment of denatured whey proteins onto the micelles (Oldfield *et al*., 2005). Removal of water by evaporation resulted in much larger

increases of ~60, 78 and 94 nm for low-, medium- and high-heat milks, respectively. These increases in micelle size were attributed to the continued attachment of whey proteins denatured during the heat treatment to the micelles during evaporation and also to soluble casein and calcium moving from the serum to the micelle (Martin *et al*., 2007). On drying and subsequent dissolution of the powder, the sizes of the micelles from low-, medium- and high-heat treatments were somewhat smaller than those in the concentrated milk, but were still considerably larger than those in the heat-treated milk. The micelles decreased in size after dissolution, up to 24 h, due to a slow re-equilibration of the casein, calcium and denatured whey proteins between the micelles and the serum (Martin *et al.,* 2007).

Caseins are relatively small, amphiphilic, randomly coiled, unstructured open proteins with a high proline content, which prevents them from forming helical structures. By contrast, the major whey proteins α-lactalbumin and β-lactoglobulin are small globular proteins. Thus, the caseins are quite stable to heat while the whey proteins, especially β-lactoglobulin, which constitutes about 50 g 100 g^{-1} of the whey proteins, are heat labile.

β-Lactoglobulin exists naturally as a dimer, which unfolds with heat at temperatures above 65°C and exposes a free sulphydryl group (SH) that is normally buried. This activated form of β-lactoglobulin can then react with other molecules of itself, other whey proteins such as α-lactalbumin or cow's serum albumin, or κ-casein, which is concentrated on the outside of the casein micelle, via disulphide (S–S) linkages. The heat-induced denaturation of β-lactoglobulin and the subsequent protein–protein interactions are very important reactions with regard to the stability of concentrated and (reconstituted) dried milk products to heat treatment. They are also important in fouling or burn-on when these products are heated in heat exchangers.

The extent of denaturation as measured by the whey protein nitrogen index (WPNI), a measure of the concentration of un-denatured whey proteins, is widely used to classify SMPs according to the intensity of the pre-heat treatment the milk receives before concentration and drying. Thus, low-, medium- and high-heat powders are characterised by WPNIs of >6.0, 1.5–6.0 and <1.5 mg kg^{-1} [American Dairy Products Institute (ADPI), 1990)]; previously the ADPI was known as the American Dry Milk Institute (ADMI). The corresponding heat treatments are of the order of 70°C for 15 s; 85°C for 1 min, 90°C for 30 s or 105°C for 30 s; and 90°C for 5 min, 120°C for 1 min or 135°C for 30 s (Kelly *et al*., 2006), although industrial conditions vary considerably. Powders are chosen for particular applications on the basis of their WPNI (Kelly *et al*., 2006). Although heat is applied in the pre-heat, evaporation and drying stages, by far the most denaturation occurs in the pre-heat stage (Oldfield *et al*., 2005).

Enzymes represent a minor component of milk proteins. However, they can have significant effects on the quality of milk and milk products. By far the most studied enzymes are the lipases and proteases. Raw milk contains one lipase, lipoprotein lipase (Deeth, 2005) and several proteases, the most significant of which is the alkaline protease namely *plasmin* (Kelly & McSweeney, 2003). Lipoprotein lipase is inactivated by high-temperature short time (HTST) pasteurisation conditions (e.g. 72°C for 15 s), the least severe pre-heat treatment used in manufacture of milk powders (Shamsuzzaman *et al.,* 1986). Therefore, action by the native lipase after manufacture of concentrated and dried products can be dismissed. By contrast, milk plasmin is quite heat stable, and is able to withstand high-heat treatments, even some ultra high temperature (UHT) treatments. Consequently, milk

powders, such as low-heat SMP, contain active plasmin that can degrade milk proteins, particularly β- and α_s-caseins, in products manufactured from milk powder. Newstead *et al.* (2006) demonstrated proteolysis in UHT-treated reconstituted milk produced from low-fat SMP. They showed that the proteolysis could be prevented by employing a pre-heat treatment sufficient to inactivate the plasmin (90°C for 30 or 60 min).

Milk can also contain bacterial enzymes, if psychrotrophic bacteria are allowed to grow to levels sufficient to produce these enzymes, usually $>10^6-10^7$ colony forming units (cfu) mL^{-1}. The most studied of these enzymes are the lipases and proteases. Both types have significant heat resistance and hence, can remain in concentrated and dried milk products, and cause defects in products made from them (Chen *et al.,* 2003; Deeth & Fitz-Gerald, 2006). The lipases can cause hydrolytic rancidity through production of free fatty acids while the proteases can cause bitterness, gelation and sedimentation. Bacterial proteases preferentially attack κ-casein and, hence, their presence can be distinguished from that of milk plasmin which prefers β- and α_s-caseins (Datta & Deeth, 2003). Enzyme action in milk powder is generally assumed to be extremely slow because of the low water content, usually 1.5–5.5 g 100 g^{-1}. However, Chen *et al.* (2003) reported that lipolysis catalysed by bacterial lipase can occur in whole milk powder (WMP) with a moisture content of <3 g 100 g^{-1}. They found levels of short-chain free fatty acids (FFAs) in a powder stored for 2 weeks at 37°C, which exceeded the flavour threshold in the reconstituted milk.

Changes to proteins during storage can have marked effects on the properties of some dried dairy products. For example, the solubility of MPC powders decreases with storage time, particularly at elevated temperatures, and can vary widely, between 32% and 98%. The insoluble material has been shown to consist of large particles (100 μm) in which the casein micelles are joined together by weak non-covalent (hydrophobic) protein–protein interactions. The main individual proteins present are α- and β-caseins. κ-Casein and β-lactoglobulin are also present in disulphide-linked protein aggregates, but they are not the main cause of the insoluble material (Havea, 2006).

The solubility of casein prepared by acid precipitation can be increased by reaction with alkalis to water-soluble caseinates. If sodium hydroxide is used to solubilise acid casein, it produces sodium caseinate, a common casein product, which is useful in the wide range of industrial application (Mulvihill & Ennis, 2003). Sodium caseinate is also the starting material for producing fractionated caseins (Murphy & Fox, 1991). This procedure is based on the fact that β-casein becomes soluble at low temperature, for example 4°C, and can be separated from the remaining micellar-bound caseins by membrane filtration. It produces two major fractions, one enriched in β-casein and one enriched in α_s- and κ-caseins.

Caseinates can be highly soluble in water and fairly flavourless if the pH during manufacturing is never higher than 7 (Walstra *et al.,* 2006). During the production of caseinates, the time for which the caseinate solution is held at high temperature should be minimised to limit the extent of browning. The duration for which the casein is exposed to high pH during dissolving should also be reduced, since this may initiate the formation of lysinoalanine and the development of off-flavours.

1.2.2 *Fat*

Most concentrated and dried milk products have low-fat contents. The exceptions are WMP and the speciality and less common cream and butter powders. Whey produced during

cheesemaking contains a small amount of fat even after separation to remove most of the fat. The fat content of whey products tends to increase with an increase in protein content as the processes used primarily to remove lactose, ultrafiltration (UF) and diafiltration (DF), retain the fat as well as the protein.

While the majority of the fat is triglyceride, milk contains fat in the form of phospholipids also, which are present in both the milk fat globule membrane and skimmed milk membrane material in approximately equal amounts. As the membranes are particulate, they are also concentrated by the membrane filtration methods used to concentrate the protein. Buttermilk powder (BMP) contains the highest concentration of phospholipids as the milk fat globule membrane is released into the butter serum (skimmed milk) when the cream is churned.

The fat in milk exists in the form of fat globules 0.2–15 μm in diameter, most in the range 1–8 μm, with an average of about 3 μm. Each globule is enveloped in a biological membrane, known as the *milk fat globule membrane*. This membrane protects the fat from attack by the naturally occurring lipase present in the milk serum and also disperses the hydrophobic fat in the hydrophilic aqueous medium. If this membrane is mechanically disrupted, non-globular or *free fat* is released from the globules. Free fat is defined as that which can be extracted by non-polar organic solvents such as hexane (Evers, 2004). Free fat can be formed if the globules are subjected to sheer forces during processing. Therefore, provided the milk has not been subjected to such forces, the fat globules in concentrated and dried products remain largely in globular form. However, in some products, free fat is produced during processing (Fäldt & Bergenstahl, 1995). For example, during drying, the fat of the milk fat globule does not shrink, but that in the milk fat globule membrane does, which causes rupture of the membrane and production of free fat. The free fat is present predominantly on or close to the surface of powder particles and in cracks and fissures in the surface of the particle (Buma, 1971). Free fat has a detrimental effect on some functional properties of powders such as flowability and dispersibility, and is more susceptible to oxidative deterioration than globular fat is during storage. However, a high free fat level in some dried products is beneficial for some applications, such as chocolate manufacture (Liang & Hartel, 2004).

A minor lipid in milk is cholesterol. While its effect on the functional properties of concentrated and dried products is negligible, it has nutritional significance. It is mostly associated with the milk fat globule membrane and skimmed membrane material and, therefore, its proportion of the total fat in a product depends on the relative significance of membrane lipids in the total fat. For example, SMP has ~30 mg cholestrol 100 g^{-1} of powder while WMP has ~90 mg 100 g^{-1}. Since the relative total fat contents of SMP and WMP are ~1 and 26 g 100 g^{-1}, respectively, the cholesterol as a percentage of the total fat in the SMP is much high than in the WMP. BMP contains ~70 mg cholesterol 100 g^{-1} (Walstra & Jenness, 1984), over twice the content of SMP because it contains both the milk fat globule membrane and skimmed membrane material.

Lecithin is a phospholipid, which is sometimes added to concentrated and dried product. The material used in the dairy industry originates from soya bean but has a similar composition to the phospholipids of the milk fat globule membrane. Phosphatidyl choline and phosphatidyl ethanolamine are the major components. One application of adding lecithin is to 'instantise' powders, that is, to improve their dissolution properties, because of its

surfactant properties. A second application is for increasing the heat stability. BMP, which contains milk fat globule membrane material rich in phospholipids, is also useful for this purpose (Singh & Tokley, 1990).

1.2.3 *Carbohydrate*

The carbohydrate in milk is almost entirely lactose which, at about 5 g 100 g^{-1}, is the single most abundant constituent. Therefore, on direct concentration of milk or whey, the final product contains a high percentage of this compound (Table 1.1 and Fig. 1.1). In the production of high-protein products, such as MPC and WPC, UF and electrodialysis or DF are used primarily to remove the lactose. Consequently, the permeates from these processes are rich in lactose and can be concentrated to produce crystalline lactose, another dried milk derivative.

The nature of lactose in the final product can have a significant effect on the product's properties and quality. Lactose is highly hygroscopic, but its form determines how much water it can absorb. The amorphous form is more hygroscopic than the most common crystalline form, α-lactose monohydrate. Lactose can be the cause of several problems in milk powders including collapse, stickiness and caking (Listiohadi *et al.,* 2005). The glass transition temperature of lactose-containing milk powders is similar to that of pure lactose, which indicates the importance of lactose in determining the physical state of these powders (Jouppila & Roos, 1994).

During preparation of powders containing high levels of lactose, seed crystals of α-lactose monohydrate are added to the concentrate before drying to enable the anhydrous α-lactose to convert to the crystalline monohydrate form before the drying stage. This decreases the risk of caking or clumping in the powder during storage, caused by the conversion of the anhydrous form to the crystalline form. In the production of lactose, concentrates of whey or permeate with 60–65 g 100 g^{-1} solids are seeded with α-lactose crystals to induce crystallisation from a supersaturated lactose solution. After crystallisation, the crystals are separated from the mother liquor, washed and dried to ~0.5 g 100 g^{-1} water.

In whey derived from cheesemaking, some of the lactose is converted to lactic acid by the starter bacteria. Excessive lactic acid content can lead to a highly sticky and hygroscopic product (Varnam & Sutherland, 2001). However, in some whey products, the lactose is hydrolysed to glucose and galactose. The resulting product is sweeter than whey and can be used as a sweetener. The hydrolysis is usually carried out with the enzyme β-galactosidase, but can also be effected with acid at high temperature in products such as permeates, which contain no protein.

Lactose, being a reducing sugar, can interact with amine groups of proteins, particularly the ε-amino group of lysine, in the initial reaction of the Maillard series of reactions. The lactose–protein interaction is initiated during heat treatment but continues during storage and, in some products lactosylated proteins constitute a significant proportion of the total proteins.

The other major carbohydrate used in concentrated and dried products is sucrose. The main product containing sucrose is sweetened condensed milk where it constitutes around 43 g 100 g^{-1}, over half of the dry matter in the product (Walstra & Jenness, 1984).

In this product, it depresses the water activity to a point where the product is shelf stable.

1.2.4 *Minerals*

Minerals, often determined as 'ash', constitute the smallest of the major groups of milk components. Milk contains a wide range of minerals (Table 1.2), the most abundant of which are potassium, calcium, phosphorus and sodium; the levels of these in a range of products are summarised in Table 1.3.

Despite their low total abundance, minerals have a significant effect on many aspects of concentrated and dried products. As for other constituents considered above, the levels of particular minerals in these products are determined by several factors, but the most significant is the process used in manufacture of the product. For example, a concentrated milk prepared by UF has a lower content of minerals than one prepared by reverse osmosis (RO). The former allows (unbound) minerals to pass through the membrane, whereas the latter retains all milk components except water. Similarly, products made by evaporation and/or drying retain all the minerals of the parent milk.

A large proportion of calcium and phosphorus (~two-thirds and a half, respectively) are intimately associated with the casein micelle and as long as the micelle is intact, significant proportions of the calcium and phosphorus are not removed by UF or DF. Conversely, if the micelle is substantially disrupted, for example by acidification, calcium and phosphorus are solubilised and pass through the UF or DF membranes. Similarly, casein prepared by rennet coagulation has substantially more calcium and phosphorus than casein prepared by acid precipitation (Table 1.3).

Calcium has a major role in the functional properties of milk products and so knowledge of the chemistry of this metal in these products can help in understanding its importance. It exists in three forms in milk, *insoluble* calcium in the form of colloidal calcium phosphate

Table 1.2 Average mineral composition of whole cow's milk.

Minerals	Concentration (mg L^{-1})	Minerals	Concentration ($\mu g\ L^{-1}$)
Sodium	530	Manganese	30
Potassium	1360	Iodine	100–770
Chloride	970	Fluoride	20
Calcium	1120	Selenium	10
Phosphorus	890	Cobalt	0.5
Magnesium	110	Chromium	2
Iron	0.5	Molybdenum	50
Zinc	3.9	Nickel	26
Copper	0.09	Arsenic	20–60
		Silicon	700
		Boron	500–1000

After Flynn & Cashman (1997).

Table 1.3 Mineral composition of liquid, concentrated and dried milk products.

Product	Sodium (mg 100 g^{-1})	Potassium (mg 100 g^{-1})	Calcium (mg 100 g^{-1})	Phosphorus (mg 100 g^{-1})	Total ash (g 100 g^{-1})
Liquid milks					
Whole milk	41–46	152–155	114–123	103	0.7
Skimmed milk	44–49	150–164	117–123	103	0.75
Concentrated milks					
Evaporated whole milk	100–108	300–368	255–263	220–247	1.5–1.7
Evaporated skimmed milk	91–110	324–330	246–290	190	1.5–1.7
Sweetened condensed whole milk	2105–130	357–402	268–300	240–250	1.8
Sweetened condensed skimmed milk	125–130	445–475	335–340	230–280	2.3
Milk powders					
Whole milk powder	310–400	1157–1300	875–910	800	5–6
Skimmed milk powder	428–530	1603–1790	1183–1260	970–1103	7.9–8.5
Casein and whey protein powders					
Ca-caseinate	50–100	100	1000–1500	800	3.5–4.5
K-caseinate	60	1650	300	800	3.3–4.0
Na-caseinate	1200–1300	20	100	800	3.3–4.0
Casein (acid)	100		80	900	2.2
Casein (rennet)	20	30	300	1500	7.5
Low-protein whey protein concentrate (~35 g 100 g^{-1})	460	1190	480	500	5.7–7.8
High-protein whey protein concentrate (65 g 100 g^{-1})	280	650	350	330	3.5–3.9

After Hargrove & Alford (1974), Posati & Orr (1976), Bassette & Acosta (1988), Jensen (1990), Caric (1993), Australian Dairy Corporation (1999), Anonymous (2003), Boumba *et al*. (2001), Fox (2003), Kim *et al*. (2005), FSANZ (2006) and Walstra *et al*. (2006).

associated with the casein micelles, *soluble* or non-micellar calcium and free *ionic* calcium. In cow's milk, the concentrations are ~20, 10 and 1.5 mM, respectively. The ionic calcium is a component of the soluble calcium. These forms are in equilibrium which means that changing the concentration of one form affects the concentration of the others. Milk with a high level of ionic calcium is known to be unstable to heat and hence this component is an important consideration in heat treating milk. For example, goat's milk has a much higher level of ionic calcium than cow's milk, and is much more unstable to high-temperature treatment (Zadow *et al*., 1983).

1.2.5 *Water*

Water content is an important parameter, which distinguishes between different products, determines several physical properties and affects the stability of products during storage. It is particularly important for powders where the ideal level is ~3 g 100 g^{-1}. The water contents of various products are shown in Table 1.1.

Water can be present in food in at least three forms: free water, adsorbed water and bound water. Free water occupies the void volume or the pores of the food. It functions as a dispersing agent, as a solvent for crystalline compounds and as a support for microbial growth. Adsorbed water is present on the surface of the macromolecules in the food matrices, whereas bound water is the water of hydration, which is bound to the product by strong hydrogen bonds (Mathlouthi, 2001).

Milk powders are generally hygroscopic and hence increase or decrease in water content according to the environmental relative humidity (RH). At a particular temperature, the water content of a powder is related to its water activity via its sorption isotherm. The water activity is important as it determines the glass transition temperature of the powder, which in turn determines its physical and chemical properties. Physical changes, such as lactose crystallisation and caking generally, occur when the storage temperature is above the glass transition temperature (Thomas *et al*., 2004).

In the sorption isotherm of lactose-containing powders, an interesting phenomenon occurs between 40 and 50% RH. A break characterised by a sharp decrease in moisture content occurs as water is released when amorphous lactose crystallises as α-lactose monohydrate (Thomas *et al*., 2004).

Although in some cases low moisture content may favour fat oxidation, high moisture content in powders is of greatest concern as it has more negative impacts which limit shelf-life. These include protein denaturation, acceleration of the non-enzymatic browning (Maillard) reactions, enzymic reactions, conversion of amorphous lactose to crystalline α-lactose monohydrate, formation of free fat in whole milk powder, caking of powder during storage and microbial growth (Early, 1992; Verdurmen & de Jong, 2003).

1.2.6 *Air*

While gases including air are present in liquid milk in small amounts, air constitutes a major proportion of some powders. It is occluded into the powder during drying in the form of vacuoles. The air content can increase immediately after drying because a partial vacuum is created in the entrapped air during formation of the particles (Thomas *et al*., 2004).

The amount of air is inversely proportional to the density of the product. The bulk density of powders is around 0.6–0.7 g mL^{-1} compared with the density of non-fat milk solids of ~1.6 g mL^{-1}. The amount of occluded air is influenced by several factors:

- *Processing stages (pre-heat treatment and evaporation)* – These influence the bulk density of powders through the extent of denaturation of the whey proteins in milk. Un-denatured whey proteins increase air occlusion, while denaturation increases particle density.

- *Foaming ability of the concentrate* – Due to its lower degree of protein denaturation, low-heat skimmed milk forms more stable foam than high-heat skimmed milk. A stable foam will hold air to the atomiser and lead to more trapped air in the powder particles produced.
- *Agitation of the concentrate* – Mechanical agitation of the concentrate causes entrainment of air.
- *Drying conditions* – High air temperatures during the critical drying stage promote occlusion of air.
- *Atomisation type* – Pressure rotary atomisers incorporate more air into droplets than atomiser nozzles.
- *Dryer feed concentration* – Low-solid feed materials have lower viscosity and foam more readily and thus lead to more air incorporation (Early, 1992).

If the air content is too high, the product is too bulky and difficult to handle. For this reason, many powders are instantised by agglomeration to increase the average particle size and increase the bulk density of the product. Agglomeration is a process in which small particles coalesce to create large relatively permanent masses, where the original particles are still identifiable. The agglomerates produced are porous clusters of particles 250–750 μm in diameter, and have a high level of entrapped air. Agglomeration improves the rehydration behaviour of the powder since the open porous configuration of the agglomerates allows water to penetrate into the particles, forcing the particle to sink. In spray dryers, agglomeration may occur within the atomiser spray, between sprays of various atomisers, between sprays and dry material introduced into the drying chamber or on a fluid bed outside the spray chamber.

As air in contact with powder particles can cause oxidation during storage, particularly if the powder contains readily accessible fat, powders are often flushed with and stored under nitrogen, or vacuum packaged. This is particularly important in high fat powders.

The bulk density of SMP is generally higher than WMP due to the lower density of milk fat relative to protein and lactose. However, this difference is somewhat counteracted by the presence of fat in whole milk powder which may hinder foaming and reduce the amount of occluded air.

1.3 Surface composition of powders

Many properties of milk powders, which are important in their storage, handling and final application (e.g. dispersibility, wettability, flowability and oxidative stability), are influenced by the surface composition rather than the bulk composition of the powder (Hindmarsh *et al*., 2007). If one of the milk components is preferentially present on the powder surface, the properties can be changed dramatically. Of particular importance is the amount of fat on the powder surface. The presence of fat renders the powder surface hydrophobic, thus decreasing its wettability and dispersibility.

During the drying process, evaporation and drying simultaneously promote the migration of milk constituents, particularly fat, protein and lactose toward the particle surface. Nijdam

& Langrish (2006) studied the migration of these components within milk droplets and particles in a spray dryer and found that the surface fat coverage is much higher than the average fat content of the powder. This implies a higher concentration of fat accumulated at the surface of each milk particle than in the interior leading to a non-uniform distribution of fat throughout the solid matrix. For instance, the surface fat coverage may reach as high as 50%, when the average fat content of the milk powder is only 15%. The fat begins to appear on the powder surface even at very low-fat contents and between 0 and 5% it increases up to 35% (Nijdam & Langrish, 2006). Kim *et al*. (2002) found that the fat on the surface was mostly free fat, and that fat globules protected by proteins were preferentially located underneath the surface fat. The next dominant milk component on the surface of powders is protein, possibly because of its surface-active nature, then lactose (Kim *et al*., 2002, 2005; Nijdam & Langrish, 2006; Shrestha *et al*., 2007) (Table 1.4).

The flowability of powders is a surface-related property and is, therefore, controlled by the powder surface composition. Of special importance is the surface fat which renders the surface of fat-containing powders sticky and causes the particles to adhere to one another thus decreasing the flowability of the powder. By contrast, SMP whose surface is made up mostly of lactose and protein flows well (Kim *et al*., 2005). Kim *et al*. (2005) have concluded that the surface fat content rather than free-fat and total fat content correlates best with flowability. The fact that high surface fat coverage is responsible for poor flowability of powders was clearly demonstrated when flowability was significantly increased by removal of the fat from the surface with petroleum ether. The dissolution rate of the powder in water also decreased with increasing surface fat (Millqvist-Fureby *et al*., 2001).

Millqvist-Fureby *et al*. (2001) used WPC to examine the effect of heat treatment of whey proteins on the powder surface composition and some functional properties of spray dried protein-stabilised emulsions. The heat-treated (denatured) protein has fewer active

Table 1.4 Surface composition[a] of industrial spray dried dairy powders and skimmed milk powders with different lactose levels.

	Bulk composition (g 100 g^{-1})			Surface composition[b] (%)			
Powders	Lactose	Protein	Fat	Lactose	Protein	Fat	References
SMP	58	41	1	36	46	18	Kim *et al*. (2005)
WMP	40	31	29	2	—	98	
CP	13	12	75	1	—	99	
WPC	8	86	6	6	41	53	
SMP:lactose (3:1)	63	26	0.8	29	61	10	Shrestha *et al*. (2007)
SMP:lactose (1:1)	75	17	0.5	31	58	11	
SMP:lactose (1:3)	88	9	0.25	39	57	5	

WMP = whole milk powder; SMP = skimmed milk powder; CP = cream powder; WPC = whey protein concentrates.

[a]Assuming that dairy powders are composed of three main components, namely, lactose, protein and fat.

[b]Based on data from X-ray photoelectron spectroscopy (XPS) or electron spectroscopy for chemical analysis (ESCA).

surface sites and competes less favourably for the interface than the untreated protein. Thus the powder surface coverage of protein decreases with increasing degree of protein denaturation before the emulsification. This leads to more leakage of fat onto the powder surface.

Nijdam & Langrish (2006) proposed that higher drying temperatures accelerate the formation of a surface skin which hampers the migration of surface-active protein towards the surface; this results in the preferential presence of lactose over protein at the surface of the milk powder particle. At the lower drying temperature of 120°C, the surface has a higher concentration of protein than lactose, even though there is more lactose than protein in the bulk powder. However, at the higher drying temperature of 200°C, this trend is reversed and more lactose appears at the surface of the powder than protein, although the ratio of lactose to protein on the surface is still generally lower than the average value in the powder. This phenomenon occurs because protein has more time to drift to the surface of the droplet at lower drying temperatures, before sufficient moisture is evaporated to cause formation of the skin (Nijdam & Langrish, 2006). The relative amounts of lactose and protein on the surface of powder particles are important as the proportion of lactose strongly influences the caking of milk powders during storage at high RH when the glass transition temperature is exceeded (Jouppila & Roos, 1994; Lloyd *et al*., 1996).

Modifying the protein content of SMP by addition of lactose affects the surface composition, sorption behaviour and glass transition temperature of spray dried powder (Shrestha *et al.,* 2007). As shown in Table 1.4, as the lactose:protein ratio increases, there is no a proportional increase in the lactose content on the surface of the powder. This suggests that the lactose migrates to the surface slower than protein and fat. Shrestha *et al*. (2007) reported that increases in lactose concentration in SMP significantly increased the water adsorption in milk powders and also lowered the water activity range at which the crystallisation occurred.

1.4 Quality issues

1.4.1 *Heat stability*

The major issue associated with concentration and drying of milk is the heat stability of the concentrated milk during sterilisation processes, in-container or UHT, and the heat stability of the powders when reconstituted. Heat instability is manifested in gelation or coagulation during heating, sedimentation after heating and burn-on or deposit formation in heat exchangers during continuous UHT treatments. Concentrated milks may also thicken and gel during storage; this may be initiated by the heating process but is considered to be a storage-related issue.

Heat stability of milk has been studied extensively in both single-strength and concentrated milks. Despite this research effort, the scientific basis of heat instability has not been completely elucidated. The effects of many compositional factors on the heat stability of unconcentrated milk are now well known, but many of these do not hold for concentrated milks. Conversely, a knowledge of the heat stability of unconcentrated milk, determined by the classical heat coagulation time (HCT) test (time to coagulate at 140°C) is not a reliable

guide to the heat stability of concentrated milk (Williams, 2002). The desired HCT of milk is 20 min at its natural pH (~6.7).

The HCT test is carried out by heating milk in a closed tube in an oil bath and observing the first signs of coagulation. For normal milk the test is performed at 140°C, but for concentrated milk, 120°C is used. The test is quite subjective and requires some experience to obtain consistent results. For this reason, alternative methods of estimating heat stability have been sought. Lehmann & Buckin (2005) observed the heat stability of recombined evaporated milk (REM) subjected to different pre-heat treatments using high resolution ultrasonic spectroscopy. They observed four stages. In the first stage, ultrasonic velocity decreases sharply while the attenuation increased due to the thermal equilibration of the sample (the time required by the sample to achieve the holding temperature) together with the fast denaturation of whey protein and precipitation of calcium phosphate onto the micelles that occurs during the first few minutes of heating. The second stage, called the pre-coagulation stage, showed small changes in ultrasonic velocity, the slope of which depended on the nature and pH of the samples. In the third stage, ultrasonic velocity and attenuation declined sharply and the attenuation profile reached a peak. This stage was identified as the coagulation point when the gel network is formed. The last stage demonstrated small changes in ultrasonic velocity and attenuation and was attributed to the end of the coagulation process.

A major factor in the heat stability of both unconcentrated and concentrated milk is pH. The HCT–pH curves for unconcentrated milk are of two types, A and B, where type A curves exhibit a distinct maximum (pH ~6.7) and minimum (pH ~6.9); while type B milks show no maximum or minimum, there is a gradual increase in HCT with pH. The majority of milks are of type A. Concentrated milk shows a much different curve with a maximum at lower pH and no minimum; the height of the maximum is much lower than the height of the maximum for type A milk. Concentrated milk is less heat stable than unconcentrated milk at all pH values, especially at pH values higher than the heat stability maximum. Since the heat stability decreases with increasing solids content, the classic heat coagulation test for concentrated milk is performed at 120°C rather than 140°C as used for unconcentrated milks. Typical HCT–pH curves for unconcentrated milk type A and concentrated milk are shown in Figure 1.2 (Singh *et al.,* 1995). This shows the maximum for concentrated milk at ~6.6; however, the actual pH of maximal heat stability depends on several factors including the pre-heat treatment and concentration level. A corresponding heat stability curve for concentrated milk given by O'Connell & Fox (2003) shows a maximum at pH ~6.45.

Milk in which acidity develops due to bacterial growth before heat treatment can produce concentrated and dried products, which are unstable to heat treatment because of the pH–heat stability relationship. Similarly, milk powders which have been stored for some time, especially if stored under unfavourable conditions (i.e. elevated temperature and/or water activity) may have a lower pH than normal and be less heat stable than fresh powders (Zadow & Hardham, 1978). The stability of powders to the relatively low pH and high temperature of coffee solutions is the basis of the coffee stability test (Oldfield *et al*., 2000).

The heat stabilities of milks, concentrates and reconstituted powders show a seasonal or stage of lactation trend although the corresponding variation in the levels of relevant

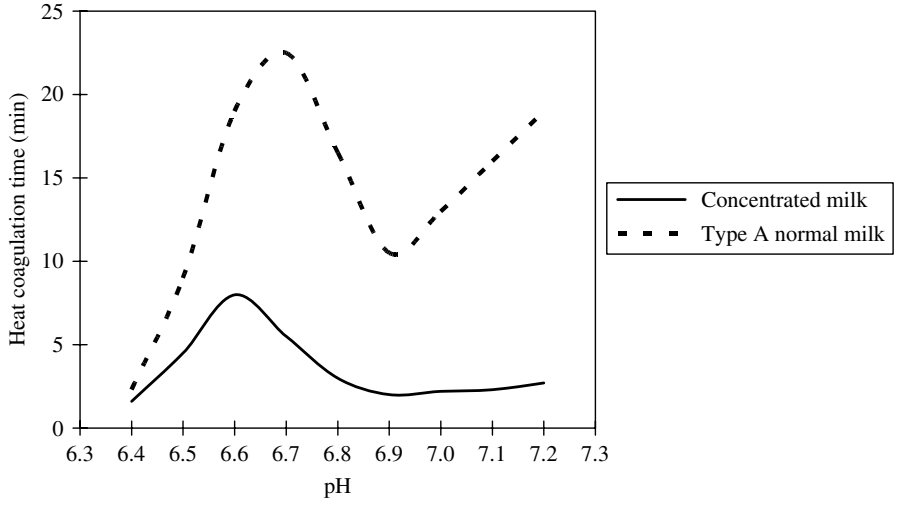

Fig. 1.2 Heat coagulation times at 140°C as a function of pH for normal (single-strength) milk and concentrated milk.

milk components is not often obvious. Singh *et al*. (1995) reported that, in New Zealand, low-heat stability corresponded with the beginning and the end of the dairying season. The seasonal variation seems to be associated with changes in the pH of maximal stability, which is also affected by the nature of the pre-heat treatment. According to Newstead *et al*. (1975), the pH of maximal stability of REM was lower than its natural pH for much of the year in New Zealand. Thus, judicious pH adjustment can improve the heat stability.

The type and sequence of processing steps have a significant effect on the heat stability of concentrates. Pre-heat treatment of the milk prior to concentration is the most important of these steps. More severe heat treatments lead to more heat-stable concentrates. The major effect of pre-heat treatments is denaturation of whey proteins to form whey protein aggregates and whey protein–casein complexes, formed largely through disulphide linkages between κ-casein and β-lactoglobulin. Concentrates with a high level of un-denatured β-lactoglobulin are significantly less stable than those with lower concentrations. This destabilisation occurs over the entire pH range. By contrast, addition of κ-casein can enhance the heat stability of concentrates (Muir & Sweetsur, 1978).

The significant effect on heat stability of denaturation and subsequent aggregation of β-lactoglobulin is clearly demonstrated by the addition of SH blocking or oxidising agents; both improve heat stability. Blocking agents include N-ethyl maleimide and iodoacetamide, and oxidising agents include hydrogen peroxide and Cu^{2+} (Walstra & Jenness, 1984). However, addition of these compounds is not practised commercially as they are not legal additives in most countries. Addition of urea to unconcentrated milk increases its heat stability (Dalgleish *et al*., 1987), but it has no beneficial effect in concentrated milk (Muir & Sweetsur, 1978).

Homogenisation, an important step when whole milks are concentrated, decreases the heat stability of the concentrated milk. However, the effect of homogenisation is minimised

by pre-heating the milk before concentration and homogenisation. Pre-heating can be in the UHT range (145°C for 5 s) (Sweetsur & Muir, 1982) or at a lower temperature (120°C for 120 s) (Newstead *et al*., 1979). Sweetsur & Muir (1983) found that the sulfhydryl interactions between β-lactoglobulin and κ-casein have a more marked effect on the heat stability of homogenised than on unhomogenised concentrated milk.

The minerals in concentrated milk have a major role in heat stability. Reduction in the mineral content before concentration (Muir & Sweetsur, 1978) or concentration by UF, which results in loss of minerals through the membrane (Sweetsur & Muir, 1980), increase the heat stability of the concentrates. The so-called salt balance theory, which was first developed over 50 years ago (Sommer & Hart, 1922), suggests that, apart from whey proteins, the ratio of calcium and magnesium to phosphate and citrate controls heat stability. The influence of other factors such as pH may be largely through their effects on the salt balance.

High ionic calcium levels are associated with low-heat stability. Thus, addition of stabilising salts, such as disodium hydrogen phosphate and trisodium citrate which reduce the calcium ion activity, can enhance the HCT of evaporated milk (de Jong & Verdurmen, 2001). They also increase the pH of the product. The heat stability can also be improved by decreasing the calcium content of the milk before evaporation by means of ion exchange (Walstra *et al*., 2006) or by adding phosphate prior to pre-heating (Horne & Muir, 1990). Hardy *et al*. (1984) suggested that heat stability relied on the mineral equilibrium which determined the concentration of soluble calcium. During processing, both calcium and phosphate tend to migrate from the serum to the colloidal phase.

Thus, in addition to pre-heat treatment, adding salts, such as phosphates and citrates, is a major means of controlling the heat stability. However, the choice of additives is not straightforward. Sometimes addition of the acidic phosphate, sodium dihydrogen phosphate, is most appropriate and sometimes addition of the basic disodium hydrogen phosphate is most appropriate. In general, if the natural pH of the milk is higher than the pH of maximal stability, addition of sodium dihydrogen phosphate may be beneficial while, if the natural pH of the milk is lower than the pH of maximal stability, addition of disodium hydrogen phosphate or trisodium citrate is recommended (Singh *et al.,* 1995). In practice, use of a pilot plant to test the most appropriate additive is the best test available to the processor as data from traditional heat stability tests are poorly correlated with behaviour of the product in commercial processing.

Hardy *et al*. (1985) reported that a heat-stable concentrate can be achieved by means of lecithin incorporation without addition of inorganic phosphate and can be processed at a higher than usual homogenisation pressure. The addition of lyophilised salted butter serum to concentrated skimmed milk shifted the pH of maximum heat stability to a higher value and, at certain concentrations, increased the maximum HCT. It was suggested that the beneficial effects of the butter serum on the heat stability may be due to the sodium chloride present. The role of sodium chloride in shifting the pH of maximum stability may be through a reduced micellar charge. Addition of sodium chloride to milk may increase the level of non-sedimentable calcium, as sodium can replace the calcium in colloidal form. This results in an increased level of soluble calcium which will reduce the micellar charge; a higher pH will then be required to gain the same net negative charge (Huppertz & Fox, 2006).

1.4.2 *Fouling*

Fouling or deposit formation occurs at the surface of heat exchangers used for heating milk and milk products. The build-up of deposit reduces the heat transfer rate and, hence, the heating medium temperature has to increase to maintain the same product temperature. This increase in temperature exacerbates the fouling. The fouling deposit also blocks the flow of product through plate and tubular heat exchangers, and increases the back pressure in the plant. When the temperature of the heating medium and/or the back pressure in the plant becomes excessive, the plant has to be shut down for cleaning. Overall, fouling is costly for the dairy industry because of the down time required for cleaning, the loss of milk, the increased cost of detergents required and the greater quantity of wastewater produced (Walstra *et al*., 2006).

Fouling is more significant at high temperatures than at low temperatures and, hence, is a significant issue with UHT processing. At heating temperatures of ~80–115°C, the fouling layer is relatively soft and consists mainly of proteins (50–70 g 100 g^{-1}), but at higher temperatures – up to 150°C – encountered in UHT plants, the fouling layer is harder and is predominantly mineral (~70 g 100 g^{-1}). The mineral is mostly calcium phosphate as this becomes less soluble at higher temperatures. The lower-temperature deposit is known as type A, while the higher-temperature deposit is known as type B. Type A is yellowish in colour, voluminous and curd-like while type B, often called milk stone or scale, is greyish in colour, hard and gritty (Burton, 1988; Walstra *et al*., 2006).

Fouling is influenced by several factors, but a major one of significance here is the solids content. Concentrated milks foul more readily than single-strength milks. There are several possible explanations for this; these include: (a) the higher content of β-lactoglobulin (Tissier *et al.,* 1984), (b) higher content of calcium (Jeurnink & de Kruif, 1995), (c) higher lactose levels which cause Maillard reactions (Jeurnink *et al.,* 1996), (d) lower pH (Singh *et al.,* 1995) and (e) higher viscosity (Kastanas, 1996) of concentrated milk compared with single-strength milk. In a recent study of fouling in concentrated reconstituted skimmed milk up to 20 g 100 g^{-1} solids, Prakash (2007) concluded that denaturation of β-lactoglobulin was most significant as the use of the SH blocking agent iodoacetamide markedly reduced fouling. High lactose levels, lower pH and high viscosity had comparatively little effect on fouling. Similarly, reduction of ionic calcium with trisodium citrate or sodium hexametaphosphate did not reduce fouling in the concentrated milk in contrast to its beneficial effect in single-strength cow's and goat's milk (Prakash, 2007; Prakash *et al*., 2007).

Denaturation of β-lactoglobulin during heat treatment is considered to be significant in most fouling situations. In fact, the intermediate unfolded form is considered the most adhesive form, and the faster the β-lactoglobulin passes this intermediate stage and aggregates with itself, other whey proteins or κ-casein, the less severe is the fouling (Grijspeerdt *et al.,* 2004). Milk or whey that has been heated to such an extent that β-lactoglobulin is completely aggregated produces minimal protein fouling (Walstra *et al.,* 2006). However, mineral deposits of largely calcium phosphate still occur in the high-temperature sections of the plant.

Another type of fouling occurs in evaporators and in the regeneration section of plate heat exchangers (Lehmann *et al.,* 1992). This is known as microbial fouling or bio-fouling. During processing at temperatures below 80°C, thermoduric bacteria, such as *Streptococcus*

thermophilus, can attach to the surface walls of heat exchangers and grow as a film. These biofilms can subsequently detach and contaminate the products when plants are run for extended times. The bacterial growth, adherence and amount of bacteria released into the product have been modelled as a function of operating time. This enables plant conditions to be optimised to minimise bio-fouling and for the amount of contamination of product by thermophilic bacteria to be predicted (de Jong & Verdurmen, 2001). Knight *et al*. (2004) devised a successful method of minimising biofilm build-up by using a temperature cycling procedure. This system effectively interrupts the growth cycle of the bacteria and prevents their rapid growth typical of the logarithmic phase.

1.4.3 *Age thickening*

Age thickening is a further ramification of protein instability. During storage the viscosity of shelf-stable milk increases and this may lead to gelation in the product. It occurs in both single-strength and concentrated milk but the mechanism of the change appears to be different for both types of milk. In single-strength milk it is largely initiated by proteolysis, whereas in concentrated milk it occurs without proteolysis (Datta & Deeth, 2001).

Viscosity is a very important parameter for concentrated milks and high-temperature processing. Controlling the viscosity is imperative in the manufacture of sweetened condensed milk. The viscosity needs to be high enough to prevent sedimentation and creaming of the fat, but not excessively high for ease of processing.

The steps taken to minimise age thickening are essentially the same as those to minimise protein instability problems during processing, that is, pre-heating of the milk prior to concentration to denature whey proteins (de Jong & Verdurmen, 2001; Walstra *et al*., 2006), and addition of stabilising or buffering salts, such as sodium and potassium hydrogen carbonate, calcium chloride, sodium and potassium phosphate, sodium and potassium diphosphate, disodium or trisodium phosphate, sodium and potassium citrate, sodium and potassium orthophosphate (Caric, 1993; Spreer, 1998; Brennan, 2006). Sterilisation of the concentrated product under intense conditions delays thickening and gelation as it does for unconcentrated milk (Datta & Deeth, 2001).

1.4.4 *Maillard reactions*

Maillard reactions are initiated by the reaction of a reducing sugar, such as lactose with amino residues on proteins, chiefly the ε-amino group of lysine. The final products of the reactions are brown-coloured melanoidins, which impart a brown colour to affected products. In addition, several intermediate compounds are formed, such as hydroxymethylfurfural and formic acid, the latter responsible for some of the pH reduction in stored dairy products. Maillard reactions cause flavour and colour changes in milk products, such as concentrated and dried products, and also reduce their nutritive value. An extreme case of Maillard browning occurs in so-called scorched particles in powders, which are a significant defect.

The first step in milk products, lactosylation, results in lactose adducts of proteins and reduces the availability of lysine, an essential amino acid. The reaction is initiated during

heating but continues during storage. Lactosylation produces ε-N-deoxylactulosyl-*D*-lysine or lactulosyl lysine, the most stable product of the Maillard reactions. Lactosylation occurs readily in the dry state. In fact, Morgan *et al*. (1998) found an average of six lactose units attached to β-lactoglobulin after storage of powder with a water activity of 0.65 for 20 h at 50°C compared with only one when left in solution at the same temperature. In both cases, the lactosylated forms were highly heterogeneous.

Guyomarc'h *et al*. (2000) reported that the degree of lactosylation of proteins can be reduced by modifying the spray drying operating condition. A low outlet temperature, preferably <80°C, together with a relatively high inlet temperature of 170–175°C was the optimal condition for producing a low degree of lactosylation and a high drying rate.

The extent of Maillard reactions in milk products is often determined by measurement of furosine, a product of the acid hydrolysis of lactulosyl lysine formed during the analysis. The furosine content is largely influenced by the processing conditions. For pasteurised milk, furosine concentration is 4–7 mg 100 g^{-1} protein, but it is much higher in more severely heated products, such as UHT-sterilised milk; Elliott *et al*. (2005) reported an average of 183 mg 100 g^{-1} protein for 16 commercial indirectly heated UHT milks. In SMP, the concentration can be in the range of 170–600 mg 100 g^{-1} protein, depending on the drying process. However, extreme pre-heating conditions can have a significant effect. At pre-heating temperatures below 105°C, the furosine content of the powders ranges between 170 and 300 mg 100 g^{-1} protein, but at 115°C it is up to 600 mg 100 g^{-1} protein. Furosine levels increase during storage of all milk powders containing lactose with levels increasing faster with increasing water activity, up to 65%, and increasing temperature (Van Renterghem & De Block, 1996).

Evaporated milk is susceptible to Maillard reactions, which influence its colour and flavour during storage, particularly at an elevated temperature. However, the brown discolouration is more marked in sweetened condensed milk as the milk is concentrated to a higher concentration. Adding sugar before evaporation leads to faster browning than adding it after evaporation (Walstra *et al.,* 2006).

Maillard reactions are a major cause of quality deterioration of whey powders during storage as they contain a relatively high concentration of lactose and protein. During storage, browning increases over time and is more obvious at a higher temperature and lower pH. The shelf-life of whey powder can be predicted by the use of models based on the kinetics of the browning reaction and the conditions and time of storage (Dattatreya *et al.,* 2007).

Lysine and the sulphur-containing amino acids are the main amino acids that are affected by Maillard reactions during the high-temperature treatments. In dried products produced by efficient spray drying, the availability of lysine is high, ~90–97% (Rolls & Porter, 1973). Significant destruction of lysine only occurs in severely heated samples, when a loss of methionine up to 10% also occurs. Although Maillard reactions are known to be more important in milk powders during storage, Jones *et al*. (1998) speculated that the reactions are initiated during spray drying.

1.4.5 *Oxidation*

Lipid oxidation during storage is a significant issue for fat-containing powders, such as whole milk powder. The fat can react easily with oxygen in the air to produce off-flavours,

especially at higher storage temperatures (>30°C). Hydroperoxides, the primary products of lipid oxidation are colourless, tasteless and odourless, but they are transformed into a complex mixture of low-molecular-weight compounds with distinctive odour, colour and flavour characteristics. These include alkanes, alkenes, aldehydes, ketones, alcohols, acids and esters. These compounds impart off-flavours to the milk powder, and limit its shelf-life (Fenaille *et al*., 2001; see also Liang, 2000).

The rate of oxidation in milk powders increases with increasing temperature. McCluskey *et al*. (1997) reported that the rate increases 10-fold for every 10°C increase in temperature. The oxidation rate also increases when the powder particles disintegrate as the amount of fat exposed to the air increases. It is interesting to note that powders are prone to oxidation largely at extremely low moisture content because under these conditions, the powder particles disintegrate and increase the area of exposed fat (Early, 1998). Conversely, the oxidation rate decreases when the milk powder collapses as this lowers the access of the air to the fat (Thomas *et al.,* 2004).

Reducing the oxygen content in the package also reduces oxidation of milk fat. When oxygen in milk powder or infant formula packages is replaced by nitrogen and/or carbon dioxide, oxidation is significantly decreased. Whole milk powder has a shelf-life of more than 12 months if packed in cans under vacuum or with an inert gas, such as nitrogen. Milk powders stored in cans or drums for medium- to long-storage times are generally packaged in a modified atmosphere of normal air mixed with additional CO_2 and N_2, 100% N_2 or reduced O_2 atmospheres. Driscoll *et al*. (1985) investigated the sensory quality of instant and regular SMPs after 4 years' storage in cans or polybags at 10, 21 and 32°C and in atmospheres of normal air, air modified with 100% CO_2 or 100% N_2. They found that powder stored under air had a much lower sensory quality than those stored under either N_2 or CO_2 at the same temperature. Packaging techniques have also been developed for dry milk powders to eliminate or reduce O_2 and, hence, reduce fat oxidation. These include gas flushing and use of oxygen absorbers (Hotchkiss *et al*., 2006).

Addition of antioxidants can extend the shelf-life of powders. Antioxidants do not improve the quality of the product, but they maintain it by preventing oxidation of labile lipid components. Examples of antioxidants are α-tocopherol, ascorbic acid and β-carotene (McCluskey *et al*., 1997). The same authors also reported that milk from cows fed on a vitamin E-supplemented diet produced a powder that was less susceptible to oxidative deterioration.

Baldwin & Ackland (1991) found that higher pre-heat treatment increased the anti-oxidant activity of whole milk powder. Heating of milk leads to denaturation of whey proteins, especially β-lactoglobulin, thus exposing SH groups and generating SH compounds, such as hydrogen sulphide and methanethiol (Al-Attabi *et al.,* 2009), which can react with free radicals of the unsaturated fatty acids and decrease the oxidation rate. These compounds also absorb some oxygen, and also help to protect the anti-oxidative vitamins, such as ascorbic acid and vitamin E. The effect of pre-heat treatment was supported by the finding of Stapelfeldt *et al*. (1997) that low-heat milk powder had lower storage stability than medium- and high-heat powders as it was subject to severe oxidative changes. During accelerated storage at 45°C and with full contact to atmospheric oxygen, the medium- and high-heat powders were less affected than the low-heat

powder. The rate of autoxidation increased steadily as water activity increased from 0.11 to 0.33.

1.5 Conclusions

Concentrated and dried milk products represent a diverse range of dairy products. They vary considerably in chemical composition, which is determined by the composition of the original milk as well as the various heating and dehydration processes involved in their manufacture. There is also variation in the distribution of chemical components within products, for example between the surface and the interior of powder particles and between the colloidal and soluble phases, which affects the products' properties. Chemical and enzymic changes continue to occur during storage of the concentrated and dried products, which can significantly affect their functional properties and organoleptic qualities. The most important chemical changes that occur or can occur during processing and storage are denaturation of whey proteins, coagulation of caseins, lactosylation of proteins and subsequent Maillard reactions, oxidation of milk fat and crystallisation of lactose. Knowledge of the chemical components of the products, their relationship to functional properties, and the changes that can occur in these components is essential for determining the optimal production and storage conditions for these products.

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2 Current Legislation on Concentrated and Dried Milk Products

M. Hickey

2.1 Introduction

Milk has long been regarded as a nutritious food but in its raw state it is an extremely perishable product. Throughout history, mankind has been keen to preserve and concentrate the nutritional properties of milk, extending its shelf life, allowing consumption in locations well removed from the farms where milking took place and also permitting the products to be taken on journeys. For millennia, fermentation methods had been used to produce cheese and fermented milks. However, it was not until the mid-1800 that successful and cost effective processes were developed to allow concentration by heat to preserve milk.

It is generally acknowledged that Gail Borden Jr. was the inventor of the process that led to the commercialisation of condensed milk manufacture. While others had developed and indeed patented such processes, these required the addition of alkali and other ingredients, resulting in products that did not match the qualities of fresh milk and were also expensive. It is said that Gail Borden Jr. had experimented for about 10 years before he finally decided that a concentrated semi-fluid product, using evaporation under vacuum, was the best form of preservation (Hunziker, 1914). He first applied for a patent in 1853, but it was not until 3 years later that the US Patent Office appreciated the originality and value of his claim sufficiently to grant him the patent for his process (Borden, 1856).

Even then his first two factories in Connecticut were not very successful and, not until 1858, when he associated himself with Jeremiah Milbank in establishing the New York Condensed Milk Company, was he able to really exploit his invention. During the Civil War, the US government ordered large quantities for its troops, and thus created a great demand for this product. Up to the 1890s, sweetened condensed milk was the only long shelf-life format that was sold in hermetically sealed cans, while unsweetened condensed milk was manufactured and sold open, largely directly to the consumer, in a similar way as market milk. The purity and keeping quality of this unsweetened condensed milk, however, was greatly superior to market milk (Hunziker, 1914).

From that time on the industry grew with great rapidity. In the 1860s, the Anglo-Swiss Condensed Milk Company was established in Switzerland by the then US Consul at Zurich, Switzerland, Charles Page and his brother George. Their first factory was built in 1866 at Cham, Switzerland. This company prospered and grew rapidly in Europe. In the 1880s, it also built manufacturing plants in New York, Wisconsin and Illinois. In 1902, the Anglo-Swiss Condensed Milk Company sold its entire American interests, factories and business to Borden and in 1904 it joined with The Nestlé Company, of Vevay, Switzerland another

successful manufacturer of condensed milk. Both Borden and Nestlé remain major players in milk product production to this day.

The US Census Report for 1910 gave a total production of 224 438 tonnes, made up of 127 133 tonnes of unsweetened and 97 305 tonnes of sweetened condensed milk, with the unsweetened product having more than doubled from 50 000 tonnes in 1904. By 1917, Borden had more than 50 factories from Maine to Washington State as well as in Canada, and there were a total of 240 milk condensing factories across 23 US States. The largest numbers were in New York (54), Illinois (39), Wisconsin (26) and Pennsylvania (20) (Hunziker, 1914).

During the rest of the twentieth century, the relevant importance of unsweetened and sweetened condensed milks has reduced, with production estimated as approximately 4.5 million tonnes (equivalent to 11 million tonnes or 2% of total milk utilisation) in 2000; production of caseins and caseinates amounted to 450 000 tonnes utilising an equivalent amount of milk utilisation, while milk powders (skimmed and whole milk) accounted for 15% of milk utilisation (Fox, 2001).

Although there were reports of dried milk in earlier times, the commercial production of milk powders began in about 1905 (Cullip, 1966; Fox, 2001). The two processes used were roller drying and spray drying. Both processes were used to more or less the same extent until the mid-1940s, but from that time on the spray process came to predominate (Knipschildt, 1969). Instant milk powder produced by the roller dried process was used for infant formula manufacture until the 1960s because of its superior dispersibility and microbiological quality, but by 2000 milk powder production was almost all spray dried, and chocolate crumb manufacture remained the only use where roller dried milk was preferred to spray dried powder (Fox, 2001).

From the 1960s, the European Community's (EU) Common Agricultural Policy (CAP) supported the manufacture of skimmed milk powders through the operation of its intervention system, and the provision of financial aids, which encouraged its use in a number of areas. This led to large quantities of excess Community milk production being converted into skimmed milk powder. It also led to the need to develop legislation to govern the definitions of such products and their composition.

Casein had been produced commercially since the early twentieth century. Early casein production was looked on as a by-product of little economic value, used mainly for industrial non-food uses. However, research into caseins in New Zealand and Australia in the 1960s resulted in caseins being upgraded for use as food ingredients, turning them into economically valuable products (Fox, 2001). Over the years, economic policies have also played an important role in the development of casein manufacture and its use. Such policies were developed in an effort to convert surplus milk production into more functional components, initially for industrial uses but later for food uses. In this context, the European Economic Community (EEC; currently known as the *European Union* or *EU*) aid for the manufacture of casein and caseinates from skimmed milk acted as a significant stimulus for production in many Member States during the following years. At the same time, the price support programmes of United States Department of Agriculture (USDA) strongly influenced the conversion of surplus milk production in their country into non-fat dry milk. There was a dramatic increase in the import of casein into the United States, that is, during the period from late 1960s to late 1970s, and by mid-1980s about a third of

world production of sodium caseinate (70 000 tonnes) was imported into the United States. Much of this casein was used in food product manufacture. Casein and caseinate production and trade are also influenced by other government policy measures. In the United States, import levies made it attractive to convert casein to caseinate in that country rather than importing caseinate products. In 1984, the annual casein production worldwide amounted to 220 000 tonnes, most of which was manufactured in the then EEC (108 000 tonnes), New Zealand (60 000 tonnes), Poland (30 000 tonnes) and Australia (20 000 tonnes).

Whey is a by-product of cheesemaking, and more recently of casein manufacture also. For many years it was regarded as a product of little, if any, economic value, whose disposal presented significant problems. It has low total solids ($\sim$6 g 100 g^{-1}), a low protein to lactose ratio and problems of lactose crystallisation. Drying costs were higher than that for milk powders, and these costs could not be recovered in the end-product prices. Furthermore, the variations in composition due to the different sources of whey produced, made it difficult to establish a standardised product for the international market. However, since the 1970s, the variety of proteins it contains, their nutritional significance and hence its economic value have led to a change in attitude to whey (Galsmar & Bergman, 1966; Zadow, 1994).

Processes including demineralisation, reverse osmosis (RO), ultrafiltration (UF), hydrolysis, selective precipitation and chromatographic separation were developed and commercialised; these developments take advantages of the nutritional and functional properties of whey proteins and can increase their content up to 90 g 100 g^{-1} in dried whey (Kelly, 1986; Zadow, 1994). Nowadays, whey components are being used in sports drinks, protein supplements and the evolving field of nutraceuticals (which inhabit the grey area between foods and pharmaceuticals), while biologically active proteins, such as lactoperoxidase, lactoferrin and immunoglobulins, are being commercially exploited (Fox, 2001).

In the late 1800s, there had also been interest in developing formulations for feeding infants based on modifying the composition of cow's milk to make it more nutritionally equivalent to human breast milk. In 1987, the first formulated baby food was introduced by the famous German chemist, Justus von Liebig. About the same time, Henry Nestlé developed a food for babies whose mothers were unable to breastfeed. His first success was a premature infant who could not tolerate his own mother's milk or any of the usual substitutes available at that time. The first modified milk product for infant nutrition was introduced in the United States in 1915 by Gerstenberger. While a relatively small proportion of total milk production on a world scale is used for infant formulae, the economic value is very high and, in certain countries, infant formulae form an important sector of the dairy industry (Fox, 2001; Anonymous, 2008).

The origins of legislation governing food production and composition date back to the Middle Ages. Many will have heard of the 'Rheinesgebot' (literally the Purity Order), which concerned the purity of beer, and originated in Bavaria in 1516 (see http://www.brewery.org/brewery/library/ReinHeit.html or http://www3.sympatico.ca/n.rieck/docs/Reinheitsgebot.html for English translation). This listed the only permitted ingredients for beer as water, barley and hops. The original order also set the price of beer at a mere 2 Pfennig $Maß^{-1}$ but, of course, this did not last. One can notice that it did not mention yeast as an ingredient; it would be more than three centuries later before the role of micro-organisms in food fermentations was recognised. Nonetheless, it was in 1987 before the requirements

of the Rheinesgebot were fully lifted in Germany as a result of a decision of the European Court of Justice (ECJ, 1987). Nevertheless, even to this day certain German beers claim they comply with the Rheinesgebot.

By the middle of the nineteenth century, there were concerns about the adulteration, purity and wholesomeness of foods, which led to the development of food legislation. Nowadays, the bases for food legislation are given as food safety, consumer protection and fair trade. The words may differ, but the fundamentals have not really changed.

The production volumes, support schemes, uses and long history of trade in traditional concentrated and dried milk products discussed above and the significance, economic value and importance of composition of infant formula have resulted in the development of legislation in many countries addressing their designations, composition, permitted ingredients and additives, hygiene and labelling. In this chapter, the food legislation on a number of concentrated and dried milk products will be discussed, with particular reference to the legislation in the European Union (EU), the United Kingdom, Ireland, the United States, Australia, New Zealand and the international codes developed within the Codex Alimentarius. Not all products addressed in this publication shall be addressed, as specific legislation does not exist for them all.

2.2 European Union legislation

2.2.1 *Access to EU legislation*

Following its adoption, EU legislation is published in the L-Series of the *Official Journal of the European Union*, and it may also be accessed using the EUR-LEX website http://eur-lex.europa.eu/RECH_naturel.do. Use of this website is facilitated greatly by knowing the type (directive, regulation, decision or com-final), the year and the number of the relevant legislation. In this chapter, the necessary information will be given when referring to specific legislation. The most recent legislation is usually available electronically in the portable document format (PDF), but earlier legislation may be accessible in HTML format only. A copy of the original Official Journal document may be requested by e-mail. Amendments to legislation are also published in the Official Journal; however, these normally just show the text that is being changed. Consolidated texts of some legislation can be accessed electronically; they come with a warning that they are not official texts. Nonetheless, such consolidated legislation, incorporating the amendments into the original text, can be very useful as they facilitate use of the documents.

2.2.2 *Vertical – legislation on concentrated and dried milk products*

The EU has vertical legislation for the following products:

- *Preserved milks* entitled '*Certain partly or wholly dehydrated preserved milk for human consumption*'; covering milk powders, condensed/evaporated milks and sweetened condensed milks.
- *Casein and caseinates* entitled '*Certain lactoproteins (caseins and caseinates) for human consumption*'.
- Infant formulae and follow-on formulae.

Preserved milks

EU legislation under this heading covers partly and wholly dehydrated milks preserved by sterilisation, through heat treatment, by the addition of sucrose and by dehydration. The following is a list of the product categories involved:

- Unsweetened condensed high-fat milk/milk/skimmed milk/partly skimmed milk – which may also be designated as 'evaporated' milk in English.
- Sweetened condensed milk/skimmed milk/partly skimmed milk.
- Totally dehydrated high-fat milk/whole milk/semi-skimmed milk/skimmed milk (i.e. products generally known as milk powders).

In the early 1970s, the EU recognised that differences in national legislation of Member States concerning these products created barriers to free movement and created unfair competition. To facilitate the establishment of a single Community market for such products, in December 1975, it adopted Council Directive 76/118/EEC (EU, 1976) addressing the definitions, compositional requirements, minimum heat treatment equivalent to pasteurisation for the basic materials referred to in the product definitions, reserved designations, permitted additives and labelling of the relevant products. The addition of vitamins was left to national legislation. Some specific definitions of certain designations reserved in the territory of certain Member States were laid down in Article 4 – these designations included '*Evaporated Milk*' in Ireland and the United Kingdom.

With the move to horizontal legislation as proposed in the White Paper on the Completion of the Internal Market in 1985 (EU, 1985a) and the enactment of horizontal provisions on additives (EU, 1994a, b, 1995c), some discussion on the ongoing requirements for such vertical legislation took place. The consensus emerged that the existing vertical legislation should continue, with the relevant horizontal provisions being removed. Council Directive 2001/114/EC (EU, 2001) was adopted to replace the original Council Directive 76/118/ (EU, 1976), which continued to define and lay down compositional standards, denominations and rules for the same product range as heretofore. Some amendments to this directive exempted certain countries from the compulsory designations.

The partly dehydrated products are defined as liquid products in which water is partly removed from milk, skimmed milk or partly skimmed milk. Cream and/or milk powder may be added but, if milk powder is added the level shall not exceed 25 g 100 g^{-1} of the total milk solids in the end product. The quantity of sugar to be added to the sweetened products is not specified directly. However, the types of sugar to be used are listed as semi-white sugar, white sugar or extra-white sugar. An additional quantity of lactose (not greater than 0.03 g 100 g^{-1} of the finished product) is authorised for the manufacture of the sweetened condensed products.

Milk powders (i.e. wholly dehydrated milk) are solid products, in which the water is removed from milk, semi-skimmed milk or skimmed milk or cream or a mixture of these raw materials so that the end product has a maximum moisture of 5 g 100 g^{-1}.

As regards prescribed treatments and without prejudice to hygiene Regulation (EC) No. 853/2004 (EU, 2004e) on products of animal origin, the preservation of the defined products shall be achieved by the following methods:

- Heat treatment [sterilisation or ultra high temperature (UHT)] for the unsweetened condensed milks.
- Addition of sucrose for the sweetened condensed milk products.
- Dehydration for dried products.

The dried milk products are normally subjected to a pre-heating stage in their manufacture that is at least equivalent, and frequently well above the minimum pasteurisation (72°C for 15 s) requirements discussed in Section 2.2.4.

In 1999, the Codex Alimentarius allowed protein standardisation in the international standards for milk powders, evaporated milks and sweetened condensed milks; this aspect will be discussed in Section 2.7.5. As a consequence of this allowance, competitors from outside the Community producing products according to those standards had an economical advantage *vis-à-vis* the Community producers. Hence, the Community dairy industry and exporters of milk powders, evaporated milks and sweetened condensed milks requested a modification of the Community rules on the protein contents of such products. Under Council Directive 2001/114/EC (EU, 2001), the adjustment of the protein content to a standard level was not allowed. In February 2007, the Commission issued proposals to modify that situation by allowing the standardisation of the protein content in the Community in line with the Codex Alimentarius standards (minimum content of 34 g 100 g^{-1}, expressed by weight in fat-free dry matter) (EU, 2007f).

The EU Commission had identified the natural protein content in fat-free dry matter in milk powder as ranging from 31 to 37 g 100 g^{-1}, and proposed standardisation to 34 g 100 g^{-1}, in line with the Codex Alimentarius standards, recognising that this was likely to lead to substantial quantities of proteins being released in the Community market. However, given the foreseen strong demand for milk protein, they saw little, if any, consequences as regards any major additional costs, in terms of intervention, export refunds, or disposal aids. The overall higher commercial value of proteins together with improved competitive strength on the world market may even provide a higher milk price when the commercial benefits are passed on to the milk producers.

The intervention price for skimmed milk powder was then based on a protein content of 35.6 g 100 g^{-1}. With Community standardisation fixed at 34 g 100 g^{-1}, it was recognised that the intervention standard should logically be aligned to that level with the consequent adaptations of the intervention price. In early October 2007, Council Directive 2007/61/EC (EU, 2007e) amending Council Directive 2001/114/EC (EU, 2001) was adopted. Since it is a Directive, transposition into national legislation of the Member States is required, and Member States are obliged to complete this by 31 August 2008. The Directive defines the raw materials that can be used for protein adjustment; these are milk retentate, milk permeate and lactose. Table 2.1 shows the final products, their designations and corresponding compositional requirements, as contained in the amended Council Directive 2001/114/EC (EU, 2001).

Besides allowing for protein standardisation, Directive 2007/61/EC (EU, 2007e) also amended Council Directive 2001/114/EC (EU, 2001) to allow the addition of vitamins and minerals as provided for by Regulation (EC) No. 1925/2006 (EU, 2006d), and removed the article that stipulated that Member States may authorise their addition. Commission Directive 79/1067/EEC (EU, 1979) contains the first series of analytical methods of analysis for the inspection of preserved milks.

Table 2.1 Designations and corresponding compositional requirements for certain preserved milks as contained in the amended Council Directive 2001/114/EC.

Category	Product name	Particular designation (English only)[a]	Fat (g 100 g^{-1})	Total milk solids[b] (g 100 g^{-1})	Protein expressed on the fat-free dry matter basis (g 100 g^{-1})
Partly dehydrated milk					
	Condensed high-fat milk		Minimum 15	Minimum 26.5	Minimum 34
	Condensed milk		Minimum 7.5	Minimum 25	Minimum 34
		Evaporated milk	Minimum 9	Minimum 31	Minimum 34
	Condensed partly skimmed milk		Minimum 1 to <7.5	Minimum 20	Minimum 34
		Evaporated semi-skimmed milk	Minimum 4 to maximum 4.5	Minimum 28	Minimum 34
	Condensed skimmed milk		Maximum 1	Minimum 20	Minimum 34
	Sweetened condensed milk		Minimum 8	Minimum 28	Minimum 34
	Sweetened condensed partly skimmed milk		Minimum 1 to <8	Minimum 24	Minimum 34
	Sweetened condensed skimmed milk		Maximum 1	Minimum 24	Minimum 34
Totally dehydrated milk					
	Dried high-fat milk/high-fat milk powder		Minimum 42	Minimum 95[b]	Minimum 34
	Dried whole milk/whole milk powder		Minimum 26 and <42	Minimum 95[b]	Minimum 34
	Dried partly skimmed milk/partly skimmed milk powder		Minimum 1.5 and 26	Minimum 95[b]	Minimum 34

Table 2.1 Continued.

Category	Product name	Particular designation (English only)[a]	Fat (g 100 g^{-1})	Total milk solids[b] (g 100 g^{-1})	Protein expressed on the fat-free dry matter basis (g 100 g^{-1})
		Semi-skimmed milk powder/dried semi-skimmed milk	Minimum 14 to Maximum 16	Minimum 95[b]	Minimum 34
	Dried skimmed milk/skimmed milk powder		Maximum 1.5	Minimum 95[b]	Minimum 34

[a]Annex II contains a total of 26 particular designations incorporating Czech, Danish, Dutch, Estonian, Finnish, French, German, Portuguese and Spanish terms; some of these designations have different compositional requirements to those listed in footnote b.
[b]The compositional standards for dehydrated milks specify max. 5% moisture by weight in finished product.
Data compiled from EU (2001).

In addition to the horizontal labelling requirements as outlined in Directive 2000/13/EC (EU, 2000a), a number of specific labelling requirements are included in the vertical legislation:

- The percentage of milk fat must be stated on the label, except in the case of condensed skimmed milk, sweetened condensed skimmed milk and dried skimmed milk/skimmed milk powder.
- The percentage of fat-free dried milk extract (i.e. non-fat milk solids) must be stated on the label in the case of unsweetened and sweetened condensed milks.
- The specified particulars as regards milk fat and fat-free dried milk extract must appear near the product trade name.
- In the case of milk powders, the label must also outline the recommendations for reconstitution, including details of the fat content of the product thus reconstituted.
- In the case of milk powders, the label must state that the product is '*not intended as a food for infants under 12 months*'.

Member States are not permitted to adopt national provisions not provided for by the vertical Directive.

Other market support regulations make reference to and contain definitions of skimmed milk powder and buttermilk powder for the purposes of these particular regulations. For example, in Council Regulation (EC) No. 1255/1999 (EU, 1999d) it is stated in Article 7 that 'the designated intervention agency of each of the Member States are empowered to buy in at the intervention price top quality skimmed milk powder made in an approved undertaking in the Community by the spray process and obtained from cow's milk produced in the Community, during specified periods each year, which meets: (1) a minimum protein

content of 34.0 g 100 g^{-1} of the fat-free dry matter, (2) preservation requirements to be laid down and (3) conditions to be determined as regards the minimum quantity and packaging.

In addition, Commission Regulation (EC) No. 2799/1999 (EU, 1999c), as amended, which allows support to be paid on skimmed milk incorporated into animal feed, has the following definitions in Article 2:

- *Skimmed milk* – defined as milk with a fat content of no more than 1 g 100 g^{-1}, and a protein content of not less than 31.4 g 100 g^{-1} of non-fatty dry extract.
- *Skimmed milk powder* – defined as the product obtained by removing the water from milk, with a maximum fat content of 11 g 100 g^{-1}, a maximum moisture content of 5 g 100 g^{-1} and a protein content of not less than 31.4 g 100 g^{-1} of non-fatty dry extract.
- *Buttermilk* – defined as the by-product of butter manufacture obtained after churning the cream and separating the solid fat, with a maximum fat content of 1 g 100 g^{-1} and a protein content of not less than 31.4 g 100 g^{-1} of non-fatty dry extract.
- *Buttermilk powder* – defined as the product obtained by removing the water from buttermilk, with a maximum fat content of 11 g 100 g^{-1}, a maximum moisture content of 5 g 100 g^{-1} and a protein content of not less than 31.4 g 100 g^{-1} of non-fatty dry extract.

Furthermore, Article 3 of the same regulation states that for the purposes of applying this Regulation, buttermilk and buttermilk powder shall be treated as skimmed milk and skimmed milk powder, respectively. While dried skimmed milk and/or skimmed milk powder are already defined in the vertical Council Directive 2001/114/EC (EU, 2001), albeit somewhat different from the above, it is unclear whether, in the absence of definitions of buttermilk and buttermilk powder elsewhere in EU legislation, these definitions could have a wider interpretation.

Caseins and caseinates

Again the motivation for the development of vertical legislation for casein and caseinates was that differences in national legislation of Member States concerning these products created barriers to free movement and unfair competition. To facilitate the establishment of a single Community market for such products, Council Directive 83/417/EEC (EU, 1983) was adopted.

Caseins are defined as the principal protein constituent of milk, washed and dried, insoluble in water and obtained from skimmed milk by precipitation by

- the addition of acid (from among those listed in column 1 of Table 2.2) or
- microbial acidification or
- using rennet or
- using other milk-coagulating enzymes – without prejudice to the possibility of prior use of ion exchange processes and concentration.

Caseinates, on the other hand, are defined as products obtained by drying caseins treated with neutralising agents as shown in column 2, Table 2.2.

Table 2.2 Technical adjuvant permitted in caseins and caseinates (including specific acids, specific neutralising and buffering agents) in the amended Council Directive 83/417/EEC.

Acid caseins	Rennet caseins	Caseinates
Lactic acid	Rennet	Hydroxides (sodium, potassium, calcium, ammonium, magnesium)
Hydrochloric acid	Other milk-coagulating enzymes	Carbonates (sodium, potassium, calcium, ammonium, magnesium)
Sulphuric acid		Phosphates (sodium, potassium, calcium, ammonium, magnesium)
Citric acid		Citrates (sodium, potassium, calcium, ammonium, magnesium)
Acetic acid		
Orthophosphoric acid		
Whey		
Bacterial cultures producing lactic acid		

EU (1983).

Specific labelling requirements are required in the case of products marketed as mixtures:

- The words 'mixtures of ....' followed by the names of the different products, which make up the mixture, in decreasing order of weight.
- An indication of the cation or cations in the case of caseinate or caseinates (e.g. sodium, potassium and/or calcium).
- The protein content in the case of mixtures containing caseinates.

Furthermore, without prejudice to the requirements in subsequent EU legislation on hygiene and food safety, the basic materials referred to in Annexes I and II of the Directive must be subjected to heat treatment, which will render them phosphatase negative. The Directive also includes definitions and compositional requirements in Annex I for edible acid and rennet caseins, and Annex II for edible caseinates obtained from caseins of both types. Table 2.3 outlines and compares the compositional standards for each product.

Commission Directive 85/503/EEC (EU, 1985c) defines the first group of methods of analysis for verifying the composition of caseins and caseinates, and Commission Directive 86/424/EEC (EU, 1986) establishes a preliminary series of sampling procedures for the chemical analysis of these products.

Subsidies were paid on skimmed milk manufactured into casein and caseinates using Article 12 of the Council Regulation (EC) No. 1255/1999 (EU, 1999d) as a basis, and the implementing rules are set out in the Commission Regulation (EEC) No. 2921/90 (EU, 1990a). Historically, the aid is set at a level that allowed the income from the sale of skimmed milk for this purpose to correspond to that derived from the sale of skimmed milk

Table 2.3 Compositional standards (g 100 g^{-1}) for edible acid caseins, rennet caseins and caseinates in the amended Council Directive 83/417/EEC.

Parameter	Acid caseins	Rennet caseins	Caseinates
Moisture	≤10	≤10	≤8
Protein (on dry matter basis)	≥90	≥84	≥88
Protein of which minimum casein	95	95	–
Fat (on dry matter basis)	≤2.25	≤2	≤2
Acidity (as mL 0.1 N NaOH g^{-1})	≤0.27	–	–
PH	–	–	6.0 – 8.0
Ash (includes P_2O_5)	≤2.5	≥7.5	–
Lactose (anhydrous)	≤1	≤1	≤1
Sediment (burnt particles)	≤22.5 mg 25 g^{-1}	≤22.5 mg 25 g^{-1}	≤22.5 mg 25 g^{-1}
Lead	≤1 mg kg^{-1}	≤1 mg kg^{-1}	≤1 mg kg^{-1}
Extraneous matter (foreign bodies)	Nil 25 g^{-1}	Nil 25 g^{-1}	Nil 25 g^{-1}
Odour	No foreign odour	No foreign odour	Very slight foreign flavours and odours
Colour and appearance	White to creamy white; no firm lumps	White to creamy white; no firm lumps	White to creamy white; no firm lumps
Solubility	–	–	Almost entirely soluble in distilled water, except for the calcium caseinate

EU (1983).

for skimmed milk powder manufacture. Commission Regulation (EEC) No. 2921/90 (EU, 1990a) lays down the conditions of the aid as follows:

- The specified composition of the casein produced (as outlined in Annex I, Annex II or Annex III); these are quite complex and differ from those in the Council Directive 83/417/EEC (EU, 1983) and are outlined in Table 2.4 (acid casein), Table 2.5 (rennet casein) and Table 2.6 (caseinates).
- The amount of skimmed milk required to produce casein.
- Records of the amounts processed and sold, and the inspection and verification rights.

Methods of analysis for the compositional requirements, including definitions for some parameters, and sampling are addressed in Annex IV of this regulation. Over the years, the level of this subsidy has fluctuated, but has been reduced to zero ϵ since late 2006 (EU, 2006e). Discussions are taking place as to whether this regulation should be retained or abolished.

Council Regulation (EEC) No. 2204/90 (EU, 1990b) allows the use of casein in the manufacture of cheese, only if such a use can be shown to be necessary. The Annex of this regulation specifies such permitted uses – to date, the use in processed cheese

Table 2.4 Compositional standards (g 100 g^{-1}) for acid casein in Annex I and Annex II of the amended Commission Regulation (EEC) No. 2921/90.

Parameter	Annex I	Annex II
Moisture	≤12	≤10
Milk protein content, other than casein, of total protein	≤5	≤5
Fat	≤1.75	≤1.5
Free acids (expressed as lactic acid – g 100 g^{-1})	≤0.3	≤0.2
Total bacterial count	–	≤30 000 cfu g^{-1}
Coliforms	–	Absent cfu 0.1 g^{-1}
Thermophilic bacterial count	–	≤5000 cfu g^{-1}

cfu = colony-forming units.
EU (1990a).

to a maximum level of 5 g 100 g^{-1} is the only such use permitted. Since the casein manufacturing subsidy has been eliminated for all practical purposes, there have also been discussions as to whether this Regulation is still necessary. At this time no decision has been taken, however, its fate is probably linked to the outcome of the discussions on the manufacturing subsidy.

Infant formulae and follow-on formulae

EU legislation on infant formulae and follow-on formulae are addressed in the context of Foodstuffs for Particular Nutritional Uses (PARNUTS). Once again, the White Paper on the Completion of the Internal Market indicated the need for full harmonisation of legislation in this area. Council Directive 77/94/EEC (EU, 1977) had already attempted approximation of such foods, but problems remained in relation to free movement of such

Table 2.5 Compositional standards (g 100 g^{-1}) for rennet casein in Annex I and Annex II of the amended Commission Regulation (EEC) No. 2921/90.

Parameter	Annex I	Annex II
Moisture	≤12	≤8
Milk protein content, other than casein, of total protein	≤5	≤5
Fat	≤1	≤1
Ash	≥7.5	≥7.5
Total bacterial count	–	≤30 000 cfu g^{-1}
Coliforms	–	Absent cfu 0.1 g^{-1}
Thermophilic bacterial count	–	≤5000 g^{-1}

cfu = colony-forming units.
EU (1990a).

Table 2.6 Compositional standards (g 100 g^{-1}) for caseinates in Annex I, Annex II and Annex III of the amended Commission Regulation (EEC) No. 2921/90.

Parameter	Annex I	Annex II	Annex III
Moisture	≤6	≤6	≤6
Milk protein content, other than casein, of total protein	≤5	≤5	≤17
Milk protein substances	≥88		≥88
Fat	–	–	≤1.5
Fat and ash content	≤6	≤6.00	–
Ash	–	–	≤6.5
Lactose			≤1.00
Total bacterial count	–	≤30 000 cfu g^{-1}	≤30 000 cfu g^{-1}
Coliforms	–	Absent cfu 0.1 g^{-1}	Absent cfu 0.1 g^{-1}
Thermophilic bacterial count	–	≤5000 cfu g^{-1}	≤5000 cfu g^{-1}

cfu = colony forming units.
EU (1990a).

products, and Council Directive 89/398/EEC (EU, 1989b) was adopted, which repealed and replaced the earlier directive and established the following common definition for such foods: '*Foodstuffs for particular nutritional uses are foodstuffs which, owing to their special composition or manufacturing process, are clearly distinguishable from foodstuffs for normal consumption, which are suitable for their claimed nutritional purposes and which are marketed in such a way as to indicate such suitability*' (see also EU, 1992b).

Within the framework of the Council Directive 89/398/EEC (EU, 1989b), infant and follow-on formulae were addressed in the Commission Directive 91/321/EEC (EU, 1991a), which was adopted in May 1991 and laid down very detailed compositional and labelling requirements for the products within its scope. The following were the definitions outlined by the Commission:

- Infants as children under 12 months of age.
- Young children as children from 1 to 3 years of age.
- Infant formulae as products '*intended for particular nutritional use by infants during the first four to six months of life and satisfying by themselves the nutritional requirements of this category of persons*'.
- Follow-on formulae as foodstuffs '*intended for particular nutritional use by infants aged over four months and constituting the principal liquid element in a progressively diversified diet of this category of persons*'.

The protein sources and other food ingredients, the suitability of which had been established as suitable for infants and young children by scientific data, were specified in the Annexes. In addition, Commission Directive 91/321/EEC (EU, 1991a) was amended a number of times and, in 2005, the Commission published a proposal to amend (or recast)

the original directive (EU, 2004h). This was based on consultations with experts from Member States, the latest advice from the Scientific Committee for Foods (SCF) and discussions at international level, particularly the ongoing work within the Codex Committee for Nutrition and Foods for Special Dietary Uses (CCNFSDU). This led to the adoption of a new Commission Directive 2006/141/EC (EU, 2006a), which amended and replaced the earlier directive. This new Directive retained definitions of infant, young child, infant formula and follow-on formula that were little changed from the original directive, but there were substantial changes in the Annexes.

The detailed compositional, ingredient and other specifications of the Commission Directive 2006/141/EC (EU, 2006a) are addressed in Chapter 9 entitled *Infant Formulae – Powders and Liquids*. There is one point of difference that is worthy of note in this chapter; it relates to the nitrogen conversion factor (NCF) used to convert nitrogen (N) to protein for the purposes of achieving the minimum and maximum levels of protein set down in Annex I.2 and Annex II.2 of the Commission Directive 91/321/EEC (EU, 1991a) and the Commission Directive 2006/141/EC (EU, 2006a). However, Commission Directive 91/321/EEC (EU, 1991a) used NCF 6.38 for cow's milk proteins and 6.25 for soya protein isolates and partial protein hydrolysates, while in the Commission Directive 2006/141/EC (EU, 2006a) NCF 6.25 was used for all permitted protein sources. This change was based on one of the recommendations in the Report of the Scientific Committee on Food on the Revision of Essential Requirements of Infant Formulae and Follow-on Formulae of April 2003 (SCF, 2003). This caused some concern in the dairy sector throughout the world, as similar discussions were taking place within the relevant Codex Alimentarius Committees at that time, and appeared to cast doubt on the long established NCF of 6.38 used for milk and milk-based products. In 2004, the CCNFSDU asked the European Society for Paediatric Gastroenterology, Hepatology and Nutrition (ESPGHAN) Committee to carry out consultations with the international scientific community to provide a proposal on this matter. A resulting paper in 2005 (Koletzko *et al*., 2005) recommended NCF 6.25 for all infant formulae and outlined, as the basis for this, that the proteins obtained from cow's milk used in current infant formulae were modified in composition, and thus that NCF 6.38 was not justified for all the milk-derived protein present. The authors recognised that individual NCFs for each individual protein source would be better, but that this was not feasible in practice. Furthermore, the recommended levels of minimum and maximum protein contents, in the draft standard under discussion, were based on the NCF 6.25. At the same time they accepted the use of NCF 6.25 as used in Codex Alimentarius Guidelines for Nutritional Labelling and NCF 6.38 for unmodified cow's milk protein and whole cow's milk in other food products. This went some way in alleviating the concerns of the dairy sector, both at the European level and worldwide.

2.2.3 *Horizontal – hygiene and food safety requirements*

The early legislative work in the EU was largely taken up with market regulation, and it was not until 1985 that the first Community hygiene measure for milk was adopted; this was Council Directive 85/397 (EU, 1985b). This initiated a process of harmonising hygiene standards within the Community in order to facilitate intra-Community trade without compromising the existing Member State hygiene rules. It covered all aspects of the production, transport and processing of milk from the farm to the final consumer.

This was followed in 1992 by a new milk hygiene Council Directive 92/46/EEC (EU, 1992a) that became effective from 1 January 1994. This Directive contained animal health requirements for raw milk, hygiene requirements for registered holdings, hygiene requirements in milking, collection and transport of milk to collection centres, standardisation centres, treatment establishments and processing establishments. For the first time, uniform EU-wide hygiene standards were created, as the earlier Directive 85/397 (EU, 1985b) applied to intra-Community trade only. Council Directive 92/46/EEC (EU, 1992a) laid down minimum compositional standards for milk, and also standards for the maximum plate count and somatic cell count for raw milk at collection from dairy farms intended for the production of certain milk-based products, including those covered in this chapter.

A major review was carried out on the EU Hygiene Directives, following a recommendation in the EU White Paper on Food Safety (EU, 2000b). Prior to this review, there were a total of 16 commodity-specific EU Directives and one Directive on general food law, which had been gradually developed in the period from 1964 to 2000, and had given a high level of protection to the consumer. However, they were comprised of a mixture of different disciplines (hygiene, animal health and official controls), and were detailed and complex. It was decided to overhaul the legislation to improve, simplify and modernise it, and separate aspects of food hygiene from animal health and food control issues. The review aimed for a more consistent and clear approach throughout the food production chain from 'farm to fork'.

A package of new hygiene rules was adopted in April 2004 by the European Parliament and the Council. They became applicable from 1 January 2006 and, in the case of milk and milk products, replaced the Council Directive 92/46/EEC (EU, 1992a). The new rules are Regulations and not Directives, making them binding in Member States without the necessity of national legislation to be enacted to implement their provisions. Instead of all the hygiene requirements being embodied in a single piece of legislation, the hygiene requirements for the dairy sector are now contained across at least six different regulations. The three main Regulations are (1) Regulation (EC) No. 852/2004 (EU, 2004d) on the hygiene of foodstuffs, (2) Regulation (EC) No. 853/2004 (EU, 2004e) laying down specific hygiene rules for food of animal origin; Annex III Section XI thereof contains specific requirements for raw milk and dairy products and (3) Regulation (EC) No. 854/2004 (EU, 2004f) laying down specific rules for the organisation of official controls on products of animal origin intended for human consumption.

In early December 2005, two important additional regulations were published – Commission Regulation (EC) No. 2074/2005 (EU, 2005b) and Commission Regulation (EC) No. 2076/2005 (EU, 2005c). In addition to laying down implementing measures and transitional measures, they also contain important amendments and derogations to the original regulations. Furthermore, in 2006, another Commission Regulation (EC) No. 1662/2006 (EU, 2006b) was published, amending Regulation (EC) No. 853/2004 (EU, 2004e), which contained a replacement to the Complete Section XI of the same Regulation. Fortunately, a consolidated version of Regulation (EC) No. 853/2004 (EU, 2004e), incorporating all the amendments up to 17 November 2007, is available on the EUR-LEX website. Microbiological criteria for foodstuffs are laid down in the Commission Regulation (EC) No. 2073/2005 (EU, 2005a).

Though largely self-contained, within these regulations, there is also significant cross-referencing to each other and to other legislation that affect the health, food safety and hygiene requirements of milk and milk product production. These include the following:

- Directive 2000/13/EC (EU, 2000a), as amended, relating to the labelling, presentation and advertising of foodstuffs.
- Council Regulation (EEC) No. 2377/90 (EU, 1990c) establishing maximum residue limits of veterinary medicinal products in foodstuffs of animal origin.
- The framework of food safety Regulation (EC) No. 178/2002 (EU, 2002a).
- Council Directive 97/78/EC (EU, 1998a) relating to veterinary checks on imports from third countries.
- Council Directive 2002/99/EC (EU, 2002b) relating to the animal health rules governing the production.
- Council Directive 96/23/EC (EU, 1996b) on measures to monitor certain substances and residues in live animals and animal products.
- Council Directives 64/432/EEC (EU, 1964) and 91/68/EEC (EU, 1991b) on animal health problems affecting intra-Community trade in bovine animals and swine.
- Council Directive 98/83/EC (EU, 1998b) as amended, on the quality of potable water.

The new hygiene regulations adopt an approach based more on outcomes, or on the Food Safety Objectives (FSOs) and hazard appraisal (analysis) critical control points (HACCP) system, than on the detailed production, milking, and process requirements of the earlier Directive. This approach may be illustrated by reference to heat treatments such as pasteurisation. The Council Directive 92/46/EEC (EU, 1992a) specified a minimum time/temperature requirement – '*. . .at least 71.7°C for 15 seconds (or any equivalent combination) or a pasteurising process using different time/temperature combinations to obtain an equivalent effect'*.

In Regulation (EC) No. 853/2004 (EU, 2004e), heat treatment of dairy products was cross-referenced to Regulation (EC) No. 852/2004 (EU, 2004d) Chapter XI and therein states, *inter alia*, in addressing heat-treated products in hermetically sealed containers '*. . .to ensure that the process employed achieves the desired objectives, food business operators are to check regularly the main relevant parameters (particularly temperature, pressure, sealing and microbiology), including by the use of automatic devices; and that the process used should conform to an internationally recognised standard (for example, pasteurisation, ultra high temperature or sterilisation . . . .)*'. Regulation (EC) No. 853/2004 (EU, 2004e) added a further requirement that, when considering whether to subject raw milk to heat treatment, food business operators (FBOs) must have regard to the HACCP system and comply with requirements imposed by the competent authority when approving the establishment or in carrying out checks under Regulation (EC) No. 854/2004 (EU, 2004f).

However, Commission Regulation (EC) No. 2074/2005 (EU, 2005b) reintroduced prescriptive time/temperature requirements for pasteurisation and UHT treatment, by way of amendment (using additional wording) of the relevant section of Regulation (EC) No. 853/2004 (EU, 2004e) (i.e. Section IX, II (II) 1). The reasons for the reintroduction of the specific time/temperature requirements are not outlined, but it may have been due to pressures for legal certainty regarding enforcement. Furthermore, it may be noted that

the high-temperature short-time (HTST) process has been increased from 71.7°C in the Council Directive 92/46/EEC (EU, 1992a) to 72°C in the Commission Regulation (EC) No. 2074/2005 (EU, 2005b). Why was this done? The reason is not stated in the legislation, but the new definition is in line with the Codex definition of pasteurisation in the Code of Hygiene Practice for Milk and Milk Products (CAC/RCP 57-2004) (FAO/WHO, 2004), and also ensures that the European definition meets the recommendations for the control of foot-and-mouth disease contained in the Article 3.6.2.5 of the Terrestrial Animal Health Code of the Office International des Epizooties (OIE), the World Organisation for Animal Health (OIE, 2007) (see also http://www.oie.int/eng/normes/mcode/en_sommaire.htm).

Some other specific requirements are also retained. An example in Regulation (EC) No. 853/2004 (EU, 2004e) is the requirement to ensure that '*During transport the cold chain must be maintained and, on arrival at the establishment of destination, the temperature of the milk must not be more than 10°C*'. In fact, this requirement is stricter in the new regulation, as the derogation in the earlier Directive regarding milk collected within 2 h of milking is removed. In addition, Chapter III of Regulation (EC) No. 852/2004 (EU, 2004d) encourages the development, dissemination and use of national or sectoral and Community Guides to Good Practice. Some key requirements from the main regulations follow.

Regulation (EC) No. 852/2004 (EU, 2004d) that sets down general hygiene requirements states the following in Article 1:

- The main responsibility for food safety rests with the FBO.
- Food safety should be ensured throughout the food chain starting with primary production.
- The chill chain must be maintained for foods that cannot be stored at ambient temperatures.
- The FBO responsibility should be reinforced by the implementation of procedures based on HACCP and on the application of good hygiene practice.
- Guides to good practice are valuable to aid FBO's compliance with food hygiene and application of HACCP system.
- It is necessary to establish microbiological criteria and temperature control requirements based on scientific risk assessment.
- It is necessary to ensure food imports are at least of the same or equivalent hygiene standard to that produced in the EU.

Specific hygiene and animal health requirements for raw milk and dairy products are laid down in Annex III, Section IX of Regulation (EC) No. 853/2004 (EU, 2004e); *they cover the following:*

- Animal health requirements.
- Hygiene on milk production holdings, including premises, equipment, milking, collection transport and staff.
- Criteria for raw milk.
- Requirements for dairy products, including temperature, heat treatment requirements, raw milk prior to processing and labelling.
- Identification marking – this replaces the health mark requirement contained in the Council Directive 92/46/EEC (EU, 1992a) (Fig. 2.1).

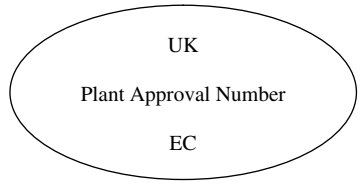

Fig. 2.1 Format of identification mark as required for milk and milk products under EU Regulation 853/2004. After EU (2004).

Table 2.7 outlines the main criteria for raw milk for the manufacture of all milk products. The main attention is drawn to the requirements of Annex II Section IX, Chapter II.III.1 (a). By way of derogation from the specific plate count requirements therein, relating to criteria for raw cow's milk, Article 12 of the Commission Regulation (EC) No. 2076/2005 (EU, 2005c) states that the maximum plate count for raw cow's milk shall apply only where such milk is to be heat treated, and has not been so treated within the period of acceptance specified in the HACCP-based procedures put in place by FBO. However, the latter regulation is a transitional measure and is set to end on 31 December 2009. After that time, unless replaced by a further derogation, the plate count requirements will apply as in Regulation (EC) No. 853/2004 (EU, 2004e), as amended. Guidance documents have also been developed by the Commission on Regulation (EC) No. 852/2004 (EU, 2004d) (see also EU, 2005e, f, 2006c) and Regulation (EC) No. 853/2004 (EU, 2004e) (see also EU, 2005d).

Microbiological criteria for milk powder, whey powder and infant formulae are laid down in the Commission Regulation (EC) No. 2073/2005 (EU, 2005a) and are outlined in Tables 2.8 and 2.9

2.2.4 *Horizontal – food additives legislation*

From the 1960s through to the mid-1970s, the EU established a series of basic directives addressing the use of colours, preservatives, antioxidants, emulsifiers, stabilisers and thickeners. Amendments were made to these over the years. Specific additive provisions were included in vertical legislation, and in other cases authorisation for their use was left to Member States. Inevitably, this led to differences between the legislative provisions of Member States and thus hindered the free movement of foodstuffs within the open market; therefore harmonisation of this area became a major priority. With the move to horizontal legislation, as proposed in the White Paper on the Completion of the Internal Market in 1985 (EU, 1985a), moves were initiated to address additives in a horizontal and more comprehensive manner. First, the use of additives in foods throughout the EU was addressed under the additive framework of the Council Directive 89/107/EEC (EU, 1989a) and second, the flavourings mentioned in the Council Directive 88/388/EEC (EU, 1988); however, in 1994 and 1995, specific additive directives were adopted addressing (1) colours (European Parliament and Council Directive 94/36/EC – EU, 1994b), (2) sweeteners (European Parliament and Council Directive 94/35/EC – EU, 1994a) as amended and (3) additives other than colours and sweeteners (European Parliament and Council Directive 95/2/EC – EU, 1995c), as amended.

Table 2.7 Criteria for raw milk supply in EU legislation.

Parameter	Requirement	Qualification
Plate count cfu mL^{-1}) at 30°C	≤100 000	Based on rolling geometric average over a 2-month period with at least two samples per month
SCC	≤400 000	Based on rolling geometric average over a 3-month period with at least one sample per month, unless the competent authority specifies another methodology to take account of seasonal variations in production levels
Antibiotic residues	Below levels specified in EU (1990b) or combined total of residues does not exceed any maximum permitted value	
Temperature during storage on farm	Immediately cooled to ≤8°C in case of daily collection or ≤6°C if collection is not daily	FBO need not comply with the requirement if, either, milk is to be processed within 2h of milking, or a higher temperature is necessary for technological reasons related to the manufacture of certain dairy products and the competent authority so authorises
Temperature during transport	Chill chain must be maintained and on arrival at destination ≤10°C	FBO need not comply with the requirement if, either, milk is to be processed within 2h of milking, or a higher temperature is necessary for technological reasons related to the manufacture of certain dairy products and the competent authority so authorises
Temperature during storage prior to processing	Upon acceptance at a processing establishment, quickly cooled to ≤6°C and kept at that temperature until processing	FBO need not comply with the requirement if processing begins immediately after milking or within 4h of acceptance at a processing establishment, or the competent authority authorises a higher temperature for technological reasons concerning the manufacture of certain dairy products
Plate count (cfu mL^{-1}) at 30°C immediately before processing	≤300 000	When milk fails to meet this criterion the food business operator must inform the competent authority and take measures to correct the situation (A transitional derogation applies under Commission Regulation (EC) No. 2076/2005 until 31/12/2009.)

cfu = colony-forming units; SCC = somatic cell count; FBO = food business operator.
Data compiled from EU (2004e, 2005c).

Furthermore, food additives must at all times comply with the approved criteria of purity. These criteria are outlined in three Commission Directives (as amended):

- Sweeteners by the Commission Directive 95/31/EC (EU, 1995a).
- Colours by the Commission Directive 95/45/EC (EU, 1995b).
- Additives other than colours and sweeteners by the Commission Directive 96/77/EC (EU, 1996a).

While these directives met the requirements of harmonising legislation in the EU and covered a more comprehensive list of additives than heretofore, they are not necessarily

Table 2.8 Microbiological criteria for milk powder and whey powder in EU legislation; the limits do not apply to products intended for further processing in the food industry.

	Sampling	Plan	Limits	
Micro-organisms	n^a	c^a	m^a	M^a
Enterobacteriaceae	5	0	10 cfu g^{-1}	10 cfu g^{-1}
Coagulase-positive *staphylococci*	5	2	10 cfu g^{-1}	100 cfu g^{-1}

cfu = colony-forming units.
[a]n = number of units comprising the sample; c = number of sample units between m and M; m = the acceptable microbiological level in a sample unit and M = maximum level for any sample unit which, when exceeded in one or more samples, would cause the lot to be rejected. However, in the case of coagulase-positive *Staphylococcus* spp. if values $>10^5$ cfu g^{-1} are detected, the batch has to be tested for staphylococcal enterotoxin.
Data compiled from EU (2005a).

easy to address within the scope of this chapter. One major difficulty is that the EU does not have the equivalent of the Codex Food Category System (FCS); to be discussed later. Consequently, the appendices of the directives contain references to foodstuffs that are not defined (or categorised) at Community level. Some references are specific and clear, for example partially dehydrated and dehydrated milk as defined in the Council Directive 76/118/EEC (EU, 1976); some name particular but undefined products (e.g. *polenta*); but others are either very general (e.g. dried powdered foodstuffs) or not clear (e.g. fine bakery wares). Within these constraints it may be worth looking at each of the specific additive directives.

The framework on additive by the Council Directive 89/107/EEC (EU, 1989a) has the following definitions:

- An additive is '*any substance not normally consumed as a food in itself and not normally used as a characteristic ingredient of food whether or not it has nutritive value, the intentional addition of which to food for a technological purpose in the manufacture, processing, preparation, treatment, packaging, transport or storage of such food results, or may be reasonably expected to result, in it or its by-products becoming directly or indirectly a component of such foods*' (Article 1.2).

Table 2.9 Microbiological criteria for dried infant formulae and dried dietary foods for special medical purposes intended for infants below 6 months of age in EU legislation; the limits do not apply to products intended for further processing in the food industry.

	Sampling	Plan	Limits	
Micro-organisms	n^a	c^a	m^a	M^a
Enterobacteriaceae	10	0	Absent cfu 10 g^{-1}	Absent cfu 10 g^{-1}

[a]n = number of units comprising the sample; c = number of sample units between m and M; m = the acceptable microbiological level in a sample unit and M = maximum level for any sample unit which, when exceeded in one or more samples, would cause the lot to be rejected. If Enterobacteriaceae are detected in any sample unit, the batch has to be tested for *Enterobacter sakazakii* and *Salmonella* spp.
Data compiled from EU (2005a).

- A processing aid is '*any substance not consumed as a food ingredient by itself, intentionally used in the processing of raw materials, foods or their ingredients, to fulfil a certain technological purpose during treatment or processing and which may result in the unintentional but technically unavoidable presence of residues of the substance or its derivatives in the final product, provided that these residues do not present any health risk and do not have any technological effect on the finished product*' (footnote to Article 1.3 (a).

This Directive excludes flavourings and substances added as nutrients, such as vitamins, minerals and trace elements, from the scope of the additives governed by its provisions. Annex II lays down three basic principles for the approval of use of additives, which may be summarised as follows:

- A technological need can be demonstrated and that need cannot be achieved by other means that are economical or technologically practical.
- Their use does not present a hazard to human health at the levels of use proposed based on the scientific evidence available.
- Their use does not mislead consumers.

Annex I of the directive lists 25 additive functional categories (Table 2.10); the definitions of these categories are given in the specific directives on colours, sweeteners and additives other than colours and sweeteners.

The colours covered by the European Parliament and Council Directive 94/36/EEC (EU, 1994b) have five Annexes as follows:

- Annex I contains a list of the 43 permitted food colours.
- Annex II lists foods that may not contain added colours except where specifically provided for in Annexes III, IV or V – this list includes preserved milks as mentioned in the Council Directive 76/118/EEC (EU, 1976), cream powder and foods for infants and young children (including infant and follow-on formulae).

Table 2.10 The 25 additive[a] functional classes listed in the EU framework additive of the amended Council Directive 89/107/EEC.

Colour	Flavour enhancer	Glazing agent
Preservative	Acid	Flour treatment agent
Anti-oxidant	Acidity regulator	Firming agent
Emulsifier	Anti-caking agent	Humectants
Emulsifying salt	Modified starch	Sequestrant
Thickener	Sweetener	Enzyme
Gelling agent	Raising agent	Bulking agent
Stabiliser	Anti-foaming agent	Propellant gas and packaging gas

[a]Two additional additive functional classes (carriers and foaming agents) are defined in the amended Commission Directive No. 95/2/EC (EU, 1995c).
Data compiled from EU (1989a).

- Annex III has a list of foods to which only certain colours may be added – none of the products in this chapter are listed therein.
- Annex IV lists 10 colours that are permitted for certain uses only – the list included E 160b (annatto, bixin, norbixin); no products covered directly in this chapter are listed as approved uses for the colours concerned.
- Annex V lists colours permitted in foods other than those named in Annexes II and III; it is divided into two parts.
- Annex V Part 1 lists 15 colours that are allowed in foods mentioned in Part 2 and in all other foods, at *quantum satis*, other than those listed in Annexes II and III.
- Annex V Part 2 lists 18 other colours that may be used singly or in combination up to levels specified in the accompanying table but for 6 of the specified foods the maximum level of each of 4 of the colours is set at 50 $mg\,kg^{-1}$.
- None of the products covered by this chapter is specifically mentioned in Annex V.

Based on the above, the following conclusions may be made as regards the use of colours in the products within the scope of this chapter:

- Colours are not allowed in infant and follow-on formulae; this is as might be anticipated.
- Since preserved milks and cream powders are listed in Annex II, they are not mentioned in Annex III, IV or V.
- The other products within the scope of this chapter, such as casein and caseinates, whey powders, whey protein concentrates, lactose and dried blends of milk powder and vegetable oil (fat filled milks), are not mentioned in Annex II. Further, none of the products are listed in Annex IV; thus, at least in theory, they can use the 15 additives listed in Annex V Part 1, but not those in Part 2 of that annex. Any use would be governed by the basic principles in the framework directive as discussed above.
- It should be noted that annatto (E160b) is listed in Annex IV. Whey powders and whey protein concentrates are not specifically mentioned in this Annex, so annatto cannot be added during manufacture; however, these products can be manufactured from whey derived from cheeses that are permitted to contain annatto colouring and thus may contain annatto based on the 'carry over' principle.

The European Parliament and Council Directive 95/2/EEC (EU, 1995c) on additives other than colours and sweeteners is quite long, complex and detailed. Its provisions may be summarised as follows:

- The latest consolidated text contains definitions of 24 additive functions [Article 1.3 (a)–(w) and Article 1.4]. Included are definitions of two functions, carriers and foaming agents, not listed in Annex I of the framework of the Directive. Additives are listed in the Annexes without specified functions. It is up to food manufacturers to assign the principal or main additive function to each additive in product labelling, recognising that an additive may have more than one function in a food.
- It is stated that casein and caseinates are not additives [Article 1.5 (h)]. This is because otherwise these products could, at least in certain uses, be considered to meet the definition of an additive as given above.

- Food additives listed in Annex I are permitted in foods in general, for one or more of the additive functions as defined in Articles 1.3 and 1.4, except of those foodstuffs listed in Annex II and Article 2.3 following the '*quantum satis*' principle (Article 2.2). However, it should be noted that Annex II has specific provisions for partially dehydrated and dehydrated milks as defined in the Council Directive 76/118/EEC (EU, 1976).
- The '*quantum satis*' principle is often misunderstood as implying that one may use as much as one likes, however, this term is defined in Article 2.8 as meaning '*that no maximum level is specified. However, additives shall be used in accordance with good manufacturing practice, at a level not higher than is necessary to achieve the intended purpose and provided that they do not mislead the consumer*'.
- Article 2.3 lists foods for which the permission to use the additives listed in Annex I does not apply unless specifically allowed for. This article lists foods of infants and young children (which includes infant and follow-on formulae), but not other products within the scope of this chapter.
- Annex I lists 114 E numbers and specific food additives that are generally permitted for use in foods not referred to in Article 2.3 or Annex II, with the exceptions referred to above regarding preserved milks, infant and follow-on formulae, most products within the scope of this chapter would be permitted, at least in theory, to use additives where their use meets the basic principles outlined in the discussion on the Council Directive 89/107/EEC (EU, 1989a) mentioned above.
- Annex II lists foods in which a limited number of Annex I additives may be used. As mentioned above, this list includes specific provisions for *partially dehydrated and dehydrated milks* as defined in the Council Directive 76/118/EEC (EU, 1976), where just 10 additives are permitted – ascorbic acid (E300), two ascorbates (E301, E304), lecithins (E322), two citrates (E331, E332), carrageenan (E407), two bicarbonates (E500ii, E501ii) and calcium chloride (E509).
- Annex III addresses conditionally permitted preservatives and antioxidants. It has four parts; Part A deals with sorbates, benzoates and p-hydroxybenzoates, Part B addresses sulphur dioxide and sulphites; Part C other preservatives and Part D other antioxidants. Of all the additives listed in this Annex, the only ones of relevance to the products of interest here are the antioxidants, gallates (E310, E311, E312), Tertiary-butyl hydroquinone (TBHQ) (E319), Butylated hydroxyanisole (BHA) (E320) and Butylated hydroxytoluene (BHT) (E321), permitted for use in milk powder for vending purposes and oils and fats for the professional manufacture of heat-treated foodstuffs (e.g. as might be used in dried blends of milk powder and vegetable oil). In each case maximum levels are specified.
- Annex IV deals with 'other permitted additives', and is the most complicated of the annexes to comprehend. It is often best to look at each additive of interest and to see whether it is permitted for a particular product of interest. Taking the example of phosphates, they are addressed as a group and the specified maximum levels apply to their use singly or in combination, expressed as phosphate P_2O_5. Note, the formula for the ion is $(PO_4)_n$ and, for the products of interest in this chapter, the following provisions apply: (1) for dried milk and dried skimmed milk a maximum level of 2.5 g kg^{-1}, (2) for partly dehydrated milk with less than 28 g 100 g^{-1} solids, a maximum level of 1 g kg^{-1}, (3) for partly dehydrated milk with more than 28 g 100 g^{-1} solids, a

maximum level of 1.5 g kg^{-1} and (4) for dried powdered foodstuffs, a maximum level of 10 g kg^{-1}.

From the above it could reasonably be assumed that the use of phosphates is allowed in all the relevant products, apart from infant and follow-on formulae addressed in Annex VI, with the level varying depending on the heading under which the product falls. This conclusion is predicated by the assumption that their use would meet the requirements of the basic principles for the use of any food additive as discussed earlier.

- Annex V addresses permitted carriers and carrier solvents and shall not be discussed further.
- Annex VI deals with additives permitted for use in foods for infants and young children. It is divided into four parts; Part 1 addresses additives permitted for use in infant formulae for infants in good health (a total of just 17), Part 2 deals with additives permitted for use in follow-on formulae for infants in good health (a total of 20) and Parts 3 and 4 deal with additives permitted in processed cereal-based foods and baby foods for infants and young children in good health, and in dietary foods for infants and young children for special medical purposes, respectively, that are outside the scope of this chapter.

As may be expected, the sweeteners section in the European Parliament and Council Directive 94/35/EC (EU, 1994a) addresses the use of 15 artificial sweeteners permitted in specified foods. Sugar- and energy-reduced products are the main uses, with a focus on desserts and similar products, confectionery, non-alcoholic drinks and beers, food supplements in syrup or chewable form, foods for use in energy restricted diets and certain dietary foods for special medical purposes. Sweeteners may not be used in food for infants and young children.

The framework on flavourings of the Council Directive 88/388/EEC (EU, 1988) contains general purity criteria, definitions, principles applicable to the use of flavourings in foods, requirements for labelling of flavours and maximum levels for undesirable substances that may be present in flavourings. However, flavourings are substances used to give taste and/or smell to food and the directive defines different categories of flavourings as follows:

- Flavouring substances (natural, natural-identical or artificial flavouring substances)
- Flavouring preparations (of plant or animal origin)
- Process flavourings (which evolve flavour after heating)
- Smoke flavourings (derived from smoke extracts and condensates)

With respect to flavouring substances, Regulation (EC) 2232/96 (EU, 1996c), as amended, sets out the basic rules for the use of these substances in or on foodstuffs in the EU. In the compilation of this Regulation, Member States informed the Commission of those flavouring substances that were authorised for use in foodstuffs at their national level, at that time. This information was compiled into a Register of about 2800 substances and adopted as Commission Decision 1999/217/EC (EU, 1999a), as amended. Among the flavourings listed in the register there are many substances that occur naturally

in animal and vegetable products, as well as artificial flavouring substances. These 2800 flavourings were submitted to the European Food Safety Agency (EFSA) who divided them into 48 groups for evaluation by their Panel on additives, flavourings, processing aids and materials in contact with food (AFC). It is intended that only flavourings listed in a positive list, so compiled, will be added to foods. EFSA aims to complete the evaluations of all the substances in the Register, for which adequate data have been received, by April 2008.

If a food contains flavourings, the word '*flavouring*' must be present in the ingredient list on the packaging. The expression 'natural flavouring' may be used only for substances or preparations, which use flavourings extracted from vegetable or animal materials. For flavourings sold to food processors and consumers, labelling is required concerning their minimum durability, conditions for storage and use, identification of the producer and identification of other substances contained in the flavourings (e.g. additives).

Forthcoming changes as regards to additives

In 2006, the European Commission adopted a package of legislative proposals that aimed to upgrade rules for additives (EU, 2007c), flavourings (EU, 2007b) and to introduce harmonised legislation on food enzymes (EU, 2007d). It also proposed the creation of a common authorisation procedure for food additives, flavourings and enzymes, based on scientific opinions from the EFSA (EU, 2007a). Three of these proposals were adopted as Common Positions, and were published as three separate communications in early March 2008; they may be accessed on the EUR-LEX website as follows:

- for additives – EU, 2008b;
- for enzymes – EU, 2008c;
- for the common authorisation procedure – EU, 2008a.

The next step is for these proposals to be debated at the European Parliament.

2.2.5 *Horizontal – labelling requirements for foods*

As mentioned elsewhere, some products within the scope of this chapter are governed by vertical legislation, such as preserved milks, caseins and caseinates and infant and follow-on formulae. The relevant legislation for these products contains some specific labelling requirements as regards use of product designations, composition, reconstitution and additional information.

Horizontal European labelling requirements for foods are contained in the EU Labelling Directive 2000/13/EC (EU, 2000a), as amended. It should be noted that the scope of this directive applies to the labelling of foodstuffs to be delivered as such to the ultimate consumer, or to mass caterers (defined as restaurants, hospitals, canteens and other similar mass caterers). Hence, they may not necessarily apply directly to the products covered by this chapter, in so far as the products may be intended for further manufacture. In such instances, the products are normally traded to meet detailed specifications between the purchaser and vendor. Compliance with such specifications, especially when written

and signed by both parties, would be governed by contract law. Nonetheless, it is worth considering the horizontal food labelling requirements, which include the following provisions:

- *Name of the food* – a hierarchy exists as regards the names/designations used: (1) if the product has a legal name specified in EU legislation, then that name should be used; (2) where there is no EU legal name, the name under which a product is sold shall be the name provided for in the legislation and administrative provisions applicable in the Member State where the product is sold to the final consumer or to mass caterers; (3) where neither of the above provisions apply, the name under which a product is sold shall be the name customary in the Member State where it is sold to the final consumer or to mass caterers; (4) description of the foodstuff, and if necessary of its use, which is clear enough to let the purchaser know its true nature and distinguish it from other products with which it might be confused; and (5) the use in the Member State of marketing of the sales name under which the product is legally manufactured and marketed in the Member State of production shall also be allowed. However, this latter situation has qualifications: first, where the other labelling requirements would not enable consumers in the Member State of marketing to know the true nature of the foodstuff and to distinguish it from foodstuffs with which they could confuse it, in which case the sales name shall be accompanied by other descriptive information that will appear in proximity to the sales name and, second, this name cannot be used if the product so named is so different, in the Member State of sale, as regards its composition or manufacture, from the foodstuff known there under that name that the provisions of the points above are not sufficient to ensure correct information for consumers.
- List of ingredients including additives; if vitamins or minerals are added, this should be indicated.
- An indication of the net quantity should be provided.
- The date of minimum durability should be indicated.
- Special conditions of storage and use that would affect the minimum durability should be provided.
- Name and address of manufacturer or seller should be given in addition to the identification mark required by the hygiene regulations outlined above.

The requirements of the Council Regulation (EEC) No. 1898/87 (EU, 1987), as amended, on the protection of dairy designations, must also be respected and substances cannot be used in their manufacture, which would be for the purpose of replacing, in whole or in part, any milk constituent. This has particular application to the names used for blends of preserved milks and vegetable oils.

2.2.6 *Horizontal – packaging legislation*

The dairy products covered in this chapter may be packed in different packaging formats, for example large tote bags with plastic liners, multi-walled paper sacks with plastic liners, plastic packs, tins and/or metal cans. These packages should comply with the general requirements of Regulation (EC) No. 1935/2004 (EU, 2004c) and the particular

requirements, such as contained in the Commission Directive 2002/72/EC (EU, 2003), as amended by the Commission Directives 2004/19/EC (EU, 2004b) and 2004/1/EC (EU, 2004a). It is normal for processors to specify to their packaging suppliers that their products comply with the requirements of these directives.

2.3 United Kingdom legislation

2.3.1 *Legislative basis*

It should be borne in mind that, up to recent times, England and Wales had common legislation, signed by the appropriate minister of the UK government and the Secretary of State for Wales. Separate but similar legislation was enacted for Scotland and Northern Ireland; however, some differences could and sometimes did occur. Then in 2000, with the establishment of the Welsh Assembly, separate but similar legislation was enacted for Wales. The primary source of legislation is by Acts of Parliament, primarily the Westminster Parliament; secondary legislation in the form of Statutory Instruments (SIs) and Statutory Regulations and Orders (SROs) are enacted under specified sections of the enabling act or acts.

Details of the current legislation in the United Kingdom as well as the separate legislation applicable to Scotland, Wales and Northern Ireland may be found via the relevant link on the (UK) Foods Standards Agency website: http://www.foodstandards.gov.uk or that of the Office of Public Sector Information (OPSI) http://www.opsi.gov.uk/legislation/uk.htm. In searching the OPSI website it is necessary to know the year and number of the Act or Statutory Instrument (SI) of interest.

2.3.2 *Background*

In the 1850s, there was increasing concern on the issues of food purity and food adulteration based on the identification of such issues by analysts and medical doctors. This led to the adoption of three separate pieces of legislation addressing food adulteration, one such was the Adulteration of Food and Drugs Act 1860 (HMSO, 1860). However, this was ineffective and a Select Committee appointed to investigate the ongoing problems identified that this was because, while legislation in place allowed the appointment of Food Inspectors and Public Analysts, it did not make this compulsory. This shortcoming led to the enactment of the Sale of Food and Drugs Act 1875 (HMSO, 1875). The main requirements of this Act were as follows:

- nothing should be added to food for sale, which would be injurious to health;
- prohibition of sale of food that was not of the proper nature, substance or quality;
- the statutory appointment of analysts;
- the empowerment of purchasers of a food to have it analysed;
- the naming of officers entitled to obtain samples for submission to an analyst.

Although it was not without its critics, the Act with subsequent amendments, enlargement and consolidation remained in force for the next 60 years (Mornier-Williams, 1951).

In the early 1930s, a Departmental Committee on the Composition and Description of Food was established to look into the whole area of definitions, standards, labelling and advertising. This Committee was in favour of a limited number of standards, the main aim of which would be to inform consumers of what they were purchasing (Mornier-Williams, 1951). In 1934, the report of this committee resulted in a new consolidated Sale of Food and Drugs Act (HMSO, 1938). This 1938 Act gave much wider powers to the Department of Health, but did not actually come into force until after the outbreak of World War II. It was agreed that all orders and regulations under this Act would be made by the Ministry for Food, which had then been established. The 1938 Act remained in place until it was replaced by the Sale of Food and Drugs Act 1955 (HMSO, 1955). Subsequently, as a result of a number of food scares in the 1980s, with causes such as salmonella, listeria and bovine spongiform encephalopathy (BSE), the Food Safety Act 1990 (HMSO, 1990), was enacted. This was a broad measure that created a more systematic structure of UK food law and tightened up on offences, enforcement powers and penalties.

The Sale of Food and Drugs Acts 1875 (HMSO, 1875) was not the sole legal instrument involved. It did not give any powers to government departments to develop compositional standards for foods in general in the area of fair trade. Various Public Health Acts gave powers to the then Local Government Board, which became the Department of Health in 1919, in the area of food, but these were restricted to the protection of health and not fair trade. Nonetheless, during the early years of the twentieth century, there were pressures on the Local Government Board, and later the Ministry for Health, from dairy companies such as Nestlé, St. Ivel and United Dairies, to develop standards for condensed and evaporated milk, claiming that their business was being seriously affected by cheap low-fat American condensed milk (Mornier-Williams, 1951; French & Phillips, 2000). An Interdepartmental Committee on Condensed Milk was established in 1920 to consider whether standards should be developed for such products under the Public Health (Regulations as to Food) Act 1907 (HMSO, 1907). This resulted in the Condensed Milk Regulations 1923 (HMSO, 1923a) and the Dried Milk Regulations 1923 (HMSO, 1923b) being adopted.

Speaking at the 1966 Spring Conference of the Society of Dairy Technology, Mr. C.W. Cullip, Production Manager of the Cooperative Wholesale Society stated that the Public Health (Condensed Milk) Regulations 1923 (HMSO, 1923a), required for full cream products, a fat content and solids-not-fat content of 9 g 100 g^{-1} and 22 g 100 g^{-1}, respectively, thus giving a minimum total solids content of 31 g 100 g^{-1}. The unsweetened skimmed product was required to have minimum total solids content of 20 g 100 g^{-1}, and in the sweetened variety, a total milk solids content of 26 g 100 g^{-1}. He said that, although the regulations had been amended in some minor aspects since that time, the compositional standards were still in place (Cullip, 1966).

Prior to joining the EU, the United Kingdom maintained separate legislation for condensed and dried milks, the latest versions of these standards in force at that time were the Condensed Milk Regulations 1959 (HMSO, 1959) and the Dried Milk Regulations 1960 (HMSO, 1960).

On hygienic aspects, in 1912 or thereabouts, the quality and purity of milk supply began to receive increased attention. World War I interfered with the enforcement of the Milk

and Dairies (Consolidation) Act 1915 (HMSO, 1915), but by 1918 a tentative system of milk grading was in operation. The production of clean and safe milk was for the first time seriously addressed under the Milk and Dairies Amendment Act 1922 (HMSO, 1922a) and the Milk (Special Designations) Order 1922 (HMSO, 1922b), which incorporated different grades for raw milk. A paper presented to the Scottish Section of the Society of Dairy Technology in 1947 outlined the early history of pasteurisation in the United Kingdom with particular focus on developments in Scotland (Smillie, 1948). It stated that even before the end of the World War I (1914–1918), official recognition had been granted to Grade A (Certified) Milk and Grade A Milk and this continued in the Ministry Food (Continuance) Act of 1920 (HMSO, 1920). However, it was also mentioned that there was sufficient hidden opposition to the recognition of pasteurised milk in order to prevent its official introduction.

When the United Kingdom joined the then EEC on 1 January 1973, European legislation began to have a major role in shaping the evolving national legislation. However, as outlined in the discussion of European legislation earlier, harmonisation of vertical legislation did not start until the 1970s and on hygienic aspects of milk production until the mid-1980s. European Directives have to be enacted into the laws of Member States, while Community Regulations are binding in their entirety on Member States. In the latter case, the relevant SIs reference the requirements contained therein and outline particular elements, such as interpretations/definitions, specify the competent authority, address administration, detail offences, defences and penalties, and specify certain schedules. Where the European regulations specify general provisions, the UK SIs may lay down more specific requirements, and may address national provisions where discretion or optional provisions are delegated to Member States.

2.3.3 *Present legislation on composition*

Condensed and dried milk

The provisions of the EU Council Directive 76/118/EEC (EU, 1976) were enacted into law under the Condensed Milk and Dried Milk Regulations 1977 (HMSO, 1977). These revoked the existing and separate regulations on condensed milk and dried milk, and were amended 10 times up to and including 2001. Following the adoption of EU Directive 2001/114/EC (EU, 2001), the 1977 regulations were replaced by the Condensed Milk and Dried Milk (England) Regulations 2003 (HMSO, 2003a). Finally, in early 2008, the Condensed Milk and Dried Milk (England) (Amendment) Regulations 2008 (HMSO, 2008) were enacted to incorporate the EU Council Directive 2007/61/EC (EU, 2007e) as regards protein standardisation and the addition of vitamins and minerals. Separate regulations were enacted to incorporate into national legislation the 2001 (EU, 2001) and 2007 (EU, 2007e) EU Directives for Wales, Scotland and Northern Ireland.

One interesting feature of the 1977 UK Regulations [as amended in 1982 (HMSO, 1982)] and the 2003 Regulations is that they contain a definition of '*total milk solids*' as follows: '*. . .all the constituents of milk other than water, including milk fat, the constituents other than milk fat being present in their natural proportions*'. The UK Food Standards Agency

has produced useful Guidance Notes on the Condensed Milk and Dried Milk (England) Regulations 2003 (UK Food Standards Agency, 2003).

Caseins and caseinates

The provisions of EU Council Directive 83/417 (EU, 1983) were enacted into law by the Caseins and Caseinates Regulations 1985 (HMSO, 1985) and its four subsequent amendments. Requirements regarding sampling and analysis, in accordance with and referencing Commission Directive 86/424/EEC (EU, 1986) were introduced through the 1989 amendment (HMSO, 1989).

Infant formulae and follow-on formulae

The provisions of EU Commission Directive 91/321/EEC (EU, 1991a), as amended, were incorporated into national legislation by the Infant Formula and Follow-on Formula Regulations 1995 (HMSO, 1995) and its amendments (HMSO, 1997, 2000, 2003b). Following the adoption of the recast EU Commission Directive 2006/141/EC (EU, 2006a), the national legislation was updated by the enactment of the Infant Formula and Follow-on Formula (England) Regulations 2007 (HMSO, 2007), and corresponding regulations in Wales, Scotland and Northern Ireland.

The new regulations were intended to come into force on 11 January 2008. However, their implementation was suspended in England and Wales following an application by the Infant and Dietetic Foods Association (IDFA) for judicial review, challenging the date by which baby milk companies need to comply with new labelling requirements. Following the hearing, the ruling issued was that the labelling aspects of the legislation should come into force on 1 January 2010, along with the new compositional rules. As a consequence, manufacturers may continue to produce and offer for sale infant and follow-on formula bearing labels that comply with the previous legislation until 1 January 2010. From then on they should be labelled in accordance with the 2007 regulations as regards the labelling of such products. The Court judgement did not affect the rules relating to advertising of infant and follow-on formula, which apply immediately. Similar legal challenges were also taken in Scotland and Northern Ireland. The outcome on the hearing in Scotland is awaited, while that in Northern Ireland was suspended pending the outcome of the former court case (UK Food Standards Agency, 2008b; Jukes, 2008). The UK Food Safety Authority is in the process of updating its guidance on the new regulations and a copy of the latest draft proposals are available (UK Food Standards Agency, 2007, 2008a).

Skimmed milk with non-milk fat and other concentrated and dried milk

On 1 January 1996 quite a number of existing UK regulations on foods were revoked, including those related cheese, butter, cream and skimmed milk with non-milk fat. Prior to that date the Skimmed Milk and Non-Milk Fat Regulations 1960 (HMSO, 1960) and the Skimmed Milk with Non-Milk Fat (Scotland) Regulations 1960 (The Stationery Office,

1960) imposed requirements in relation to labelling and advertising. Some sections of these regulations had already been amended on the basis of the EU Council Regulation 1898/87/EEC (EU, 1987). All that now remains is a requirement in the Food Labelling Regulations 1966 (HMSO, 1996) to have a warning on products consisting of skimmed milk together with non-milk fat, capable of being used as a substitute for milk, and that are not formulated for infants, that the product is unfit for, or not to be used as, food for babies. It is likely that product names, in accordance with the now repealed regulations outlined above, would be regarded as customary names in the United Kingdom, having due regard for the constraints of the Council Regulation 1898/87/EEC (EU, 1987); however, this has not been tested in the courts up to this time.

Other concentrated and dried milk products

These are not standardised or specifically regulated under UK legislation. However, it should be borne in mind that the customary use of names for these products would apply, for example to products such as buttermilk powder. Also the requirements of the Food Labelling Regulations 1996 (HMSO, 1996), as amended, apply to all such product names.

2.3.4 *Present legislation on hygiene*

The new EU hygiene and food safety regulations, which are outlined in Section 2.2.3 above, are implemented in England by The Food Hygiene (England) Regulations 2006 (HMSO, 2006). Wales and Northern Ireland have similar but separate regulations, for example, the Food Hygiene (Wales) Regulations 2006 (The Stationery Office, 2006b) and the Food Hygiene Regulations (Northern Ireland) 2006 (The Stationery Office, 2006c). In Scotland, implementation is by The Food Hygiene (Scotland) Regulations 2006 (The Stationery Office, 2006a).

2.3.5 *The Dairy UK Code of Practice for HTST pasteurisation*

Dairy UK is an organisation that represents the UK dairy industry and, in 2006, it published an updated Code of Practice to apply the best practice, and to seek to ensure that adequate controls and monitoring procedures are in place for HTST milk pasteurisation (Dairy UK, 2006). This updates the previous UK Dairy Industry Federation Code of Practice (Dairy Industry Federation, 1995) by addressing the requirements in the new EU hygiene regulations, and in particular, the requirement in Regulation (EC) 852/2004 (EU, 2004d) that FBOs should apply the principles of HACCP to ensure product safety. Furthermore, it applies the findings of research undertaken into the effectiveness of HTST processing conditions on the destruction of *Mycobacterium avium* subsp. *paratuberculosis* (MAP) in the intervening period. In this regard, it recommends that the minimum holding time of 15 s at 72°C be increased to a minimum of 25 s and used for drinking milk, based on the experimental evidence that this increases the effectiveness of the destruction of MAP under certain conditions (Grant *et al*., 2005). While this recommendation has been adopted widely by the UK industry, and supported by many retailers, it is a recommendation that is voluntary and is not a legal requirement for HTST heat treatment, which remains at 72°C for 15 s.

Pasteurisation has long been recognised as the most important critical control point (CCP) in ensuring the safety of milk and dairy products. The Dairy UK code aims to help ensure that products have been adequately and correctly pasteurised and post-pasteurisation contamination has been avoided. One example is a discussion on the phosphatase test, used to verify the adequacy of milk pasteurisation. This Code applies to 'normal' HTST pasteurisation, but not to high-temperature pasteurisation or UHT processing, where different considerations would apply.

2.4 Irish legislation

2.4.1 *Introduction*

The Irish Parliament (Oireachtas) consists of the President and two Houses of the Oireachtas (Dail Eireann and Seanad Eireann), which are responsible for enacting new legislation in Ireland. A proposal for new legislation is published as a Bill, which becomes an Act and is declared as law when it is agreed by both houses of the Oireachtas and signed by the President. Acts frequently give powers to specific ministers of governments (particularly those with responsibility for Health and Agriculture and Food) to make secondary laws known as SIs, and can be written in the form of Regulations or Orders and detail specific rules and give enforcement powers to a particular authority. The similarity to the UK legislative process, on which it was based, will be apparent. Indeed, from the foundation of the state in 1922 until mid-century, reference was often made to Acts of the UK Parliament, such as the Food and Drugs Act 1875 (HMSO, 1875), as discussed earlier, which had application in the whole of Ireland at the time of its enactment. For instance such references are contained in the Dairy Produce Act 1924 (The Stationery Office Dublin, 1924) and the Sale of Food and Drugs (Milk) Act 1935 (The Stationery Office Dublin, 1935).

Until its joining the then EEC in 1973, Ireland had quite limited legislation governing the composition of foods. Indeed it may be argued that these were not really required, because as an exporter of the majority of its food products, Irish manufacturers frequently adhered to the compositional standards and requirements of its main export market, the United Kingdom, and production for the home market reflected the same require ments.

Until 1990, the hygiene, production and marketing of milk and milk products in Ireland were regulated by the Dairy Produce Acts 1924 to 1984, the Creameries Acts 1928 to 1934, the Milk and Dairies Acts 1935 to 1956 and the Regulations and Orders issued under these Acts. The Compendium of Food Law in Ireland (Food Safety Authority of Ireland, 2003) contains a list of these acts and their implementing regulations and orders. These provisions had laid down a definition of Grade 'A' pasteurised milk, including prescribed heat treatment options, certain hygienic requirements for milk production and animal health, inspections of animals and dairies and other such details.

The European Communities (Standards for Heat-Treated Milk in Intra-Community Trade) Regulations 1990 (The Stationery Office Dublin, 1990) were adopted primarily to implement and update legislation following the adoption of the Council Directive 85/397/EEC (EU, 1985b). When the EU hygiene Council Directive 92/46/EC (EU, 1992a)

and its amendments were adopted in the period 1992 to 1994, the European Communities (Hygienic Production and Placing on The Market of Raw Milk, Heat-Treated Milk and Milk-Based Products) Regulations 1996 (The Stationery Office Dublin, 1996) were enacted to implement the EU provisions. The text of the Irish Regulations was a consolidated text containing the amendments that had been made to the original Council Directive 92/46/EC (EU, 1992a), and was thus a useful reference.

2.4.2 *Present legislation on hygiene*

The 1996 Regulations (The Stationery Office Dublin, 1996) remained in force until 2006 when they were replaced by the European Communities (Food and Feed Hygiene) Regulations 2005 (The Stationery Office Dublin, 2005) and its amendments SI 387 of 2006 (The Stationery Office Dublin, 2006a), and SI 56 of 2007 (The Stationery Office Dublin, 2007a), which implement the EU Regulations (EC) No. 852/2004, (EC) No. 853/2004, (EC) No. 854/2004, (EC) No. 882/2004, (EC) No. 2073/2005, (EC) No. 2074/2005 and (EC) No. 2076/2005 (EU, 2004d, e, f, g, 2005a, b, c), respectively; insofar as they relate to the Irish Department of Agriculture and Food, who are responsible for foods of animal origin; these regulations include the provisions governing milk and milk products. It should be noted that the European Communities (Hygiene of Foodstuffs) Regulations 2006 (The Stationery Office Dublin, 2006a, b) implement EU Regulation (EC) No. 852/2004 (EU, 2004d) for those foods under the control of the Irish Department of Health and Children.

As the latest European legislation was in the form of regulations that were directly applicable in all Member States, the national implementing regulations address aspects, such as commencement, enforcement, administration, offences, penalties, while referring to the specific regulations concerned for the detailed provisions.

2.4.3 *Present legislation on specific products*

Condensed and dried milk

The provisions of EU Council Directive 76/118/EEC (EU, 1976) as amended were incorporated into Irish legislation by the European Communities (Dehydrated Preserved Milk) Regulations, 1980 (The Stationery Office Dublin, 1980), and its subsequent amendments. These are quite short, implementing the Directive and detail aspects, such as commencement, enforcement, administration, offences and penalties, while referring to the Directive itself as regards the detailed provisions. Following the replacement with the updated EU Council Directive 2001/114 (EU, 2001), the new provisions were incorporated by the European Communities (Dehydrated Preserved Milk) Regulations 2003 (The Stationery Office Dublin, 2003). Again these reference the requirements of the Directive.

The provisions of the Council Directive 2007/61/EC (EU, 2007e) of early October 2007 that amends the Council Directive 2001/114/EC (EU, 2001) to allow protein standardisation and the addition of vitamins and minerals, is yet to be incorporated into Irish law. However, the amending Directive obliges Member States to complete this transposition by 31 August 2008 and the Irish dairy industry is keen to have these matters addressed at an early date.

Caseins and caseinates

The European Communities (Caseins and Caseinates) Regulations, 1985 (The Stationery Office Dublin, 1985) give effect to EU Council Directive 83/417/EEC (EU, 1983), the composition, manufacturing characteristics, labelling of edible caseins and caseinates, while the European Communities (Caseins and Caseinates) (Methods of Sampling and Analysis) Regulations 1987 (The Stationery Office Dublin, 1985, 1987) give effect to EU Directives 85/503/EEC (EU, 1985c) and 86/424/EEC (EU, 1986) on the methods of sampling for the chemical analysis of edible caseins and caseinates.

Infant formula and follow-on formula

The European Communities (Infant Formulae and Follow-on Formulae) Regulations 1998, and a subsequent 2000 amendment, gave effect to Commission Directive 91/321/EEC (EU, 1991a), and its two subsequent amendments, on the compositional, labelling and marketing provisions for infant formulae and follow-on formulae intended for infants (meaning children under the age of 12 months) in good health.

Following the adoption of the recast EU Commission Directive 2006/141 (EU, 2006a), the national legislation was updated by the enactment of the European Communities (Infant Formulae and Follow-on Formulae) Regulations 2007 (The Stationery Office Dublin, 2007b), which replaced the 1998 Regulations as of 1 January 2008. These Regulations comprise 49 pages, include all the requirements of the EU Directive, and give further effect to EU Commission Directive 92/52/EEC (EU, 1992b) on infant formulae and follow-on formulae intended for export to third countries. However, they point out that they do not give effect to those aspects of EU Commission Directive 2006/141/EC (EU, 2006a) that amend the Commission Directive 1999/21/EC (EU, 1999b).

Other concentrated and dried milk products

These are not standardised or specifically regulated under Irish legislation. However, it should be borne in mind that the customary use of names for these products, as referred to in the EU Labelling Directive 2000/13/EC (EU, 2000a), as amended, incorporated into Irish Law under the European Communities (Labelling, Presentation and Advertising of Foodstuffs) Regulations 2002 (The Stationery Office Dublin, 2002) would apply.

2.5 United States legislation

2.5.1 *Introduction and background to US legislation*

At the outset it should be pointed out that this chapter shall address the US federal legislation. Up to 1900, there was little federal legislation addressing food standards; the individual states controlled domestically produced and distributed foods; however, this control was markedly inconsistent from state to state. From early 1880s the USDA Division of Chemistry (renamed the Bureau of Chemistry in 1901), under Harvey Wiley who had been appointed its chief chemist in 1883, began researching the adulteration and misbranding

of food (and drugs) on the market. Though without any regulatory powers, the Division published their findings in a 10-part series entitled Foods and Food Adulterants. Based on these results, Wiley started to lobby for a federal law to set standards for food and drugs in interstate trade. In this he was assisted and supported by state regulators, consumer bodies, medical doctors, pharmacists and certain journalists. Their efforts coincided with a general trend for increased federal regulations in all matters pertinent to safeguarding public health. State laws provided varying degrees of protection against practices such as misrepresenting the ingredients of food products or medicines (Swann, 2008). It should also be added that in the early 1900s, the food industry strongly supported national food legislation in order to obtain national uniformity in regulatory requirements and to build credibility for the food supply (Porter & Earl, 1992).

Despite considerable debate on the issue of constitutionality surrounding States' rights, Congress enacted the Food and Drugs Act 1906 (Pub. L. No. 59–384 34 STAT. 768), sometimes called the 'Wiley Act' in honour of its chief advocate (see http://www.fda.gov/opacom/laws/wileyact.htm). This act was aimed at '*preventing the manufacture, sale, or transportation of adulterated or misbranded or poisonous or deleterious foods, drugs, medicines, and liquors, and for regulating traffic therein, and for other purposes*'. Congressional Acts identify and grant broad authority to federal agencies to interpret their provisions into the United States Code, with the relevant enforcement agencies identified.

Under the Food and Drugs Act 1906, responsibility for administration and their examination for 'adulteration' or 'misbranding' was granted to the Wiley's USDA Bureau of Chemistry (USDABOC). Over the years the name of this body has changed to the more familiar Food and Drug Administration (FDA). The evolution and development of this organisation shall be discussed a little later. Despite the vigour with which the USDABOC pursued its new powers, the intentions of the original Act it did not really succeed in establishing a regulation at the federal level. *Firstly*, it regulated the adulteration and mislabelling of foods in interstate trade in their original packages, based on their labelling. However, when bulk packages were opened and repacked, responsibility for control reverted to the individual states. *Secondly*, it did not have the clear mandate to develop standards for foods. *Thirdly*, in challenges, the courts upheld State regulations that differed from, or were additional to, those imposed at the federal level (Porter & Earl, 1992). Over the next 25 years amendments were made to the original Act, but in early 1930s, because of ongoing problems, the federal regulators, consumer groups and the media pressed again for a new act with more powers and scope.

It took 5 years to be passed but the Federal Food, Drug, and Cosmetic Act (FFDCA) of 1938 was finally adopted (see http://www.fda.gov/opacom/laws/fdcact/fdctoc.htm). This Act is sometimes referred to as Title 21, Chapter 9 of the United States Code (21 USC 9). As well as extending the scope of the earlier act to cover cosmetics and therapeutic devices, this new Act repealed the Food and Drugs Act 1906 and contained the following new provisions of relevance to food:

- Allowed that safe tolerances be set for unavoidable poisonous substances.
- Permitted standards of identity, quality and fill-of-container to be set.
- Authorised the inspection of manufacturing premises.
- Added the use of court injunctions to the previous penalties of seizures and prosecutions.

This law, although it has been subject to frequent amendments in the intervening years, remains the basis for federal regulation by the FDA to the present day. Some of the amending acts of relevance to the dairy and related sectors include

- Infant Formula Act of 1980 (Pub. L 96–359).
- Nutrition Labelling and Education Act (NLEA) of 1990 (Pub. L 101–535).

Some other acts of interest are

- The Filled Milk Act 1923 (see http://www.fda.gov/opacom/laws/milkact.htm), this has definitions of filled milk.
- The Fair Packaging and Labelling Act 1967 (see http://www.fda.gov/opacom/laws/fplact.htm).

Evolution and development of the FDA

As mentioned earlier, when the Food and Drugs Act 1906 was passed into law, responsibility for its administration and for the examination of food for 'adulteration' or 'misbranding' was granted to the USDABOC. Over time this body has changed its name to the Food and Drugs Administration and has transferred the department of government under which it operates on a number of occasions, according to the following timeline:

- *1927* – the non-regulatory duties of the USDABOC were transferred with the USDA and its name was changed to the Food, Drug and Insecticide Administration.
- *1930* – the name was shortened to the more familiar Food and Drug Administration (FDA).
- *1940* – it was transferred to the Federal Security Agency.
- *1953* – the agency was transferred again, this time to the Department of Health, Education and Welfare (HEW).
- *1968* – became part of the Public Health Service within HEW.
- *1980* – the education function was removed from HEW to create the Department of Health and Human Services (DHSS) – the FDA's current home.

The FDA is now responsible for about 80% of the US food supply. The exceptions are as regards the safety, wholesomeness, labelling and packaging of meat, poultry and certain egg products, which are the responsibility of the US Department of Agriculture (Swann, 2008).

2.5.2 *The 'Code of Federal Regulations'*

The Code of Federal Regulations (CFR) is the consolidated source of the general and permanent rules developed by the relevant US government departments and/or their administrative agencies and also published in the Federal Register. It is divided into 50 titles that represent broad areas that are subject to Federal regulation. For instance, Title 7 covers Agriculture, administered by the USDA, and Title 21 deals with Food and Drugs, administered by the

FDA. Each volume of the CFR is updated once every year and is issued on a quarterly basis. It is published by the Office of the Federal Register, an agency of the National Archives and Records Administration.

- Titles 1–16 are updated as of January 1
- Titles 17–27 are updated as of April 1
- Titles 28–41 are updated as of July 1
- Titles 42–50 are updated as of October 1

Each title is divided into chapters; each chapter is further subdivided into parts that cover specific regulatory areas. For example, Part 131 covers milk and cream, including a number of the milk products addressed in this chapter. Large parts may be subdivided into subparts. All parts are organised in sections, and most citations in the CFR are provided at the section level. The full format of such citations are as in the following example for Non-fat Dry Milk 21 CFR Part 131 Subpart B § 131.125. However, an abbreviated form, with just the part and subpart letter, such as 21 CFR § 131.125, is also quite common. Columns 1 and 2 of Table 2.12 list the CFR references for the products addressed in this chapter. The latest CFR is also available online at http://www.access.gpo.gov/nara/cfr/cfr-table-search.html#page1. The online documents are available as ASCII text and PDF files.

2.5.3 *Hygiene requirements for milk and certain milk products*

The United States Public Health Service (USPHS), through its agency FDA, does not have direct legal jurisdiction in the enforcement of milk hygiene standards throughout the country, except where milk and milk products move across State boundaries. Therefore, as regards intra-state control the USPHS serves in an advisory capacity; its functions are intended primarily to assist the State and Local Regulatory Agencies in their local situations. The aims of the USPHS are as follows:

- To promote the establishment of effective and well-balanced milk hygiene programmes in each State.
- To stimulate the adoption of adequate and uniform State and Local milk control legislation.
- To encourage the application of uniform enforcement procedures through appropriate legal and educational measures.

The interest in the USPHS milk hygiene programme derives from two important public health considerations. *Firstly*, the USPHS has promoted increased milk consumption, due to its importance as a major nutritional source for the maintenance of good health, especially that for young children and the elderly. *Secondly*, the recognition that milk had the potential, historically, to serve as a significant vehicle for the transmission of disease. Indeed, in the past, milk especially in a raw state for direct consumption, has been associated with major disease outbreaks.

The development of milk hygiene standards The USPHS and FDA activities in the area of milk hygiene began in the early 1900s with studies on the role of milk in the spread of

disease. These studies led to the recognition that effective control required the application of hygiene procedures and practices throughout the full product chain from production, through handling and processing to product distribution. Subsequent research identified and evaluated the hygiene requirements that should be adopted and enforced to control disease. These included studies that led to improvement of the pasteurisation process. One such example was in 1950 when it was proposed that raw milk containing the causative organism of Q-Fever (*Coxiella burnetii*) might be the cause of a significant number of cases of this disease in California (Bell *et al*., 1950). The effect of pasteurisation on this organism was investigated (Lennette *et al*., 1952), and based on a joint research project by USPHS and University of California (Enright *et al*., 1957), which showed that there could be some survivors if high numbers were present in raw milk, led to the recommendation that the low-heat treatment (LHT) pasteurisation temperature be raised from 61.7°C for 30 min to 62.8°C for 30 min to ensure adequate destruction. No change in HTST pasteurisation was necessary as a result of this research.

Over the years, the incidence of milk-borne illness in the United States has been reduced significantly. The FDA is recognised as having made a major contribution to the improvement of the national milk supply through its technical assistance, training, research, standards development, evaluation and certification activities. Despite the progress that has been made, occasional milk-borne outbreaks still occur, and this requires continued vigilance throughout the full product chain from farm to table. The situation has been complicated by the introduction of new products and processes, the use of new packaging materials and new marketing patterns. This has led to considerable efforts being expended in the development and use of the HACCP-based systems throughout the industry.

The Pasteurised Milk Ordinance

Federal US legislation on milk and some milk products is laid down in the Grade 'A' Pasteurised Milk Ordinance (PMO) (USPHS, 2005) and in the CFR (US National Archives and Records Administration, 2007). In 1924, the USPHS developed the model regulation that came to be known as the Standard Milk Ordinance for voluntary adoption by State and Local Milk Control Agencies. To provide for the uniform interpretation of this Ordinance, an accompanying Code was published in 1927 (USPHS, 2005), which provided administrative and technical details on compliance. Ongoing revisions of the PMO incorporate new knowledge and technology into the legislation and make it effective and practicable. The Ordinance has been revised and updated many times in the intervening period, and the PMO 2007 Revision (USPHS, 2007) is the latest version published, the title changing to the present one in 1965.

The PMO is used as the hygiene regulation for milk and certain milk products, for interstate carriers; it is recognised by the Public Health Agencies, the milk industry, and many others as the national standard for milk hygiene. It represents a consensus of current knowledge and experiences and, as such, is said to represent a practical and equitable milk hygiene standard for the United States. It has been adopted by 46 of the 50 states for their own standards – California, Pennsylvania, New York and Maryland being the exceptions, and these have adopted similar standards. However, as stated earlier, where it is adopted locally, its enforcement becomes a function of the Local or State authorities.

Table 2.11 Standards for grade 'A' milk products in the United States.

Product	Criterion	Requirement
Grade 'A' non-fat dry milk	Butterfat (g 100 g^{-1})	≤1.25
	Moisture (g 100 g^{-1})	≤4.00
	Titratable acidity (g 100 g^{-1})	≤0.15
	Solubility index	≤1.25 mL
	Bacterial estimate	≤30 000 cfu g^{-1}
	Coliforms	≤10cfu g^{-1}
	Scorched particle disc B	≤15.0 g^{-1}
Grade 'A' whey for condensing and/or drying	Temperature	Maintained at a temperature of ≤7°C (≤45°F) or ≥57°C (≥135°F), except for acid-type whey with a titratable acidity of ≥0.04 g 100 g^{-1} or a pH of ≤4.6
Grade 'A' pasteurised or condensed whey and whey products	Temperature	Cooled to 10°C (50°F) or less during crystal-lisation, within 72 h of condensing
	Coliforms	≤10 cfu g^{-1}
Grade 'A' for dry whey, dry whey products, dry buttermilk and dry buttermilk products	Coliforms	≤10cfu g^{-1}

cfu = colony-forming units.
Adapted from USPHS (2007).

This model milk regulation, now titled the Grade 'A' Pasteurised Milk Ordinance (Grade 'A' PMO), that is the 2007 Revision, incorporates the provisions, including limited compositional, physical and microbiological standards, governing the processing, packaging, and sale of some milk products, including buttermilk and buttermilk products, whey and whey products, and condensed and dry milk products (Table 2.11).

2.5.4 *US standards of identity and labelling*

The FDA has established federal standards of identity for a number of dried and concentrated milk products (see columns 1 and 2 of Table 2.12). These standards define the minimum quality requirements for such products in terms of minimum and maximum compositional requirements for constituents such as milk fat, total milk solids, required and permitted ingredients (e.g. vitamins A and D), optional ingredients including additive functional categories (where permitted – see Table 2.13), and processing requirements. Most milk products with a standard of identity must conform to the FDA standard and regulations published in the CFR. In recent years, the standards of identity for certain dairy products whose names include certain nutrient content claims, were revoked, for example evaporated skimmed milk, sweetened condensed skimmed milk and low-fat dry milk. The main compositional requirements of the EU, US and Codex legislations relating to fat, milk total solids and protein in fat-free dry matter are compared in Table 2.14. It may be observed that these compositional standards, while very similar, also have some differences.

Table 2.12 Concentrated and dried products covered by US Federal Standards of Identity (contained in the CFR) and the USDA Grading and Inspection Programme.

Product	Standard of identity (CFR reference)	US standards and USDA specifications as published by USDA
Concentrated (condensed) milk	21 CFR part 131 subpart B § 131.115	
Sweetened condensed milk	21 CFR part 131 subpart B § 131.120	
Evaporated milk	21 CFR part 131 subpart B § 131.130	USDA CID A-A-20072B−1994[a]
Non-fat dry milk	21 CFR part 131 subpart B § 131.125	US standards for grades of non-fat dry milk (roller process)-1984
		US standards for grades of non-fat dry milk (spray process)-2001
		US standards for grades of instant non-fat dry milk-2001
Non-fat dry milk (fortified with vitamins A and D)	21 CFR part 131 subpart B § 131.127	
Dry whole milk	21 CFR part 131 subpart B § 131.147	USDA specifications for instant DWM–1993
		US standards for grades of DWM – 2001
Dry cream	21 CFR part 131 subpart B § 131.149	
Infant formula1[b]	21 CFR part 105 subpart B § 105.65	
	21 CFR part 106 subpart B § 106.20	
	21 CFR part 106 subpart B § 106.25	
	21 CFR part 106 subpart B § 106.30	
	21 CFR part 106 subpart B § 106.90	
	21 CFR part 106 subpart C § 106.100	
	21 CFR part 106 subpart D § 106.120	
	21 CFR part 107 subpart A § 107.3	
	21 CFR part 107 subpart B § 107.10	
	21 CFR part 107 subpart B § 107.20	
	21 CFR part 107 subpart B § 107.30	
	21 CFR part 107 subpart C § 107.50	
	21 CFR part 107 subpart D § 107.100	
Edible dry casein (acid)		• US standards for grades of edible dry casein (acid) – 1968
Sodium caseinate	21 CFR part 182 subpart B § 182.1748	
Dry buttermilk and dry buttermilk product		• US standards for grades of dry buttermilk and dry buttermilk product – 2001

(*continued*)

Table 2.12 Continued.

Product	Standard of identity (CFR reference)	US standards and USDA specifications as published by USDA
Dry whey	21 CFR part 184 subpart B § 184.1979	• US standards for dry whey – 2000
Reduced lactose whey	21 CFR part 184 subpart B § 184.1979a	
Reduced minerals whey	21 CFR part 184 subpart B § 184.1979b	
Whey protein concentrate	21 CFR part 184 subpart B § 184.1979c	• USDA specifications for dry whey protein concentrate – 2003

CFR = Code of Federal Regulations; USDA = United States Department of Agriculture; CID = commercial item description.
[a]Rather than a standard or specification, this is a USDA approved CID.
[b]The requirements for infant formula are also detailed in Chapter IV, section 4.1.2 of the Federal Food, Drug, and Cosmetic Act 1934 as amended by the Infant Formula Act 1980 and subsequently.
US National Archives and Records Administration (2007) and USDA (1968, 1984, 1993, 2000, 2001a, b, c, d, 2003).

Table 2.13 Additive functional classes permitted in US standards of identity for certain concentrated and dried milks including lactose.

Product	CFR reference	Emulsifiers	Stabilisers	Anti-caking agents	Antioxidants	Carriers for vitamins	Flavourings
Concentrated milk	21 CFR § 131.115	×	×	×	×	√	√
Evaporated milk	21 CFR § 131.130	√	√	×	×	√	√
Sweetened condensed milk	21 CFR § 131.120	×	×	×	×	√	√
NFDM	21 CFR § 131.125	×	×	×	×	×	√
NFDM fortified with vitamins A and D	21 CFR § 131.127	×	×	×	×	√	√
DWM	21 CFR § 131.147	√	√	√	√	√	√
Dry cream	21 CFR § 131.149	√	√	√	√		√
Lactose	21 CFR § 168.122	×	×	×	×	×	×

CFR = Code of Federal Regulations; NFDM = non-fat dry milk; DWM = dried whole milk.
× = not permitted.
√ = permitted.

Table 2.14 Comparison of chemical compositions (g 100 g^{-1}) of evaporated, condensed, sweetened condensed and dried milks in EU and US legislation and Codex product standards[a].

	Fat			Total milk solids			Protein (on fat-free dry matter basis)		
Product name (EU designations)	EU	US	Codex	EU	US	Codex	EU	US[b]	Codex
Condensed high-fat milk	≥15	See note[c]		≥26.5			≥34		
Condensed milk	≥7.5	≥7.5		≥25	≥25.5		≥34		
Evaporated milk[d,e]	≥9	≥6.5 to ≤16.5	≥7.5	≥31		≥25	≥34		≥34
Condensed partly skimmed milk[e]	≥1 to <7.5	See note[c]	≥1 to <7.5	≥20		≥20	≥34		≥34
Evaporated semi-skimmed milk[e]	≥4 to ≤4.5			≥28			≥34		
Condensed skimmed milk	≤1	See note[c]	≤1	≥20		≥20	≥34		≥34
Sweetened condensed milk	≥8	≥8	≥8	≥28	≥28	≥28	≥34		≥34
Sweetened condensed and partly skimmed milk	≥1 to <8		≥1 to <8	≥24		≥1 to <8	≥34		≥1 to <8
Sweetened condensed skimmed milk	≤1		≤1	≥24		≥24	≥34		≥34
Dried high-fat milk/high-fat milk powder[f,g]	≥42	≥40 to <72	≥42	≥95	≥95	≥95	≥34		≥34
Dried whole milk/whole milk powder	≥26 to <42	≥26 to <40	≥26 to <42	≥95	≥95	≥95	≥34		≥34

(*continued*)

Table 2.14 Continued.

Product name (EU designations)	Fat			Total milk solids			Protein (on fat-free dry matter basis)		
	EU	US	Codex	EU	US	Codex	EU	US[b]	Codex
Dried partly skimmed milk/partly skimmed milk powder	≥1.5 to <26		≥1.5 to <26	≥95		≥95	≥34		≥34
Semi-skimmed milk powder/dried semi-skimmed milk[c]	≥14 to ≤16			≥95			≥34		
Dried skimmed milk/skimmed milk powder	≤1.5	≤1.5	≤1.5	≥95	≥95	≥95	≥34		≥34

[a]Care should be exercised in using this table as different designations are used for similar products in the EU, United States and Codex Alimentarius – the following notes address some of these issues.
[b]The US Code of Federal Regulations does not specify minimum protein levels for these products.
For further details regarding the references for US CFR sections and Codex standards, refer to Tables 2.12 and 2.18.
[c]These are particular designations outlined in Annex II for certain products listed in Annex I of amended EU Council Directive 2001/114/EC (EU, 2001).
[d]With the exception of non-fat dry milk, the US Code of Federal Regulations no longer specify compositional standards for light, low-fat or skimmed products – nutritional claims on the use of such terms are governed by 21 CFR Part 101; nutritional labelling of foods is compulsory in the United States.
[e]Codex standards for these products are designated as evaporated milks.
[f]Codex standards for milk powders and cream powders designated this product as cream powder.
[g]The US Code of Federal Regulations refers to this product as dry cream.

In the United States, milk products without a standard of identity must conform to regulations specified in the NLEA of 1990. This Act, which amended the FFDCA 1938, authorised the FDA to require nutrition labelling of most foods regulated by the Agency; and to require that all nutrient content claims (i.e. 'high-fibre', 'low-fat'), and health claims be consistent with regulations. The full provisions of the regulations for nutrition labelling and other provisions became effective for most products in May 1994. These new requirements are in addition to the previously established labelling requirements for statement of identity (21 CFR 101.3), net contents declaration (21 CFR 101.105), ingredient list (21 CFR 101.4) and name and place of manufacturer or distributor (21 CFR 101.5).

US infant formula requirements are those detailed in Chapter IV, Section 4.1.2 of the FFDCA 1934 as amended by the Infant Formula Act 1980 as amended (see http://www.fda.gov/opacom/laws/fdcact/fdcact4.htm#sec412) and given in 21 CFR Parts 105, 106 and 107 as outlined in Table 2.12.

2.5.5 *The USDA specifications and grading schemes for certain milk products*

The USDA has developed 32 standards and grades for milk products, including those concentrated and dried milk products listed in the third column of Table 2.12. Dairy standards aid in the marketing of milk and milk products by providing a common terminology in trade through the development, improvement and interpretation of standards, specifications and quality improvement programmes. Dairy grading assists the dairy industry in marketing high-quality dairy products, and provides customers and consumers with confidence in buying them, by providing manufacturers and sellers with independent inspection and assessment of product quality. Grades are based on standards developed by USDA Dairy Programmes' experts in cooperation with industry representatives.

These standards and grading schemes are voluntary; nonetheless, they are used widely. Furthermore, all dairy products offered for sale to the federal government under the dairy price support program, or sanctioned under such programmes as the Dairy Export Incentive Program (DEIP), must be inspected by the USDA graders. Under the grade label programme, packaging can bear an official identification indicating the US grade. It is specified that the products that bear the USDA grade label must comply with any requirements specified in the corresponding CFR, where such exist, as well as meeting the specific additional requirements in the USDA specification or grading standards for that product. It is a prerequisite for grading that the products are manufactured in production plants that are officially approved as meeting the requirements of the USDA's General Specifications for Dairy Plants Approved for USDA Inspection and Grading Service (USDA, 2002). Updated registers of such plants and the products concerned are available on the USDA website (USDA, 2008).

Almost all milk products can be graded, but the service is used most widely for butter, cheddar cheese, instant and regular non-fat dry milk. However, other cheeses, dry whey, dry whey protein concentrate, acid casein, dry buttermilk, dry buttermilk products and dried and condensed milk are also included in the programme. To meet the US 'extra' grade standard for instant non-fat dry milk, the product must have a sweet and pleasing flavour, a natural colour and satisfactory solubility as specified. It must also meet the specified levels of moisture, fat, bacteria, scorched particles and acidity (USDA, 1984, 2001c, d).

Dry buttermilk (USDA, 2001a) and regular non-fat dry milk (USDA, 1984), in bulk intended for manufacturers of ice cream, bakery products and some processed meats, can be graded either US 'extra' or 'standard'. The latter lower grade may be the result of slightly higher moisture or scorched particle levels or some other quality factors that differ between the two grades. The 'extra' and 'standard' grades for dry whole milk (DWM) are based on quality factors that include a maximum bacterial content (USDA, 1993, 2001b). Dry whey is graded for flavour, appearance, milk fat and moisture. It must have a good, sweet taste to earn the 'extra' grade (USDA, 2000). Standards for edible acid casein (USDA, 1968), dry whey protein concentrate (USDA, 2003) and dry buttermilk product (USDA, 2001a) also apply slightly higher standards for 'extra' grade compared to 'standard' grade.

For other milk products for which no US grade standards have been established, there is also a USDA programme for official quality approval. Such products may earn the 'Quality Approved' rating that is based on a USDA inspection of the product and the plant where the product was made.

2.5.6 *Food additives in US legislation*

In Section 2.5.4 it was indicated that regarding standards of identity, where such exist, in certain cases, food additive functional classes are listed (e.g. stabilisers, emulsifiers) under optional ingredients. A total of 32 such additive functional classes are defined in 21CFR§170.3 (o). Specific individual additives are not listed in the standards of identity. Hence, it is worth considering briefly how additives are regulated in the United States. The definition of a food additive is given in the FFDCA 1938, as amended, in Section 201(s) and (t) and in the CFR (21CFR§170.3). Ingredients and substances (including food additives) that may be used are classified into four legal categories as follows:

- *New food additives* that have no proven track record of safety and must have prior review and approval as regards safety by the FDA before marketing. These additives may receive the generally recognised as safe (GRAS) status.
- *Colour additives* that are used in foods, drugs, cosmetics and medical devices and must be approved by the FDA before they can be marketed.
- *GRAS* for substances whose use in food has a proven track record of safety based either on a history of use before 1958 or on published scientific evidence, and that need not be approved by the FDA prior to being used; the various means of achieving this classification are given in 21CFR§170.30 and 21CFR§170.31.
- *Prior sanction* for substances that were assumed to be safe for use in a specific food by either the FDA or the USDA before 1958. An example here is the use of nitrate as a preservative in meat because it was sanctioned before 1958; however, it cannot be used on vegetables because they were not covered by the prior sanction.

There are four subsequent parts of the CFR that list and define the substances for food use:

- *Part 181* – prior-sanctioned food ingredients.
- *Part 182* – substances GRAS.

- *Part 184* – direct food substances affirmed as GRAS.
- *Part 186* – indirect food substances affirmed as GRAS.

A list of all the ingredients and substances are given at the start of each of the above parts, giving the section reference for each compound. As mentioned elsewhere, some of the above parts list ingredients and substances other than additives; for example Part 182 lists sodium caseinate, spices and other natural seasonings and flavourings; Part 184 lists reduced lactose whey, reduced minerals whey and whey protein concentrate; and Part 186 lists hydrogenated fish oil. In addition, Part 189 lists substances prohibited from use in human food.

A further useful reference point on substances for use in food in the United States is the Food Additive Status List on the FDA website (FDA, 2006). This lists substances alphabetically and outlines their status and limitations for use. For a brief overview on this topic, a short publication of the International Food Information Council (IFIC) prepared with the assistance of the FDA can be found useful (IFIC, 2005). In this regard, there have been petitions to the FDA concerning the use of milk protein concentrate (MPC) as an ingredient (see that of the National Family Farm Coalition (NFFC) http://www.fda.gov/ohrms/dockets/dailys/04/apr04/042904/04p-0202-cp00001-vol1.pdf). While the issue on the use of MPC in the United States is an economic one (see http://www.fda.gov/ohrms/dockets/dailys/00/Nov00/110700/c000007.pdf), the NFFC based one of their objections to MPC on the grounds that its GRAS status has not been established by the FDA.

2.6 Legislation in Australia and New Zealand

2.6.1 *Introduction*

In terms of tonnes of milk equivalent (TME), the top five exporters of dairy products are New Zealand (13.5 million TME), EU 25 Member States (13.2 million TME), Australia (4.8 million TME), the United States (3.5 million TME) and Argentina (2.4 million TME). As regards milk powders (whole, semi-skimmed and skimmed), New Zealand produced 1 million tonnes and Australia 355 000 tonnes in 2006 (IDF, 2007). Hence, it is worth addressing, very briefly, the legislation of Australia and New Zealand.

2.6.2 *The 'Joint Food Standards Code'*

Historically, both countries had separate legislation and indeed that remains partly the situation to this day. However, in 1995, Australia and New Zealand signed a Joint Food Standards Setting Treaty that committed both countries to the development and implementation of a single set of food standards. The Food Standards Treaty provides for a bi-national agency, now called *Food Standards Australia New Zealand* (*FSANZ*), to undertake the relevant food standards development for both Australia and New Zealand. FSANZ has produced a food standards code that is regularly updated and contains the joint standards developed to date (FSANZ, 2007).

The Australia New Zealand Food Standards Code took full effect in Australia and New Zealand on 20 December 2002. However, certain parts of the code, including those covering

Table 2.15 Compositional (g 100 g^{-1}) requirements in FSANZ standard for dried, evaporated and condensed milks.

Product	Water	Milk fat	Milk solids	Milk protein in milk solids-not-fat
Condensed whole milk		≥8	≥28	≥34
Condensed skimmed milk		≤1	≥24	≥34
Evaporated whole milk		≥7.5	≥25	≥34
Evaporated skimmed milk		≤1	≥20	≥34
Dried whole milk	≤5	≥26		≥34
Dried skimmed milk	≤5	≤1.5		≥34

Data compiled from FSANZ (2000).

country of origin labelling, maximum residue level limits, processing requirements and food safety requirements apply in Australia only; New Zealand has retained separate legislation in these areas. In the context of this chapter, the most relevant standards are those for dried milks, evaporated milks and condensed milks (FSANZ, 2000) and infant formulae, as amended, (FSANZ, 2002). The fat and/or protein content of the milk used to make the products covered by this standard, may be adjusted to comply with the compositional requirements set out in this Standard (Table 2.15), by the addition and/or withdrawal of milk constituents in such a way as not to alter the whey protein to casein ratio of the milk being adjusted.

The sections of the Food Standards Code that cover Food Safety Standards may be found in Chapter 3 Part 3.2; Primary Production Standards in Chapter 4 Part 4.2; Food Labelling is covered in Chapter 1 General Food Standards Part 1.2; and Food Additives in Chapter 1 Part 1.3 on substances added to Food Standard 1.3.1 and Schedules 1 to 5 of the same (FSANZ, 2007). These Chapters apply in Australia only; they do not apply in New Zealand.

The FSANZ Food Standards Code does not specify details of materials permitted to be added to or used to produce food packaging materials. However, packaging when used must not cause food to be unsafe or tainted. It is the responsibility of food manufacturers to ensure that their products are safe and packaging suppliers are expected to ensure that their products are suitable for the intended use. Compliance with recognised international food standards, such as those of the EU or the United States, is regarded as reasonable evidence that materials are suitable for food use.

2.6.3 *New Zealand-specific legislation*

In New Zealand, food is regulated under the Food Act 1981, related acts and regulations issued under that Act. The Food Safety Regulations 2002, New Zealand Food Standards and the Dietary Supplements Regulations 1985 were made under the Food Act 1981 and the act involves the following:

- Defines relevant terms, such as food and sale.
- Outlines prohibitions on sale (including unfit food).

- Prohibits misleading labelling and advertising.
- Provides powers of enforcement and offences.
- Contains provisions to make regulations and food standards.

Under the Food Act, the Minister of Food Safety has the power to issue food standards that set minimum requirements for the quality and safety of food for sale. Other relevant New Zealand legislation includes:

- The Food Hygiene Regulations 1974, which set food hygiene requirements, includes registration of food premises. These do not apply to dairy processors. It is anticipated that a new food safety regime will replace the Food Hygiene Regulations 1974.
- The Animal Products Act 1999 gives domestic dairy manufacturers the option of operating under either a registered Risk Management Programme (RMP) or an approved Food Safety Programme (FSP).
- The Animal Products (Dairy Processing Specifications) Notice 2006 lays down the requirements for dairy products and their manufacture, including requirements for risk management programmes as required under the Animal Products Act 1999.
- The Weights and Measures Regulations 1999 addresses labelling requirements for net contents on packaged foods.
- The Fair Trading Act 1986 prohibits false or misleading representation of goods or services.

Further information on food legislation in New Zealand may be obtained from the New Zealand Food Safety Authority (NZFSA) website at http://www.nzfsa.govt.nz/labelling-composition/publications/regulation-of-food-in-nz/index.htm#P89_10435 and actual legislation may be accessed and downloaded at the New Zealand Legislation website at www.legislation.govt.nz. In the latter case, it is appropriate to have the full details of the legislation that is being sought.

2.7 The international perspective – Codex Alimentarius

2.7.1 *What is Codex Alimentarius?*

In the period 1961 to 1962, the Joint FAO/WHO Food Standards Conference established the Codex Alimentarius Commission (CAC), and asked it to implement the joint FAO/WHO foods standards programme and to create the Codex Alimentarius, the name that derives from Latin and translates as *food code*. The Codex Alimentarius has now become the global reference point of national food control agencies, food producers and processors and international food trade. Its influence extends to all continents, and has contributed to the protection of human health and safety and fair trade practices worldwide.

The Codex Alimentarius system provides the opportunity for all member countries to join the international community in formulating and harmonising food standards and ensuring their implementation. It also allows them a role in the development of guidelines and codes of good practice related to hygienic processing and recommendations relating to compliance with those standards. In addition, the Codex Alimentarius has an

increasing relevance to the international food trade. The application of the Sanitary and Phytosanitary Measures Agreement (SPS Agreement) and the Agreement on Technical Barriers to Trade (TBT Agreement) both encourage the international harmonisation of food standards. These Agreements cite international standards, guidelines and recommendations as the preferred measures for facilitating international trade in food. As such, Codex Alimentarius standards have become the benchmarks against which national food measures and regulations are evaluated within the legal parameters of the World Trade Organisation (WTO) Agreements.

2.7.2 *Codex Alimentarius Commission membership and structure*

The Codex Alimentarius Commission (CAC) is an international inter-governmental body. Its membership is open to member nations and associate members of the FAO and/or the WHO. As of July 2007, it had 175 member countries and 1 member organisation (the European Community). Presently, the CAC meets annually, and the venue alternates between the FAO headquarters in Rome and WHO headquarters in Geneva. Nominated senior officials represent member governments at CAC meetings. National delegations may also include representatives of the industry, consumers and academia. A significant number of other international governmental organisations, for example the Office International des Epizooties (OIE), the WTO and recognised international non-governmental organisations (NGOs), such as the International Dairy Federation (IDF), the Confederation of the Food and Drink Industries of the EU (i.e. Confédération des Industries Agro-Alimentaires (CIAA)) and the International Special Dietary Foods Industries (ISDI) may also attend in an observer capacity. Observers are allowed to contribute to the meeting at all stages except in final decisions. This is the exclusive prerogative of member governments.

The CAC has established two types of subsidiary committees: (1) Codex Committees and (2) Coordinating Committees. The former type committee is subdivided into General Subject Committees (currently ten in number), and are so-called because of the horizontal nature of their work, and Commodity Committees (currently 21 in number of which 16 are active), which develop the standards for specific foods or classes of foods. There are also five Regional Coordinating Committees whose role is to ensure that the CAC is responsive to regional interests, and particularly the needs of developing countries. The CAC also establishes *ad hoc* Intergovernmental Task Forces given stated tasks on specific topics. Currently, there are three such task forces on foods derived from biotechnology, antimicrobial resistance and quick frozen foods. The CAC structure is shown in Figure 2.2.

The main aims of the Codex Alimentarius are as follows:

- Protecting consumer health.
- Ensuring fair trading practices.
- Facilitating international trade.

2.7.3 *Codex Alimentarius standards*

The Codex Alimentarius consists of 13 volumes, which contain general principles, general standards, commodity standards, definitions, codes, methods and recommendations and, as

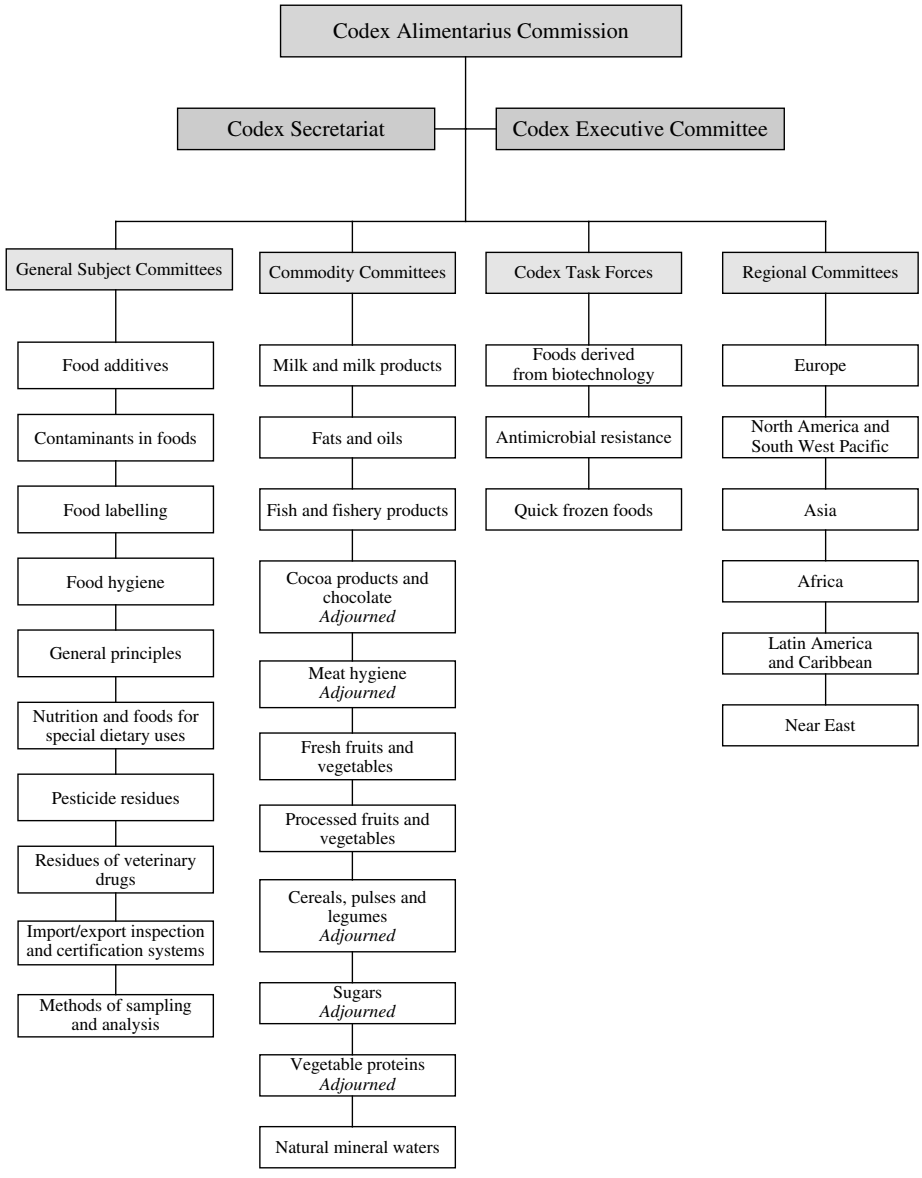

Fig. 2.2 Structure of the Codex Alimentarius Commission.
Note: not all the commodity committees are included.

of July 2006, its content is shown in Table 2.16 (FAO/WHO, 2006e). As may be seen, in addition to individual food commodity standards, it encompasses food labelling, food additives, food hygiene, contaminants, nutrition and foods for special dietary uses, and methods of analysis and sampling.

The CAC has established a number of principles on scientific basis for its decision making (Randell & Race, 1996). These principles ensure that the quality and food safety

Table 2.16 Content of the Codex Alimentarius (as of July 2006).

Category	Number of Codex standards, guidelines and codes of practice
Food commodity standards	186
Food commodity-related texts	46
Food labelling	9
Food hygiene	5
Food safety risk assessment	3
Sampling and analysis	15
Inspection and certification procedures	8
Animal food production	6
Contaminants in foods (maximum levels, detection and prevention)	12
Food additives provisions (covering 292 food additives)	1112
Food additives-related texts	7
Maximum limits for pesticide residues (covering 218 pesticides)	2930
Maximum limits for veterinary drugs in foods (covering 49 veterinary drugs)	441
Regional guidelines	3

Data compiled from FAO/WHO (2006e).

provisions shall be based on sound science and, that in establishing food standards, other legitimate factors may be considered that are relevant to consumer's health and the promotion of fair trade. The Codex Alimentarius standards and related texts are subject to revision, as and when deemed necessary by the CAC and its subsidiary bodies, to ensure that they are consistent with, and reflect, current scientific knowledge. Any member of CAC may identify and present new scientific or other information to the relevant body that may warrant a revision.

The Uruguay round of multi-lateral trade negotiations held under the General Agreement on Tariffs and Trade (GATT), which took place between 1986 and 1994, led to the formation of the WTO on 1 January 1995. For the first time, GATT agreements included agriculture and food in its scope; however, the Marrakesh agreement of 1994 also included the agreements on sanitary and phytosanitary measures (commonly referred to as the SPS Agreement) and on technical barriers to trade (commonly referred to as the TBT Agreement). These agreements acknowledge the need for the harmonisation of international standards to minimise the risk of sanitary, phytosanitary and other technical standards becoming barriers to international trade. Thus, the SPS and TBT Agreements gave formal recognition to international standards, guidelines and recommendations of international organisations, including the CAC, as reference points for facilitating international trade and resolving disputes. Hence, the role of Codex Alimentarius in this regard is now well recognised.

2.7.4 *Codex Alimentarius – general standards*

CAC has, however, developed a number of general standards and codes of practice that are of relevance to concentrated and dried milk products. In this regard, four such standards are as follows:

- The Recommended International Code of Practice General Principles of Food Hygiene CAC/RCP 1-969, Revision 4 (2003) (FAO/WHO, 2003e).
- The Codex Code of Hygiene Practice for Milk and Milk Products CAC/RCP 57-2004 (FAO/WHO, 2004).
- The Codex General Standard for Food Additives (GSFA) CODEX STAN 192-1995 (FAO/WHO, 1995).
- The Codex General Standard for the Use of Dairy Terms CODEX STAN 206-1999 – often referred to by the acronym GSUDT (FAO/WHO, 1999c).

Codex hygiene codes of practice

Codex Alimentarius has developed a general hygiene code to cover all foodstuffs and a specific code for milk and milk products. These hygiene codes consist of 10 Sections, common to both horizontal and specific codes, as outlined in Table 2.17. The aim of the general code, the Codex Recommended International Code of Practice General Principles of Food Hygiene (FAO/WHO, 2003e), is to provide a sound basis for ensuring food hygiene. It should be used in conjunction with any specific hygiene code for the sector concerned, together with the Codex Guidelines for the Establishment and Application of Microbiological Criteria for Foods (FAO/WHO, 2003a) and the Codex Principles and Guidelines for the Conduct of Microbiological Risk Assessment (FAO/WHO, 2003b). An appendix to the general code addresses the HACCP system and contains guidelines for its application.

Table 2.17 Section headings of Codex Alimentarius codes of hygiene.

Introduction	
Section I	Objectives
Section II	Scope, use and definitions
Section III	Primary production
Section IV	Establishment: design and facilities
Section V	Control of operation
Section VI	Establishment: maintenance and sanitation
Section VII	Establishment: personal hygiene
Section VIII	Transportation
Section IX	Product information and consumer awareness
Section X	Training
Annexes	Where necessary in specific codes

Data compiled from FAO/WHO (2003e, 2004).

The Code of Hygiene Practice for Milk and Milk Products (FAO/WHO, 2004) should not be looked at in isolation, but in conjunction with the general code and the other hygiene texts referred to above. References are also made therein to a Codex Guidelines for the Validation of Food Hygiene Control Measures that is still under development. The objective of this code is to provide specific guidance on achieving the general hygiene requirements of Codex commodity standards for milk products. The code uses an FSO approach, outlining hygiene principles (in bold font), explanatory narratives (in italic font) and guidelines for the application of the principles (in normal font). Forty pages long, it expands, in particular, on the requirements of Section 3 (Primary Production) and Section 5 (Control of Operations). The guidelines outline what is required, but do not go into detail on how to achieve the requirement. Annex I contains guidelines for the primary production of milk, additional provisions are given for the production of milk to be used in raw milk products. Annex II gives guidelines for the management of control measures during and after processing. In many cases, the guidelines are of a general nature, for example it states that for perishable products the storage temperature should be sufficient to maintain product safety and suitability throughout the shelf life, but a specific storage temperature is not indicated.

Appendix A outlines typical microbiostatic control measures that are used, such as refrigeration, water activity control, pH reduction, use of preservatives, modified atmosphere packaging, whilst Appendix B outlines typical microbiocidal control measures that are used, such as pasteurisation, microfiltration, high-pressure treatment and commercial sterilisation. Performance criteria are established for pasteurisation as the heat treatment designed to achieve at least a 5 $\log_{10}$ reduction of *C. burnetii* in whole milk of 4 g fat 100 g^{-1}. The process criteria state that, according to validations carried out on whole milk, the minimum pasteurisation conditions are those having bactericidal effects equivalent to heating every particle of milk to 72°C for 15 s (continuous flow) or 63°C for 30 min (batch pasteurisation). It goes on to say that similar conditions can be obtained by joining the line connecting these points on a log time versus temperature graph. It cautions that extrapolation of this graph to temperatures outside the temperature range of 63–72°C, in particular above 72°C, must be treated with care, as the ability to have them scientifically validated is beyond current experimental techniques. It also states that where there are changes in composition, processing or use of the end product, the necessary changes to the scheduled heat treatment should be established and the efficiency evaluated.

Food additives and Codex Alimentarius

CAC is also developing a General Standard for Food Additives (GSFA) and an associated Food Category System (FCS) that is contained in Annex B of the GSFA. The FCS is a means for assigning food additive uses in the Standard (FAO/WHO, 1995). All foods are included in the system, but the food category descriptors used in the FCS are not intended to be legal product designations, nor are they intended for labelling purposes. The FCS is hierarchical, and an additive permitted in the general category is taken as permitted in all sub-categories, unless otherwise stated. All dairy products and dairy analogues, except those that are fat emulsions (e.g. butter and dairy spreads) and foods for particular nutritional uses (such as infant formulae and follow-up formulae), come under the general category

01.0. It is worth mentioning that caseins and caseinates were previously in FCS 12.9.5 but have been moved to 01.5.1 in recent years. The following scheme outlines the FCS for concentrated and dried milks:

01.0 **Dairy products and analogues**, excluding products in food category 02.0 – this is defined to include all types of dairy products that are derived from the milk of any milking animal (e.g. cow, sheep, goat, buffalo). In this category, a 'plain' product is one that is not flavoured, nor contains fruit, vegetables or other non-dairy ingredients, nor is it mixed with other non-dairy ingredients, unless permitted by relevant standards. Analogues are products in which milk fat has been partially or wholly replaced by vegetable fats or oils.

01.3 **Condensed milk and analogues (plain)** – defined to include plain and sweetened types of condensed milk, evaporated milk and their analogues (including beverage whiteners). Includes products based on skimmed, partly skimmed, low-fat and whole milk, blends of evaporated skimmed milk and vegetable fat, and blends of sweetened condensed skimmed milk and vegetable fat.

- **01.3.1** **Condensed milk (plain)** – this is defined as obtained by partial removal of water from milk to which sugar may have been added. For evaporated milk, the water removal may be accomplished by heating and it also includes partially dehydrated milk, evaporated milk, sweetened condensed milk and khoa (cow's or buffalo's milk concentrated by boiling).
- **01.3.2** **Beverage whiteners** – this is defined as milk or cream substitute consisting of a vegetable fat-water emulsion in water with milk protein and lactose or vegetable proteins for use in beverages such as coffee and tea. Also includes the same type of products in powdered form, condensed milk analogues, blends of evaporated skimmed milk and vegetable fat and blends of sweetened condensed skimmed milk and vegetable fat.

01.5 **Milk and cream powder and powder analogues** – this is defined to include plain milk powders, cream powders or a combination of the two and their analogues. Includes products based on skimmed, partly skimmed, low-fat and whole milk.

- **01.5.1** **Milk powder and cream powder (plain)** – this is defined to include milk products obtained by partial removal of water from milk or cream and produced in a powdered form, and includes casein and caseinates.
- **01.5.2** **Milk and cream powder analogues** – this is defined to include products based on a fat-water emulsion and dried for use other than as a beverage whitener (01.3.2). Examples include imitation dry cream mix and blends of skimmed milk and vegetable fat in powdered form.

01.8 **Whey and whey products, excluding whey cheeses** – this is defined to include a variety of whey-based products in liquid and powdered forms.

- **01.8.1** **Liquid whey and whey products, excluding whey cheeses** – whey is defined as the fluid separated from the curd after coagulation of milk, cream, skimmed milk or buttermilk with milk-coagulating enzymes during the manufacture of cheese, casein or similar products. Acid whey is obtained after the coagulation of milk, cream, skimmed milk or buttermilk, mainly with acids of the type used for the manufacture of fresh cheese.

01.8.2 Dried whey and whey products, excluding whey cheeses – whey powders are defined as prepared by spray or roller drying whey or acid whey from which the major portion of the milk fat has been removed.

13.0 Foods intended for particular nutritional uses – defined as foods for special dietary use that are specially processed or formulated to satisfy particular dietary requirements that exist because of a particular physical or physiological condition and/or specific disease and disorder. The composition of these foods must differ significantly from the composition of ordinary foods of comparable nature, if such foods exist. Dietetic foods other than those in 13.0 are included in the categories for their standard counterparts.

13.1 Infant formulae, follow-up formulae and formulae for special medical purposes for infants – defined as foods that are intended for infants and for young children as defined in the sub-categories 13.1.1, 13.1.2 and 13.1.3.

13.1.1 Infant formulae – defined as human milk substitute for infants (aged no more than 12 months) that is specifically formulated to provide the sole source of nutrition during the first months of life up to the introduction of appropriate complementary feeding. Product is in a liquid form, either as a ready-to-eat product, or is reconstituted from a powder. Products, other than those under food category 13.1.3 may be hydrolysed protein and/or amino acid based or milk based.

13.1.2 Follow-up formulae – defined as food intended for use as a liquid part of the complementary feeding of infants (aged at least 6 months) and for young children (aged 1–3 years). They may be ready-to-eat or in a powdered form to be reconstituted with water. Products, other than those under food category 13.1.3, may be soy-based hydrolysed protein and/or amino acid-based or milk-based protein.

13.1.3 Formulae for special medical purposes for infants – defined as foods for special dietary use that are specially processed or formulated and presented for the dietary management of infants and may be used only under medical supervision. They are intended for the exclusive or partial feeding of infants with limited or impaired capacity to take, digest, absorb or metabolise ordinary infant formulae or certain nutrients contained therein, or who have other special medically determined nutrient requirement, whose dietary management cannot be achieved only by modification of the normal diet, by other foods for special dietary uses, or by a combination of the two.

From the above it may be seen that the food categories of main interest to us here are those in the following:

- 01.3.1 (condensed milk plain);
- 01.3.2 (beverage whiteners);
- 01.5.1 (milk and cream powders plain);
- 01.5.2 (milk and cream powder analogues);
- 01.8.2 (dried whey and whey products);

- 13.1.1 (infant formulae);
- 13.1.2 (follow-up formulae).

Discussion on permitted additives shall be confined to these categories.

The GSFA is made up of three Tables: (1) Table 1 lists additives alphabetically that are permitted for use under specified conditions in certain food categories or individual food items; (2) Table 2 lists food categories or individual food items in which food additives are permitted; and (3) Table 3 lists additives permitted for use in food in general, unless otherwise specified in its Annex 3, in accordance with Good Manufacturing Practice (GMP).

Of the above products it should be pointed out that Annex 3 to Table 3 specifies the food category 01.8.2 (i.e. dried whey and whey products, excluding whey cheese), food category 13.1.1 (infant formulae) and 13.1.2 (follow-up formulae) as ones in which the use of additives is governed by Tables 1 and 2 only; therefore, Table 3 does not apply to these food categories.

As regards food category 08.1.2 (dried whey and whey products), at this time there are 29 additives adopted in Table 2 for the food category 01.8.2. As regards food category 13.1.1 (Infant formulae), at this time there are no additives adopted. However, this is likely to change, as 25 additives are under consideration, with 23 at Step 7 and likely to be adopted shortly, with the remaining 2 at Steps 3 and 4, respectively. As regards food category 13.1.2 (follow-up formulae) the situation is similar to that for infant formulae; no additives are yet adopted, but 29 additives are under consideration, with 26 at Step 7 and likely to be adopted shortly, with the remaining 3 at Step 3 or 4.

The products covered by the other food categories under consideration are all allowed, at least in principle, to use the additives in Table 3. In all cases the use of additives should be justified as necessary. In the more recent milk product standards, a table format has been used to show the food additive functions that are technologically justified in the particular product(s) within the scope of the standard. A full list of additives permitted in these products may be found by checking the list under the relevant food category on the Codex website. Looking up the relevant food category in the latest working version of Table 2 will show those additives whose use has been adopted, but also list those still being considered under the Codex step approval procedure. To check on the status of a particular additive, it may be easier to check this in the latest working version of Table 1, where additives are listed in alphabetical order.

Development of the GSFA is work in progress. As additive provisions are adopted, they will be incorporated into the GSFA and published on the Codex website at http://www.codexalimentarius.net/gsfaonline/index.html?lang=e.

At this time, additive provisions are included in all relevant adopted Codex product standards. However, it is the intent of Codex Alimentarius to have the GSFA as the single reference source for additives. This will mean that at some stage the additive provisions in individual standards will be moved to the GSFA. This will not be an easy task and will be carefully monitored by all interested sources. The Codex Committee on Food Additives is a body that addresses the food safety aspects of additives and their use, while the relevant Commodity Committee addresses the technical justification for use in specific products. The completion of the GSFA is likely to take a few more years.

Codex General Standard for the use of dairy terms

From the time of its adoption in 1958 until 1999, the Codex Code of Principles for Milk and Milk Products formed the basis for the identity of milk products, and sought to prevent confusion arising between milk products and imitation milk products. Following the establishment of the Codex Committee for Milk and Milk Products (CCMMP) in 1994, a review was started to update the Code of Principles to ensure conformance to the Codex requirements for food standards. The revision was finalised at the third session of the CCMMP in Montevideo-Uruguay in 1998, and the standard was adopted by the CAC in Rome in June 1999 as the GSUDT (FAO/WHO, 1999c). It contains definitions of milk, milk products, composite milk products and reconstituted and combined milk products and also defines dairy terms. A guide to the nature, intent and implications of the GSUDT has been published by the International Dairy Federation (IDF, 2005).

The GSUDT defines milk in Section 2.1 as follows: *Milk is the normal mammary secretion of milking animals obtained from one or more milking without either addition to it or extraction from it, intended for consumption as liquid milk or for further processing*. Section 4.3.2 states that milk products, which are modified in composition by the addition and/or withdrawal of milk constituents, may be named with a name of the relevant milk product, in association with a clear description of the modification, provided that the essential product characteristics are maintained and that the limits of such compositional modifications shall be detailed in the standards concerned as appropriate. It should be noted that these provisions merely set out the conditions to be observed in the use of the milk product names for modified products. It neither prohibits nor promotes such modifications.

2.7.5 *Codex Alimentarius standards for concentrated and dried milks*

There is significant international trade in condensed and dried milk products. For example, in 2006 worldwide exports of whole milk powder were reported as 1.75 million tonnes and of skimmed milk powder as 1.11 million tonnes (IDF, 2007). The importance of such trade in these and other condensed and dried milk products has led to the development and updating of eight dairy-based product Codex standards (FAO/WHO, 2000, 2001, 2003c, d, 2006a, b, c, d), an infant formula standard (FAO/WHO, 2007) and a standard for sugars that include lactose (FAO/WHO, 1999a). These standards are listed in Table 2.18, and the standards developed by CCMMP follow the structure outlined in Table 2.19.

It can be noted that there are three standards covering various blends of skimmed milk and vegetable fat in evaporated, sweetened condensed and dried forms of milk. The development of these standards came about because of concerns of certain Far Eastern Asian countries that the existence and designations of such products would be compromised by the adoption of the Codex GSUDT in 1999 (FAO/WHO, 1999c). At the time of its adoption therefore the CAC requested the CCMMP to consider, as a matter of urgency, the need to elaborate a standard for products where the milk components had been substituted wholly or partially by non-milk components. Since these types of products appeared to be widespread in Asia, CAC also requested the Coordinating Committee for Asia to consider this issue in parallel to the Committee on Milk and Milk Products (FAO/WHO, 1999b). Despite some initial resistance, the three standards were adopted in 2006.

Table 2.18 Codex Alimentarius standards for dairy-based products.

Standard title	Reference (see text)
Codex standard for evaporated milks	CODEX STAN A-3-1971 Rev.1 1999
Codex standard for sweetened condensed milks	CODEX STAN A-4-1971 Rev.1 1999
Codex standard for whey powders	CODEX STAN A-15-1995
Codex standard for milk powders and cream powders	CODEX STAN 207–1999
Codex standard for edible casein products	CODEX STAN A-18-1995
Codex standard for a blend of evaporated skimmed milk and vegetable fat	CODEX STAN 250–2006
Codex standard for a blend of skimmed milk and vegetable fat in powdered form	CODEX STAN 251–2006
Codex standard for a blend of sweetened condensed skimmed milk and vegetable fat	CODEX STAN 252–2006
Codex standard for sugars[a]	CODEX STAN 212–1999 (amended 1–2001)
Standard for infant formula and formulas for special medical purposes intended for infants[b]	CODEX STAN 72–1981 (amended 4–1997), Rev. 2007

[a]This standard includes lactose; it replaced earlier individual sugar standards, which included a lactose standard.
[b]Formerly CAC/RS 72–1972, which was adopted as a worldwide standard in 1981.

Codex commodity standards for dairy-based products are generally developed by the CCMMP. However, in the case of infant formulae, responsibility for development is under the CCNFSDU, due to the importance of nutritional and related compositional requirements. In the case of lactose, the General Standard for Sugars was developed by the Codex Committee for Sugars (which is now adjourned).

In general terms, the additive provisions in Section 4 of the Codex commodity standards are much more restrictive than those in the Codex GSFA at this time. As mentioned elsewhere on food additives within the Codex Alimentarius, there are ongoing discussions at the Codex Committee on Food Additives (CCFA) as to how to incorporate the additive provisions into the GSFA, but it is likely to be some years before the transposition takes place. In the interim, where manufacturers and traders of such products wish to follow the Codex commodity standards, it is wise to follow the additive provisions contained in the published standards and not in the GSFA.

As mentioned in Section 2.5.4 above, Table 2.14 attempts to compare the main compositional requirements for evaporated, condensed, sweetened condensed and dried milks in Codex standards, EU and US legislation. Considerable agreement can be noted. This is not really surprising as the European Commission, the governments of EU member States and the US government are all active participants in the relevant Codex committees that develop the Codex standards. It is getting increasingly difficult to understand whether it is the national and regional legislation that influences Codex standards or *vice versa*. The case of the protein standardisation of preserved milks, discussed above, is an example of a Codex standard influencing EU legislation. On the other hand, the new EU Commission Directive 2006/141/EC (EU, 2006a) on Infant Formula had a significant influence on the

Table 2.19 Overall structure of dairy product commodity standards.

Section number	Section title	Subsection number	Subsection title
1	Scope		
2	Description		
3	Essential composition and quality factors		
		3.1	Raw materials
		3.2	Permitted ingredients
		3.3	composition
4	Food additives		
5	Contaminants		
		5.1	Heavy metals
		5.2	Pesticide residues
6	Hygiene		
7	Labelling[a]		
		7.1	Name of the food
		7.2	Declaration of milk fat
		7.3	Declaration of milk protein
		7.4	List of ingredients
		7.5	Labelling of non-retail containers
8	Methods of Sampling And Analysis		
Annex[b]	Annex		
Appendix[b]	Appendix		

[a]Not all labelling subsections are used in individual standards; those used are numbered 7.1, 7.2, etc., in sequence.
[b]Annexes and Appendices are included only in certain cases and normally include wording that is intended for voluntary application by the commercial partners and not by the governments. However, in the case of infant formula, the two Annexes contain the detailed compositional requirements for the products therein and are an inherent part of that standard.

NCF of 6.25 for protein being adopted in the 2007 revision of the Codex standard for infant formula (FAO/WHO, 2007).

While Codex standards and other developments within CAC have an increasing effect on national and regional legislation, processors, traders and marketers need to be aware that compliance with the legislation of the country or countries where products are sold is still very important. Variations between such legislation and national standards and any consequential disputes that arise regarding these, may be best left to governments to resolve. It is always wise to ensure you do not win battles but lose wars!

2.8 Private standards and specifications

Many of the products discussed in this chapter are used for further processing into other foods, rather than for sale to final consumers. In such cases, detailed specifications are agreed between the parties concerned that can run from 1 or 2 page documents to 15 or 20 pages. Customer-generated specification documents may be provided. The details included in such documents typically cover raw materials including additives and all ingredients, sometimes specifying individual suppliers, processing conditions involved, detailed nutritional information, physico/chemical composition (sometimes specifying the methodology to verify compliance) packaging details (suppliers and legislation requirements, labelling, potential allergens) and other requirements regarding aspects such as suitability for vegetarians, kosher and halal status.

Alternatively, reference is sometimes made to industry body generic specifications by organisations such as the American Dairy Products Institute (ADPI). These are generally one-page documents and are largely based on relevant USDA and CFR requirements; nonetheless, they are used or referenced by customers outside the United States. ADPI has general specifications for the following products available on its website http://www.adpi.org/products.asp:

- Dry whole milk (DWM)
- Non-fat dry milk (NDM)
- Instant non-fat dry milk (INDM)
- Dry buttermilk (DBM)
- Dry buttermilk product (DBMP)
- Dairy product solids (DPS) – products of milk or whey modified by removal of protein/lactose/minerals
- Dry whey (acid type)
- Dry whey (sweet type)
- Whey protein isolate (WPI) – obtained by the removal of sufficient non-protein constituents from whey so that the finished dry product contains protein not less than 90 g 100 g^{-1}
- Lactose (milk sugar)

For milk products see http://www.adpi.org/product_drymilk.asp; for whey products and lactose see http://www.adpi.org/product_whey.asp. These specifications are also useful to get a brief overview of the requirements of US regulations, standards and grades. This should not substitute for consulting the actual legislation as discussed above.

Methods of analysis used to show compliance with legislative requirements and specification details also need to be considered. Some legislation specifies or details the methodology to be used. Where this is not the case, the methodology validated by organisations such as the IDF, the International Standards Organisation (ISO) and the Association of Analytical Communities (AOAC) International are widely respected and used. In recent years, the IDF and ISO issue joint standards.

2.9 Conclusions and possible future developments

This chapter has endeavoured to cover the legislation in the EU, the United Kingdom, Ireland the United States, Australia, New Zealand and the international standards developed by the CAC. Such legislation covers the majority of products in the category of concentrated and dried milks. However, it does not cover all the products that are the subject of this publication.

It is always more difficult to anticipate what may occur in the future than to review the past. Legislative developments usually lag behind technology. Nowadays, they tend to be initiated to address issues of food safety, trade problems or misleading consumers, with an increasing preference for horizontal rather than vertical legislation. Amendments to existing legislation is likely to continue if and as necessary. The development of sectoral codes of practice may replace or substitute specific and detailed legislation, not just for hygiene but also for labelling and compositional requirements.

The work programme of the CCMMP is scheduled for completion at its next session, likely to be held in early 2010. At that time the committee is likely to be adjourned *sine die*. Hence, new standard development or revision of existing standards is unlikely to be undertaken in the foreseeable future. From then on the developments that affect dairy products at Codex level are likely to take place in the horizontal committees such as Codex Committee on Food Labelling (CCFL), Codex Committee on Food Additives (CCFA) and Codex Committe on Nutrition and Special Dietary Uses (CCNFSDU).

One area where further legislation may emerge is the field of environment. Discussions on climate change, food miles and carbon footprints at national, regional and international levels may lead to further legislative initiatives. The country of origin of products is coming to the fore again and and its discussion has been expanded to include the even more difficult issue of the origin of the individual ingredients of compound or composite products. This makes matters even more complex as regards origin. Also nutritional concerns regarding health, obesity, disease risks and the like may well result in increased levels of clearer and simpler nutritional labelling. Such demands are likely to be balanced by a corresponding concern to avoid increasing the legislative burden on the food industry, where operating margins are narrow. Furthermore, new and unexpected problems will continue to arise, preventing any sense of complacency from setting in.

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3 Technology of Evaporators, Membrane Processing and Dryers

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3.1 Introduction

Research in the manufacture of concentrated and dried dairy products has shown an impressive expansion in recent years. A new and more sophisticated technology of spray drying, including three-stage drying, has been developed. The evaporation technique, which is the classical concentrating method, has been markedly improved (Carić, 1994). Membrane separation technology, where ultrafiltration (UF), microfiltration (MF), reverse osmosis (RO), diafiltration (DF) and nanofiltration (NF) are alternatives, can fractionate species of different molecular weight that pass through a semi-permeable membrane, wherein a pressure gradient is applied between both sides of the membrane. This kind of separation–concentration process has been growing in importance since the 1990s; industrial development is conditioned by the need of lowering costs, ecological demands and the need of common technologies that are able to combine different unit operations (Castro & Gerla, 2005).

Spray drying is one of the most common preservation methods, resulting in low water activity (a_w). Dry milk products contain less than 4 g 100 g^{-1} of water, which also means decreased volume and weight of the product, ease of handling and reduced cost of storage and transport.

Percy, in his patent granted in the United States in 1872, first described the principle of spray drying, and he is considered to be the inventor of spray drying technology. Further developments formed the basis for the manufacture of the first industrial spray drying equipment in 1905. Many inventions in the following years had improved the technology of concentrated and dried dairy products. One significant innovation in drying technology, economy and quality of the powdered products was a two-stage drying, that is, the instantisation method (Carić, 1994).

In the 1980s, the three-stage drying procedure was developed and it brought a further enhancement to the quality of dry products and a simultaneous reduction in energy consumption. Recently, spray drying plants have been designed to produce specifically tailored powders according to end-users' demands. Due to the outstanding achievements in technology, techniques of concentrating and drying ensure that undesirable thermal effects on milk nutrients have practically been eliminated.

3.2 Evaporators

3.2.1 *Principles of evaporation*

Concentration of milk and milk derivatives is a common unit operation in the dairy industry. Milk is concentrated in powder plants prior to spray drying or drum drying (sometimes this method of drying is known as *roller drying*). Whey from either cheesemaking or casein production is always concentrated in anticipation of further processing. This may be de-sugared and spray dried or drum dried.

Concentration of milk or milk derivatives usually has one or more of the following motives:

- To facilitate subsequent processing, for example crystallisation; however, whey is concentrated to a very high level of solids and subsequently cooled in order to form crystals.
- To gain capacity and save energy in the case of drying operations. Evaporation takes far less energy than drum drying or spray drying; therefore, the concentration of dry matter is increased to ~50–64 g 100 g^{-1} (Walstra & Jellema, 1985). The drying capacity of the spray dryer or drum dryer is maximally utilised in this manner.
- To increase the payloads in the case of shipping and storage capacity. Shipping costs for products with limited value like whey, which contain only 5–6 g 100 g^{-1} dry matter, are significant. Concentration to about 26 g 100 g^{-1} saves a factor of 4–5 on the shipping costs. Also storage capacity is utilised much better in the case of concentrated products. The ice water consumption to cool the product in order to maintain its microbial quality is also far less.
- To capture significant water sources. Water is relatively cheap, but the huge quantities available in cheese-, milk powder- or whey- processing plants enhance investments in evaporation equipment.

Milk and milk derivatives contain heat-sensitive whey proteins that would denature when brought to boiling point under atmospheric conditions. Therefore, evaporators for the dairy industry always operate under vacuum conditions that enable boiling at moderate temperatures (Walstra & Jellema, 1985; see also Westergaard, 2004). The boiling point of milk and derivatives is not equal to water, but slightly higher due to the solutes present. The boiling point of the milk rises with increased dry matter contents, as expressed in the following equation:

$$\Delta T = 0.513 * m_2 \tag{3.1}$$

where ΔT = boiling point elevation (°C) and m_2 = molality (moles of solute kg^{-1} solvent) (mol kg^{-1}).

The boiling point elevation of fresh milk is 0.15°C. There is hardly any variation, because the osmotic pressure of the milk is practically equal to the osmotic pressure of the blood, which is constant (Walstra & Jenness, 1984).

The degree of concentration of milk or milk derivatives depends on the subsequent processing. In the case of the drying of milk, the dry matter content may go up to approximately

50–55 g 100 g^{-1}. In the case of whey, prior to shipping, the maximum of dry matter content is about 30 g 100 g^{-1} due to the crystallisation of lactose. At lactose crystallisation plants, the whey is concentrated up to ~64 g 100 g^{-1} (Walstra & Jellema, 1985).

3.2.2 *Evaporation techniques and systems*

Falling film evaporators are basically vertically mounted heat exchangers that heat liquids to their boiling point and separate the vapour formed and the remaining liquid.

The heat exchangers are mounted inside a vacuum vessel. The product is heated while flowing down in vertical pipes, or more rarely, in vertical plates, due to gravity. An even distribution of the product flow over the tubes is achieved by a pre-distributor followed by a distribution plate (Fig. 3.1). The product is deflected by a flat or slightly concave plate, and it then forms a shallow layer of liquid on a perforated plate. The shallow layer of liquid on the distribution plate percolates through the holes of the plate onto the spaces between the adjoining pipes of the heat exchanger. The small vertical pipes on top of the distribution plate enable flashing, in case of a temperature drop of the liquid at entrance. The heating at the outside of the pipe is done by steam.

Due to the heating, water and other volatile components evaporate. At the underside of the tubes, the vapour and the concentrated liquid are separated. The separation of the gas and droplets of milk is enhanced by use of demisters or centrifugal force separating devices. A pump, which can operate under vacuum conditions, returns the concentrated liquid to the top of the next section of falling film evaporator to be concentrated further.

Another essential component in falling film evaporators is the vacuum pump that maintains the vacuum in the vacuum vessel. It is necessary to maintain vacuum not only at the beginning of the operation, but also during the operation. Almost all products contain

Fig. 3.1 A general view of the distribution plate in an evaporator showing the flow distribution system. Reproduced with the permission of NIZO Food Research B.V., Ede, The Netherlands.

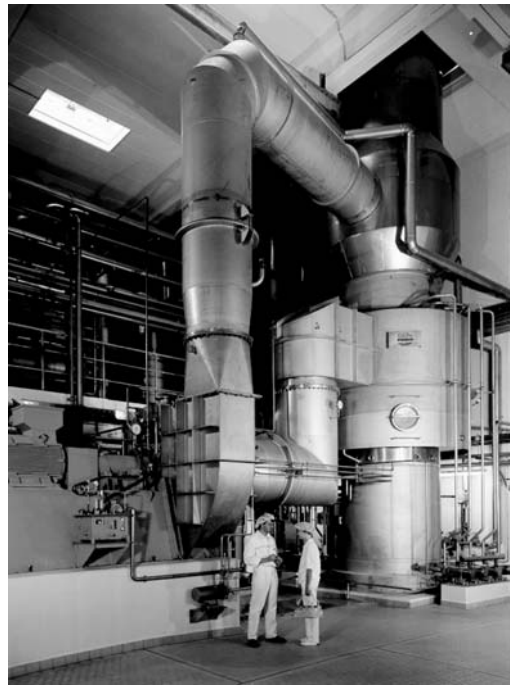


Fig. 3.2 An overall view of a mechanical vapour compression (MVR) heating system of an evaporator (P Kuipers, GEA Wiegand).

dissolved gases and volatile components; these have to be removed. The vacuum level has to be maintained, but efficient heat transfer in the pipes is also lowered by non-condensable gases. High standards for maintaining the vacuum in the vessel are essential in order to minimise the loss. A water ring pump is usually used. The concentration of the milk or derivatives is monitored by measuring the Brix level. The quality of the condensate is monitored by measuring the conductivity.

Two different heating systems are common in the industry known as *thermal vapour recompression* (TVR) and *mechanical vapour recompression* (MVR) (Waalewijn & Bouman, 1996; see also Fig. 3.2). In the former heating system, that is TVR, the vapour released by the product in the first section is used for heating the second section. The pressure in each successive boiler is lower, causing a lower temperature in that section, so the steam condenses at the outer surface of the tubes. The heat of condensation of the steam released by the milk is in this manner used to evaporate the water from the remaining concentrate. Up to seven effects evaporators can be used. Although every effect reduces the steam consumption, it adds to the investment costs and hold-up of the machine. The latter aspect increases residence time distribution and, thus, product loss. In the last effect, the steam from the product is nullified by condensing it; by cooling it in a heat exchanger – a so-called condenser – with surface water. The heating of the surface water is restricted by environmental laws. In many countries, the maximum temperature of the spent cooling water is 30°C. Hence, the surface water may only be heated up by a couple of degrees of centigrade during the summer. The condenser, therefore, uses large quantities of water.

The last stage of the evaporator usually contains the most concentrated liquid. The temperature gradient over the condenser, the temperature gradient over the last stage and the boiling point elevation due to concentration of solutes result in a minimum boiling temperature around 40°C. The maximum inlet temperature is set by the product properties, usually ~70°C.

In the MVR heating system, the vapour released by the product is compressed by means of a heavy-duty fan or, more rarely a compressor. The compressed vapour condenses at the outside of the pipes. As all vapour from the product leaves the boiler after transferring its latent heat of condensation at the tubes, the system is highly efficient with regard to energy consumption. Modern heavy-duty fans can raise the steam condensation temperature up to 6°C (Anonymous, 2008; www.piller.de/exhaust_vapour_compress.html). However, at start-up, there is no steam from the product; therefore, steam is injected, and the comparative parameters of the TVR and the MVR are as shown in Table 3.1.

Properties of the milk derivatives, such as solubility products, are highly dependent on the temperature and degree of concentration. Therefore, the fixed temperature in an MVR can be advantageous or disadvantageous, depending on the product made, with respect to the fouling.

The MVRs are often used to increase the capacity of existing TVR systems. As the power consumption increases at higher dry matter contents, due to the boiling point elevation, the MVRs are used in the first sections.

Table 3.1 The main technical differences between thermal vapour recompression (TVR) and mechanical vapour recompression (MVR) heating systems.

	TVR	MVR
Main heat source	Steam	Electricity
Energy consumption per tonne water evaporation	0.15–0.25 tonne steam/tonne	10–20 kWh/tonne
Product temperature	Range	Constant
Heat sink	Necessary	Unnecessary
Robustness	No moving parts in evaporator boiler, and vacuums leaks can be partly compensated by increased temperature gradients	Large high-speed fan is vulnerable to imbalance, and vacuum leaks destroy basic principle
Maintenance costs	Limited	Rotating parts
Capacity	Fixed	More flexible due to frequency-controlled fan
Temperature of released water	Range	Fixed
Effect of fouling on performance	Fouling can partly be compensated by a larger temperature drop over the pipes	Pressure drop by fan is limited, fouling decreases maximum dry matter content
Limitation in maximum dry matter content	Viscosity	Viscosity or boiling point elevation

3.2.3 *Plant design of evaporator configuration*

Prior to the evaporation process, the product is heated. This has two aims: (1) it brings the product to a temperature above the boiling temperature of the first stage and (2) the heating is also used to inactivate some micro-organisms. Depending on the product, the prior heating can be through pasteurisation as in the case of whey, but ultra-high temperature (UHT) sterilisation, as in infant food, is also used commonly.

In the case of using the MVRs, the condensate is usually used to heat the incoming feed. In the case of using the TVRs, this can be done also, but the temperature of the condensate of all stages together is rather low; or when only the condensates of the first stages of the TVR are applied, at which temperatures are high, the flow of incoming feed and condensate are unbiased.

The concentrate can also follow different process flows. For example, for shipping the whey concentrate, it is cooled down to temperatures below 10°C; for spray drying, the product ends up in a buffer tank. The runtime of an evaporator is much lower than that of most spray dryers. Depending on the product being concentrated, the runtimes may vary from 4 to 20 h (Walstra & Jellema, 1985), whereas spray dryers can operate for a period of 1 or 2 weeks or more. The downtime of the evaporator is usually bridged by two or more buffer tanks.

The runtime of the evaporators is limited by fouling and microbial growth. The poor solubility of calcium salts at elevated temperature and the concentration of the salts are the main reasons for the fouling in the case of whey. In the case of milk, denaturation of the proteins is an important factor. Fouling is machine-dependent and typical causes of fouling are (a) suboptimal control of the start-up and shut-down, (b) design and plugging of distribution plates and (c) insufficient flow over the pipes. In the last sections of the TVRs and next to the condenser, the temperature is ideal for the growth of many micro-organisms. The runtime of evaporators can further be reduced by microbial growth of spore-forming bacteria, especially when remnants of product stay behind after the cleaning.

The condensate released by the evaporator can be of excellent quality and it may be utilised for many purposes. For example, as it is free of calcium ions; it is suitable as feed for the steam boiler. It is used as a cleaning liquid also due to the absence of calcium ions, as this is a desirable feature. The water, however, in spite of its low organic pollution, is vulnerable to microbial growth due to its elevated temperature. Moreover, the quality of the condensate can fluctuate depending on the feed, operational conditions and design of the evaporator. The latter aspect has often led to the underutilisation of this potential. It should be noted that the condensate of TVRs not only contains water vapour from the product but also a significant amount of steam condensate; the proportion depends, of course, on the number of evaporation stages. Sometimes the condensate of the first sections is kept separate from that of the last. The pollution in the latter stages is usually higher.

In the TVR evaporators, their share of the steam consumption is significant. In cheese-making plants, the TVRs are the largest steam consumers by far when concentrating the whey.

3.2.4 *Heat economy in evaporator installation*

The TVR heating systems nullify the steam released by the product. The destruction of the latent heat of condensation makes the TVRs worse than MVR systems with respect

to energy consumption, where all latent heat of condensation is utilised. TVRs are often equipped with a thermocompressor where the steam jet pump uses the steam pressure generated by the boiler to boost the low-pressure steam generated by the first stage. This system can, depending on the steam pressure available, count approximately for one extra stage with respect to steam consumption. Therefore, the overall steam consumption of the evaporator itself is 1/(number of stages and number of thermocompressors) tonne per steam tonne water evaporation. The mixing of the condensate from the various sections yields an average temperature much lower than that of the feed. To compensate, an additional steam input is necessary. Estimating a temperature difference between the entrance temperature of the feed and the average outlet temperature of the condensate, of 30°C, an additional steam consumption of 50 kg steam/tonne feed is needed.

The energy input of the MVR comes, apart from the start-up with steam, from the electric power supply. This energy source is more expensive than the steam kilowatt per hour; however, the efficiency of the MVR system is far better, leading to an overall better energy consumption figure. Depending on the installed heated surface area, gas content of the feed, the boiling point elevation of the product and the efficiency of the fan, the power consumption lies between 10 and 20 kWh/tonne water evaporation. In addition, the installation of a plate heat exchanger (PHE), in which the heat load of the condensate and the feed are in counter-current exchange can minimise the heating demands. The net steam demand for the feed is estimated at 15 kg of steam/tonne, depending on the ratio of the concentrate and feed.

3.2.5 *Cleaning of evaporators*

Cleaning in the dairy industry is of eminent importance. Evaporators used in the dairy industry are no exception to this. Evaporators in the dairy industry operate under conditions, especially with respect to temperature range and concentration, which enhance fouling.

The size of the equipment and its corresponding hold-up, the limited run time and the concentrated product introduce considerable product losses at cleaning. Bouman (1985) reported that after 20 h of operation, whole milk losses at m^{-2} heat-exchange surface reached 1.3 kg in a 4-effect evaporator and 1.5 kg in a 7-effect evaporator. For a large evaporator, with several thousand square metres of heated area, each cleaning will cost several tonnes of product. Besides product losses and the corresponding disposal costs, energy consumption and consumption of chemicals during cleaning contribute to the operational costs. Another aspect that should not be neglected is the downtime of the evaporator. The cleaning of this large equipment takes significant amounts of time, usually 2–3 h (Brinkman & van Voskuilen, 1992), which takes cleaning-in-place (CIP) capacity and often requires costly cooling of intermediate products, for example whey, to maintain microbial quality prior to evaporation.

The composition of the deposits formed in an evaporator depends on the product evaporated. Milk deposits are presented as a matrix of protein with which minerals are associated, and in which fat is embedded (Jeurnink & Brinkman, 1994). The mechanism of the milk deposit removal was studied in detail by Jeurnink & Brinkman (1994). According to them, the best results can be obtained by the following procedure: (a) a pre-rinse to remove residual milk, (b) an alkaline cleaning, (c) an intermediate rinse, (d) an acid cleaning and

(e) a final rinse. For removal, two mechanisms are important: first, swelling of the deposit after contact with the alkaline solution and, second, mechanical action caused by the shear stress of the flowing cleaning solution. It appears that the cleaning solution penetrates into the spongy structure of the deposit layer, causing a swelling from which cracks propagate, which in turn accelerates further penetration of the cleaning solution. Cracks appear, especially near the stainless steel wall, because the deposit layer swells but the stainless steel does not. The deposit is subsequently carried away in large lumps with the cleaning solution. The alkali concentration and the flow rate are both of major importance. The alkali concentration should not be increased above 1 g 100 g^{-1}, as it turns the protein into an impermeable gel or polymer, which hampers penetration of the cleaning fluid and, in turn, dramatically slows down the cleaning rate (Jeurnink & Brinkman, 1994). A flow rate slightly above the maximum feed rate of the product is usually enough. Good results were obtained by cleaning at temperatures of 70°C.

Whey deposits are represented as a complex of protein together with calcium salts, containing mainly calcium phosphate (Jeurnink & Brinkman, 1994) in the case of dilute whey. The deposit of concentrated whey contains a considerable amount of calcium citrate. Jeurnink & Brinkman (1994) compared two different cleaning procedures for whey. The results of an acid followed by an alkaline cleaning were far better than an alkaline followed by an acid cleaning (Fig. 3.3a, b) (Jeurnink & Brinkman, 1994). It resulted within a couple of minutes in sharp peaks of concentrated fouling. The peaks of fouling are discarded, whereas the rest of the cleaning solution is returned to the CIP system. On-line monitoring of such peaks, for example using NIZO's Opti Cip+ system that monitors both calcium concentration and the non-dissolved parts (Asselt *et al*., 2002), can save cleaning fluids by recirculation. It is also useful in monitoring the fouling behaviour of the evaporator.

The wetting rates in an evaporator should ideally be high during cleaning. This is difficult to achieve at the downside of the distribution plates. These plates easily get fouled, especially in the case of flash evaporation. In the cleaning procedure, there is usually no flashing at entrance, as concentration of the alkaline solution should be prevented and cooling of the cleaning solution is not desired. The situation is worsened by support pins for the distribution plates. Remnants of deposits often stay behind after the cleaning. These spots are excellent hiding places for spore-forming thermophilic bacteria, which can flourish in the subsequent run.

The cleaning of the collection bowl and the gas–liquid separator devices is done by nozzles.

3.2.6 *Evaporation versus membrane filtration*

Fat globules in milk are easily damaged in RO membrane filtration systems (Walstra & Jellema, 1985); therefore, concentration of whole milk is mostly done in evaporation systems. For most other products both techniques may be applied. Evaporation and membrane filtration do both have their own benefits. Although large-scale factories have been built on either technique, often the techniques are combined. Some relevant technical information regarding the concentration of a product using the evaporation and membrane filtration systems is shown in Table 3.2.

The first concentration is often done in a membrane filtration system. In the past, this was usually carried out using RO; nowadays, NF is often used. The higher flux of NF

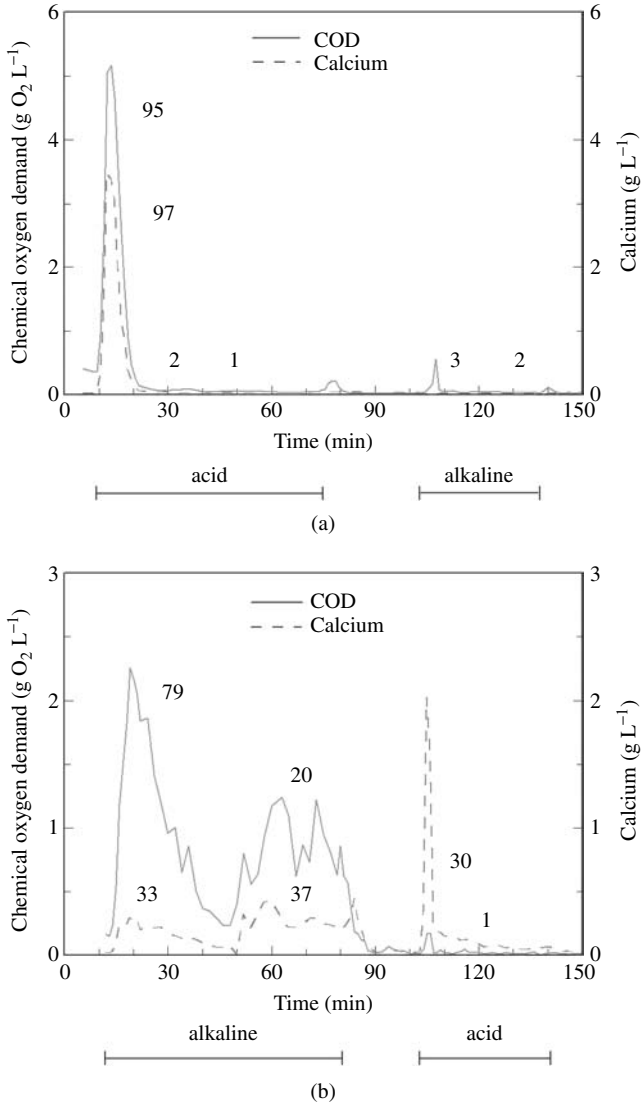


Fig. 3.3 Whey deposit removal gram per litre in an evaporator after operating for 20 h using (a) pre-rinse – alkaline wash – rinse – acid wash – final rinse or (b) pre-rinse – acid wash – rinse – alkaline wash – final rinse. Note: Cleaning temperature was 70°C, the cleaning solutions were 1 g 100 $g^{-}1$ NaOH and 1 g 100 $g^{-}1$ HNO_3. After Jeurnink & Brinkman (1994), and reproduced with permission of Th.J.M. Jeurnink.

membranes in comparison to RO is a benefit, but the main advantage is that, by using NF, the whey can be demineralised and concentrated simultaneously to approximately 25 g 100 g^{-1} total solids and 47 g 100 g^{-1} demineralisation (van der Horst, 1995). This can replace the traditional method of concentration by evaporation and electrodialysis or ion exchange. However, product properties and plant layout are decisive with respect to choice of the unit operations.

Table 3.2 The main technological and operational difference between an evaporator and membrane filtration system to concentrate milk and related products.

	Membrane filtration	Evaporation MVR
Power consumption	2–3 kWh/tonnes water	10–20 kWh/tonne water
Building height	Limited <3 m	10 m and more
Cleaning	Special cleaning agents	CIP
Operational temperature	<10°C 50–60°C	55–70°C
Removed water quality	Depends on the life time of membrane and concentration level	Constant
Concentration level	<10 mPa.s	<100 mPa.s
Downscaling/testing	Small units can be rented easily for *in situ* tests	Significant installation costs needed for *in situ* testing, almost no equipment for rent

MVR = mechanical vapour recompression; CIP = cleaning-in-place.

3.3 Membrane filtration technology

3.3.1 *Principles of membrane filtration*

Milk is a constituent of many foods and food products. The functionality of the various components in milk could be utilised more effectively if they were available separately. Fractionated milk components enable a more constant quality of consumer products, for example cheese, and the development of new products, such as edible coatings and bioactive peptides. Therefore, milk fractionation will lead to a more efficient and diverse use of milk. Membrane separation technology (i.e. RO, NF, UF and MF) seems a logical choice for the fractionation of milk, because many milk components can be separated on size (Brans *et al*., 2004).

In all the membrane filtration processes, there is a separation of fluid mixtures by selective permeation through a semi-permeable membrane under a pressure gradient, where particles are separated according to their dimensions (Carić, 1994). Figure 3.4 illustrates the spectrum of application of membrane separation processes in the dairy industry (Anonymous, 2003). There is no sharp division between the processes (Fig. 3.4); membrane pore size and the applied-pressure difference are often used to distinguish the processes from each other. In MF, the hydrostatic pressure differences used are in the range of 0.01–0.1 MPa; the UF operates at a slightly higher pressure, usually between 0.01 and 0.055 MPa. The operating pressure in RO is between 2 and 10 MPa (Carić, 1994). NF, also called loose RO, operates at a pressure between UF and RO, that is, 2–4 MPa (Mistry & Maubois, 2004). The general flow patterns of the various membrane separation systems are illustrated in Figure 3.5 (Anonymous, 2003).

The flux by reverse osmosis (RO) is proportional to the difference between applied hydrostatic pressure and osmotic pressure of the feed. It can be expressed by the following relation:

$$J_w = \frac{\Delta P - \Delta \pi}{R_c + R_m}. \tag{3.2}$$

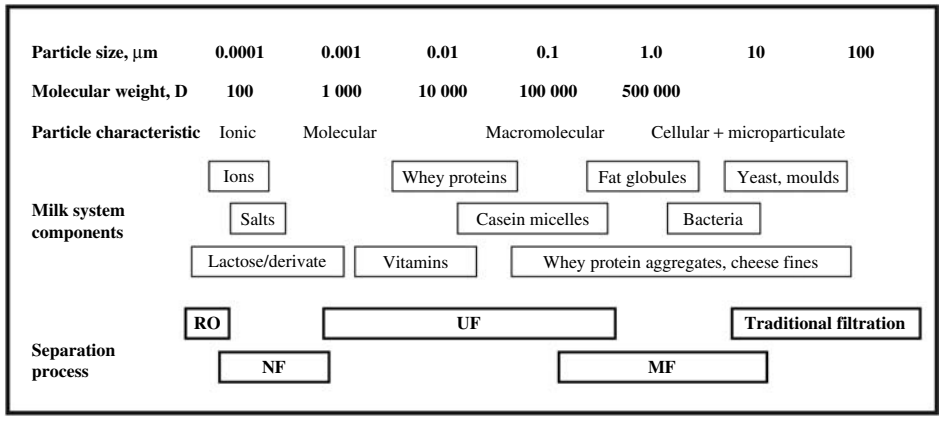


Fig. 3.4 Spectrum of application of membrane separation processes in the dairy industry. (Anonymous, 2003). Reproduced with permission of Tetra Pak A/B, Lund, Sweden.

J_w is the filtration rate, R_c is the resistance of the cake, which accumulates on membrane surface, R_m is the resistance of the membrane and ΔP and $\Delta\pi$ are the differences in the hydrostatic and osmotic pressures between the feed solution and the filtrate.

The osmotic pressure of any species may be approximated for dilute solutions by the van 't Hoff equation:

$$\pi = n\ CRT \tag{3.3}$$

where C is the molar concentration of the solute, R is the gas constant, T is absolute temperature and n is the number of ions after dissociation (e.g. for NaCl, $n = 2$; for $BaCl_2$, $n = 3$).

NF, UF and MF are conceptually very similar to RO. The main difference between the processes is the degree of permeability of the membranes and, consequently, in the size of separated particles (Carić, 1994). In NF, low-molecular-weight compounds (200 and 1000 Da), such as dissolved mineral salts are removed, whereas other organic materials are concentrated. UF and MF membranes allow small molecules to permeate together with water molecules, while macromolecules (UF membranes) and colloids, bacteria and suspended particles (MF membranes) are concentrated. For high molecular weights, the molar concentrations are small and the pressure difference across the membrane is generally much lower for UF and MF than for the RO. Therefore, equation 3.2 becomes

$$J_w = \frac{\Delta P}{R_c + R_m}. \tag{3.4}$$

When solvent permeates the membrane, the retentate concentration at the feed side is increased. Under steady state conditions, the convective mass transfer due to filtration is equal to the diffusive transport in the opposite direction caused by concentration-gradient driving force:

$$J_w C - D\frac{dC}{dx} = 0 \tag{3.5}$$

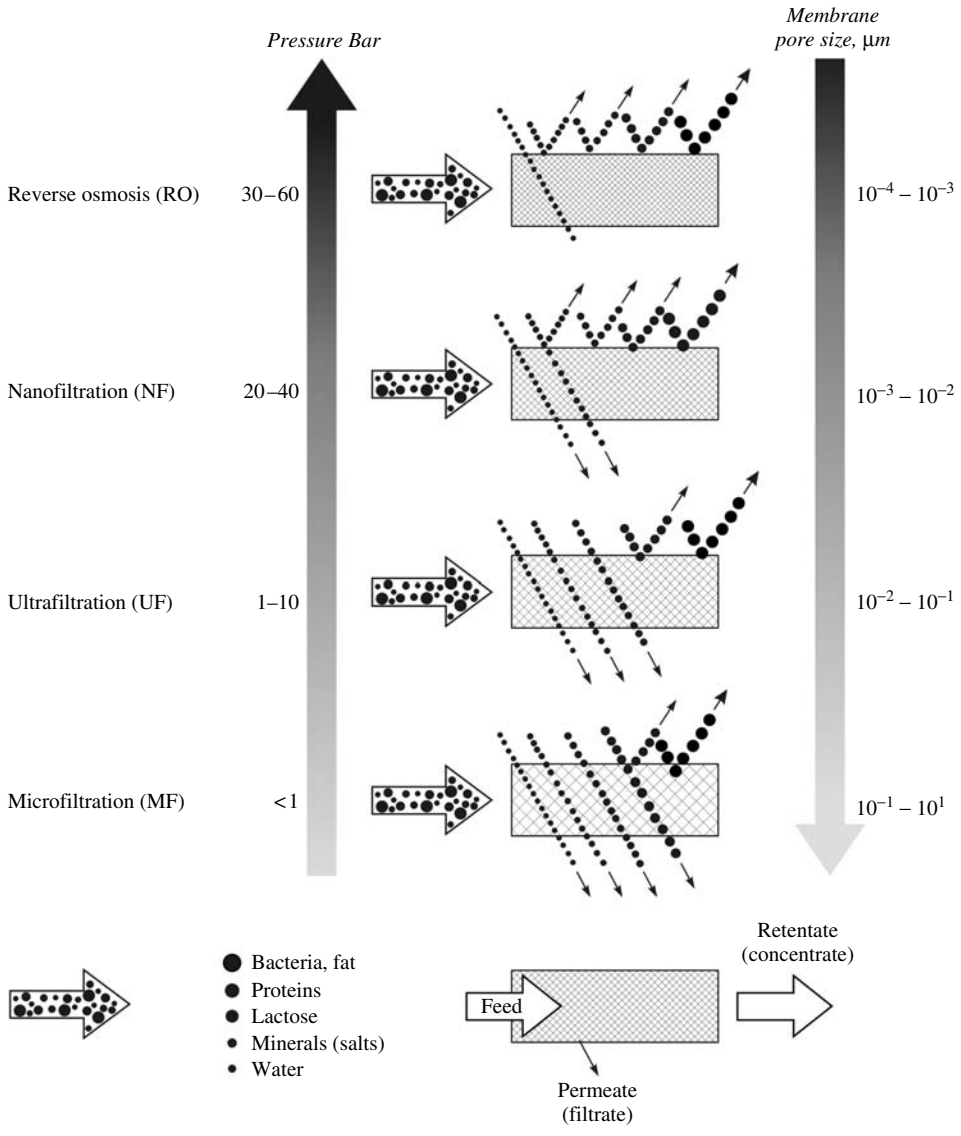


Fig. 3.5 Principles of membrane filtration. (Anonymous, 2003). Reproduced with permission of Tetra Pak A/B, Lund, Sweden.

where D is the solute diffusibility, C is the solute concentration and x is the distance from the membrane surface. Equation 3.5 can be integrated across the bound layer to give

$$J_w = \frac{D}{\delta} \ln \frac{C_s}{C_b} \tag{3.6}$$

where δ is the stagnate boundary layer thickness. C_s and C_b denote solute concentrations at the membrane and in the bulk of solution, respectively. Mass transfer coefficient, K,

replaces D/δ and equation 3.6 becomes

$$J_w = K \ \text{In} \ \frac{C_s}{C_b}. \tag{3.7}$$

In practice, membrane filtration becomes technically feasible only after the development of asymmetric membranes and introduction of cross flow (CF) filtration. In such of an arrangement, the feed flows tangentially over the surface of the membrane sweeping retained molecules away from the membrane (Carić, 1994, 2004).

Membrane filtration data could be expressed as a concentration factor (UF degree), which is the volume or weight reduction achieved by the concentration:

$$F_c = \frac{Q_m}{Q_r} \tag{3.8}$$

where F_c is the concentration factor, Q_m is the quantity of initial feed (L, kg) and Q_r is the quantity of the retentate (L, kg).

The concentration factor can also be expressed as the ratio of the final to the initial concentration of any of the components retained by the membrane. The essential component of milk concentrating in the retentate is protein. Thus, the concentration factor in dairying is often expressed as the ratio of protein in the retentate and in the feed,

$$F_c = \frac{P_r}{P_m} \tag{3.9}$$

where P_r is the concentration of protein in the retentate (g 100 g^{-1}) and P_m is the concentration of protein in milk or whey (g 100 g^{-1}).

Permeability (P) is the ratio between the volume of the permeate side and the retentate side of the membrane. It is usually expressed as a percentage:

$$P = \frac{V_p}{V_f} \tag{3.10}$$

where V_p is the volume on the permeate side and V_f is the volume on the feed side.

Rejection or retention (R) is the ability of the membrane to retain molecules, defined as

$$R = \frac{V_f - V_p}{V_f} = 1 - \frac{V_p}{V_f} = 1 - P. \tag{3.11}$$

DF is a particular mode of operation of UF and MF plants in which small molecules are washed out from a retentate stream. DF is usually performed continuously when wash liquid (deionised water) is fed continuously at the rate equal to that at which permeate is removed from the system. A typical example is concentrating either skimmed milk or whey to reduce the lactose content in the retentate.

All membrane filtration techniques feature CF filtration, in which the feed solution is forced through the membrane under pressure. The solution flows over a membrane and the concentrate (retentate) is retained while the permeate is removed. The membranes are categorised by their molecular weight cut-off; supposedly, the molecular weight of the smallest molecule will not pass through the membrane. However, owing to various interactions,

a membrane cannot be selected purely on the basis of molecular weight cut-off. As a matter of form it should be mentioned that traditional or conventional filtration (dead-end filtration) is usually used for separation of suspended particles larger than 10 μm, while membrane filtration separates substances of molecular sizes less than 10^{-4} μm (Anonymous, 2003).

Current membrane processes for milk have a rather low capacity due to strong flux decline by fouling, or processes are energy demanding because of the high CF velocity that is required to control fouling. In addition, methods to control fouling have increased the complexity in equipment and operation. A number of factors are important in achieving a good combination of retention and transmission of components in the feed. The first is uniformity in membrane pore size. Due to polydisperse pores, components that should be retained pass the membrane through the larger pores. Smaller pores cause retention of components that should be transmitted. Using traditional polymer membranes, milk fractions could not be obtained in the desired purity, probably because of the wide pore-size distribution. Secondly, it is important that the process conditions are similar over the whole membrane area. This has led to the development of the uniform transmembrane pressure (UTP) concept and isoflux and gradient porosity (GP) membranes. The introduction of ceramic membranes and the use of the UTP concept enabled the commercial application of membranes for the reduction of bacteria and spores in milk and other dairy products. The UTP concept maintains a constant transmembrane pressure over the length of the module by applying a CF at the permeate side. Isoflux and GP membranes have a spatial change in the membrane resistance to ensure uniform process conditions. The depth of fouling leads to smaller effective pores and to different retention characteristics. Cake layer formation leads to a different retention behaviour, as the cake retains small particles that should pass the membrane (Brans *et al*., 2004).

3.3.2 *Membrane filtration techniques and systems*

Membrane filtration is carried out in different module configurations, such as plate and frame, hollow fibre, spiral wound and tubular. Each type of module consists of membranes whose characteristics determine the processing efficiency. Particularly important membrane characteristics include pore size, pore-size distribution and the type of macromolecular material of which the membrane is composed. Practical requirements for membrane materials are (a) good film formers; (b) very thin film that minimises resistance to permeate flow; (c) hydrophilic groups that bind water molecules so that they will not enter the membrane and (d) high swelling ability and strength in the wet state (Carić, 1994).

The first generation of membranes consisted of cellulose acetate, which was limited in temperature and pH tolerance (e.g. $t < 50^{\circ}C$ and $3 < pH < 8$). Also, they were susceptible to micro-organisms and disinfectants. However, the second generation of membranes were made of synthetic polymers (mainly polysulfone or polyolefin derivatives) permitting a wider range of working temperature (up to 100°C) and pH (2–12). These membranes were susceptible to damage by some disinfectants and mechanical stress. The most widely used membrane material in the dairy industry is polysulfone.

Currently, the third generation of membranes are mineral in type and manufactured from zirconium oxide on a graphite support. Temperature tolerance is up to 400°C and pH tolerance covers the whole range. The new membranes are highly resistant to mechanical

stress and high pressure. It is possible to obtain very high protein concentrations using these membranes. The advantage of these membranes for high temperature usage may not be recognised in dairy processing as milk components are thermosensitive, especially whey proteins, which are denatured at temperatures higher than 60°C.

The significant growth of industrial membrane processes was made possible with the development of asymmetric membranes, where only the thin surface layer is an active part of the membrane. Being very thin, it permits much better water flux than earlier membranes. Permeate components that pass the active layer will pass the supporting porous layer easily. Deposit formation (fouling) is markedly decreased, and it is limited to the surface of the membrane (Carić, 1993, 1994, 2004).

The plate-and-frame membrane system is composed of oval plastic support plates between which membranes in pairs are placed. The plates have a pattern of curved ribs so that when the two plates are pressed together with a membrane in between, flow channels are formed. Fine parallel straight slits are engraved in the surface of the membrane, through which the permeate is led to the edge of the plate; such construction is simple to control and enables individual replacement of faulty membrane pairs (Carić, 1994). Plate-and-frame modules are used for UF and RO separation techniques and the membrane materials are polymers (Anonymous, 2003).

The hollow fibre module consists of a bundle of 1000 or more tightly packed hollow fibres with an internal diameter of 0.5–1.4 mm. The fibres are about 1 m long, grouped and sealed at each end in an epoxy plate. The feed passes through the centre of the tubes, allowing the permeate to exit radially. A standard module has a 2.5 m^2 active membrane area. In case of membrane damage, the module can be successfully repaired without replacement. The main advantage of this module type is cleaning by back flushing to remove deposits (Carić, 1994). In addition, hollow fibre modules are used in UF applications and the membranes are made of polymers (Anonymous, 2003).

By contrast, the spiral wound module is composed of membranes separated by a sheet of plastic mesh 1 mm thick. On both sides of a membrane, two layers of absorbent material are used to collect permeate. All five layers are then rolled to form a spiral, and the spaces between the membrane and the absorbent layer are sealed. A perforated stainless steel tube is inserted into the centre of the roll. The permeate flows radially, both through the membrane and the absorbent material and through the perforations in the central tube out of the module. Commercial spiral wound modules are 0.9 m in length, 0.1 m in diameter and the membrane area is ~3 m^2. The advantage of this module type is low hold-up volume and large membrane area (Carić, 1994). Membrane and permeate spacer material is polymer and a spiral wound module is used for NF, UF and RO filtration systems (Anonymous, 2003).

A tubular membrane module is an assembly of tubes, ~3.6 m long, with a membrane area of 2.5 m^2. One tube consists of a membrane of a synthetic fibre inserted into a perforated stainless steel tube. The membrane insert tube is replaceable and the whole assembly of tubes is put together with an end cap (Carić, 1994). Tubular filtration systems based on polymers are available for UF and RO applications, while a tubular module with ceramic membranes is used for MF and UF (Anonymous, 2003).

A relatively newly developed process is the vibration membrane system, which is suitable for the separation of milk proteins. In this system, the vibration energy is used to improve the flux rate (Mistry & Maubois, 2004).

3.3.3 *Membrane filtration configurations*

Membrane filtration plants are designed for continuous or batch operation. The latter type is of a small capacity and used only by small- to medium-size dairy processors. A schematic illustration of a batch plant is shown in Figure 3.6a. The advantage of such a unit is its simple operation. However, its energy costs are relatively high due to the circulation of concentrate from the module to the feed tank at atmospheric pressure and back into the module under pressure.

In a continuous unit operation 'feed and bleed' (see Fig. 3.6b), the milk is re-circulated through a number of modules, connected in a series. Every module in this assembly functions according to a specific constant concentration rate that is always higher in the subsequent module. Some of the concentrate is pumped back to the start of the inner loop where more feed is added to compensate for the volume of concentrate removed. The

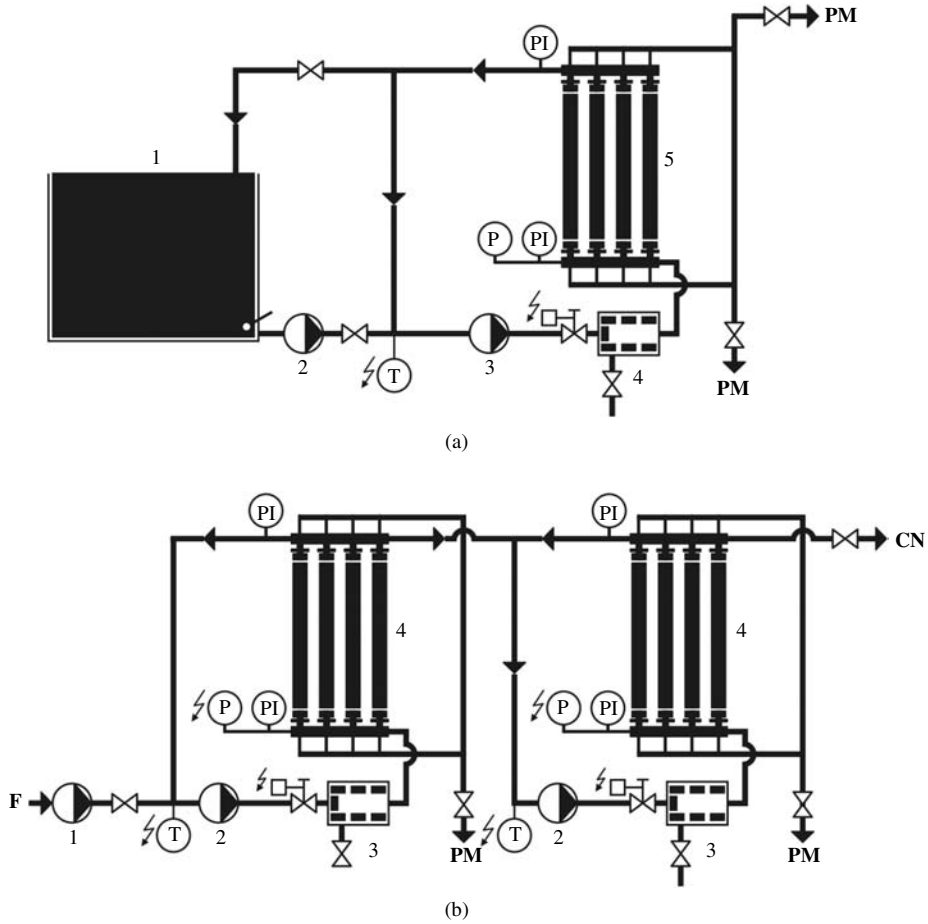


Fig. 3.6 Illustrations of ultrafiltration (UF) plant. (a) Batch system: 1, balance tank; 2, feed pump; 3, circulation pump; 4, filter; 5, UF modules; PM = permeate. (b) Continuous system: 1, feed pump; 2, circulation pump; 3, filter; 4, UF modules. F = feed; PM = permeate. After Carić (1994).

concentration rate of the final product is adjusted by increasing or decreasing the quantity of the product inlet. Two main advantages of the continuous method are (1) less pumping energy is required, that is, lower operation costs and (2) better microbial quality because of the shorter residence time of the product during operation.

For example, a UF configuration unit is composed of a battery of UF membrane modules, a heat exchanger, a filter, storage tanks, pumps (for feed, recirculation permeate, retentate and cooling water), pipelines, valves and automatic control systems for pressure, temperature, flow and dry matter measuring units. The RO configuration unit is composed of a battery of RO membrane modules, filter, storage tanks, high-pressure pump, pipelines, valves and pressure and temperature indicators (Carić, 1994). MF configuration is based on two units operated in series in a filter loop system which contains one centrifugal pump for circulation of the retentate and one for circulation of permeate (Anonymous, 2003).

3.3.4 *Heat economy in membrane filtration*

Membrane processes are not associated with phase change and are energy sparing in relation to conventional concentration process-like evaporation or freezing, eliminating the use of large volumes of cooling water. No heat is required to accomplish phase change, minimising the risk of damaging thermolabile foods and avoiding flavour loss that is the result of the elimination of volatile substances (Castro & Gerla, 2005).

In general, the energy consumption for membrane filtration plants depends mainly on capacity, pressure, concentration factor, rate of recirculation and viscosity of the feed. In the dairy industry, membrane filtration plants are usually combined with plants for evaporation and/or drying to achieve the maximum heat economy.

3.3.5 *Application of membrane filtration in the dairy industry*

The four types of module construction for membrane separations have been developed for commercial applications and practically all have successful application in industrial engineering. In addition to concentration and fractionation of whey and milk, membrane separation has been introduced into industries related to foods, pharmaceuticals, textiles, sea water desalination and waste water treatment (Carić, 1994).

An important UF application is in the processing of various dairy products such as fermented milks (yoghurt, ymer), certain varieties of cheese (Feta, Camembert, Ricotta, Mozzarella) and whey protein concentrates (WPC) (Carić, 1994). However, RO is used for the concentration of whey, UF permeate and condensate; whilst one application of NF is the practical desalination of whey, UF retentate or permeate. Finally, MF is basically used for bacterial reduction in skimmed milk, whey and brine as well as for defatting whey intended for the production of WPC and for protein fractionation (Anonymous, 2003).

In general, for further industrial application, the capacity of membrane processes should be increased; appropriate fouling control is also important regarding the economics of the process. Although flux enhancement has got a lot of attention in terms of understanding and practical improvement (with inserts and back pulsing), achievement of a high selectivity (full retention of large components and full transmission of small ones) deserves more attention. High selectivity is not only dependent on the membrane but is just as

much dependent on the process conditions and interactions between the components in the feed. New types of membranes with narrow pore-size distribution are currently becoming available on the market (Brans *et al*., 2004).

Application of membrane separation techniques in cheesemaking is reviewed in detail by Mistry & Maubois (2004). The use of membrane filtration technology in processing of dried milk products, dried whey and whey-based products [WPC, whey protein isolates (WPI)], novel dried products and infant formulae are detailed in Chapters 7, 8 and 9.

3.3.6 *Cleaning of membrane filtration systems*

Background

Membrane filtration systems inevitably become fouled after a period of operation. Fouling in dairy systems can be generally classified as follows:

- Proteinaceous fouling is caused by the deposition of both native and denatured dairy proteins.
- Calcium phosphate is the predominant salt foulant in milk systems and can precipitate in several forms. Although, this may represent only a minor component of the total deposit mass, it can have a disproportionate effect on total flux decline and the ease of fouling removal. This is suspected to be due to calcium forming protein–protein and protein–membrane bridges, resulting in dense and strongly bound layers (Marshall & Daufin, 1995).
- Lactose has been shown to be of little importance (Kulozik, 1995) and lipids are generally regarded to be of lesser importance, except for feed streams where the lipid content is initially high (Marshall & Daufin, 1995).

Over the past couple of decades, extensive material have been published on the different aspects of cleaning of membrane filtration systems and the following are recommended for further reading (Smith & Bradley, 1988; Tzeng & Zall, 1991; Bohner & Bradley, 1992; Eckner & Zottola, 1993; Bird & Espig, 1994; Bird & Bartlett, 1995; Bartlett *et al*., 1995; Cabero *et al*., 1999; Lawrence *et al*., 1999; Saboya & Maubois, 2001; Huiting *et al*., 2002; Tran-Ha *et al*., 2002; Clark, 2003; Raspe, 2004; Blanpain-Avet *et al*., 2004a, 2004b, 2004c; Madaeni & Mansourpanah, 2004; Kumar & Anand, 2004; Milnes, 2005; Mourouzidis-Mourouzis & Karabelas, 2007; Askew *et al*., 2008).

Fouling minimisation/control

In the first instance, strategies can be put in place to delay the onset of fouling and/or reduce the extent. For example, Matthews *et al*. (1978), Smith & MacBean (1978) and Hickey *et al*. (1980) all showed that increasing the temperature and the pH of dairy feeds prior to UF improved the flux by forming bulk calcium phosphate crystals, thus preventing their precipitation inside the membrane pores during filtration. Similarly, citrate chelates calcium more strongly than phosphate, and thus the addition of this compound to a dairy feed solution can reduce the concentration of calcium ions and colloidal phosphate available in the bulk system (Fox & McSweeney, 1998).

Furthermore, fouling is the limiting factor in all applications of membrane filtration of milk and whey (Carić *et al*., 2000). Different fouling mechanisms can take place, for example, adsorption, pore blocking, cake layer formation and depth fouling. Concentration polarisation also decreases the flux and can affect selectivity. The insertion of the Kenics static mixer, as a turbulence promoter during skimmed milk MF, improves the permeate flux more than 700% at the same feed flow rate (Krstić *et al*., 2003, 2004). The choice of a method for fouling control must be technically and economically feasible, scalable to production output and well suited for CIP. A summary of methods to enhance membrane performance, principles and possible disadvantages is shown in Table 3.3 (Brans *et al*., 2004).

From a hydrodynamic perspective, operation below the critical flux is well known to limit cake formation (Howell, 1995). Similarly, use of a gradual, stepwise increase in operating pressure during start-up can result in a more labile polarised layer that flows more easily under shear (Chen *et al*., 1997). At high-solid concentrations, the use of a constant (low) pressure driving force achieved by permeate recycle or the use of tapered membranes is useful for ceramic membrane systems (Gesan *et al*., 1993, 1997). Similarly, turbulence promoters are common in polymeric systems. Other hydrodynamic methods of reducing fouling rates include periodic reversal of the permeate flow (back pulsing) and reduction or oscillation of the feed pressure (Finnigan & Howell, 1990; Nystrom & Howell, 1993).

Table 3.3 The advantages and disadvantages of different methods used to enhance the performance of a membrane filtration system.

Method	Working principle	Possible disadvantage
High CF velocity with UTP concept	Enhance back transport by turbulent flow; low transmembrane pressure	High power consumption, high investment and operating costs
Turbulence promoters	Enhance back transport by microturbulences close to membrane	Difficult cleaning (hygiene), increased power consumption
Back pulsing (-washing, -flushing, -shocking)	Remove cake by reversing the transmembrane pressure	Upscaling, difficult to control pressure in large systems
Pulsating CF	Create velocity fluctuations in the feed to promote back transport	Upscaling, difficult to control pressure waves in large systems
Air slugs	Increase shear and mixing close to membrane	Difficult to control air bubble size, foaming and denaturation
Scouring particles	Increase shear and mixing close to membrane	Wear of membrane and pumps, denaturation
Acoustic/ultrasonic waves, sonication	Promote back transport of deposits by vibrations and cavitations	Power consumption and heating, damage to sensitive compounds
Vibrating modules	Increase shear close to membrane	Upscaling, expensive equipment
Rotating disk	Increase shear close to membrane	Upscaling, aseptic seals
Electric fields	Introduce electric force field to keep charged particles from membrane	Electrolysis, gas production, heating and power consumption

CF = cross flow; UTP = uniform transmembrane press.
Adapted from Brans *et al*. (2004).

The use of vibratory shear-enhanced filtration (VSEP) is a more recent approach. This is a commercialised technology that uses a sequence of parallel disc membranes located above a torsion spring that moves the stack back and forth at around 50–60 times s^{-1} (Petala & Zouboulis, 2006). Flux enhancements of 300–400% have been observed in these systems relative to a standard spiral wound membrane (Akoum *et al*., 2003a, 2003b, 2004). The disadvantage of this system is that the use of disc membranes limits the membrane area that can be provided (i.e. up to 151 m^2) (Akoum *et al*., 2005) and leads to a relatively large processing volume per unit of membrane area. Rotating disc units produce similar effects (Bouzerar *et al*., 2003; Jaffrin *et al*., 2004; Frappart *et al*., 2006).

Another approach, which is yet to be commercialised, is the use of ultrasound to minimise 'cake' fouling; however, experimental results show that the use of ultrasonics at low power levels can significantly enhance the permeate flux of dairy whey solutions with an enhancement factor of between 1.2 and 1.7 (Muthukumaran *et al*., 2005a, 2007; Teng *et al*., 2006; Kentish & Ashokkumar, 2006). The use of an ultrasonic field of frequency between 30 kHz and 1 MHz induces localised flow disturbances. In addition, the variation of acoustic pressure can cause small microbubbles of air to form through the process known as cavitation. These bubbles grow under the influence of the acoustic field to a critical size and then collapse. The collapse events cause further turbulence on a microscopic scale. In particular, cavitation collapse at a membrane surface can generate microjets, which can scour a membrane surface.

Scanning electron microscopy (SEM), atomic force microscopy (AFM) and X-ray photoelectron spectroscopy were used to comprehensively characterise the surface of unused MF and UF membranes; the fouled layer on the surface of membranes used for skimmed milk filtration and the internal fouling within the used membranes. Using these complementary techniques it has been shown that internal fouling, during filtration of skimmed milk, proceeds by protein–polymer and protein–protein interactions (James *et al*., 2003).

Castro & Gerla (2005) examined the resistance to whey UF at bench scale using alternatively hollow fibre and spiral membranes. In the case of hollow fibre membranes, the principal resistance is due to the fouling inside the membrane's pores, while the major resistance when using the spiral cartridge originated from the polarisation of the concentration.

Membrane cleaning aspects

Irrespective of the use of fouling minimisation techniques, regular cleaning of membranes with chemicals is unavoidable. The purpose of membrane cleaning is usually two fold: (1) to restore membrane flux and (2) to reduce the microbiological load of the system, so as to remove all pathogens and to ensure dairy product quality. Disinfection of membrane surfaces is essential for aseptic processing.

The nature of the cleaning cycle is somewhat dependent on the nature of the fouling deposit. In general, membranes are cleaned daily, in a cycle that can take 2–3 h (Begoin *et al*., 2006a; Delaunay *et al*., 2006). All stages of cleaning are best conducted at minimal transmembrane pressures and maximum CF velocities (Bird & Bartlett, 2002). A typical cleaning cycle would commence with a simple rinse with water at elevated temperatures. As this rinse is conducted at lower transmembrane pressures and higher CF velocities than during the production cycle, it is effective in removing the remaining feed solution as well

as loose deposits on the membrane surface. This is generally followed by an alkali cleaning cycle, an acid clean and then a final cycle of alkaline–hypochlorite cleaning with water rinses between each cycle (Begoin *et al*., 2006a). Recently, Rabiller-Baudry *et al*. (2008) reported that the final water rinsing step is crucial, particularly with cleaning solutions containing surfactants.

Alkaline cleaning cycle

Alkaline detergents generally contain a combination of an alkaline agent (sodium or potassium hydroxide, NaOH or KOH) and a surfactant. An anti-foaming agent may also be included (D'Souza & Mawson, 2005). It is important to operate at the optimum pH where the protein is swollen to its maximum size (Bird & Fryer, 1991). Bird and Bartlett (2002) and Bansal *et al*. (2006) found that 0.2 g 100 g^{-1} caustic solution had the optimum cleaning, whilst Makardij *et al*. (1999, 2002) found that the optimum recovery was achieved with a caustic concentration of 0.4 g 100 g^{-1}. Rabiller-Baudry *et al*. (2008) indicate that an efficient solution would have pH 11.5, include surfactants to ensure an interfacial energy of less than 30 mJ m^{-2} and contain at least one surfactant with a strongly apolar contribution. Some operating staff tend to use more cleaning agent thinking that it is better, but this is an inappropriate strategy for an alkaline cleaner. At higher pH, the high ionic strength of the solution can cause the protein to shrink, making removal more difficult. An inordinately high cleaning pH will also shorten membrane life. Muthukumaran *et al*. (2005b) have shown that ultrasound can also be employed during the alkaline cleaning cycle to enhance flux recovery and/or reduce chemical demand (Fig. 3.7).

Bird & Bartlett (2002) reported an optimum temperature for caustic cleaning of WPC deposits of 50°C. An elevated caustic cleaning temperature enhances protein solubility and speeds up chemical reactions such as peptide bond hydrolysis. In addition, higher temperatures lead to a reduction in the cleaning solution viscosity and a corresponding increase in the Reynolds number. The same authors also reported that cleaning at temperatures

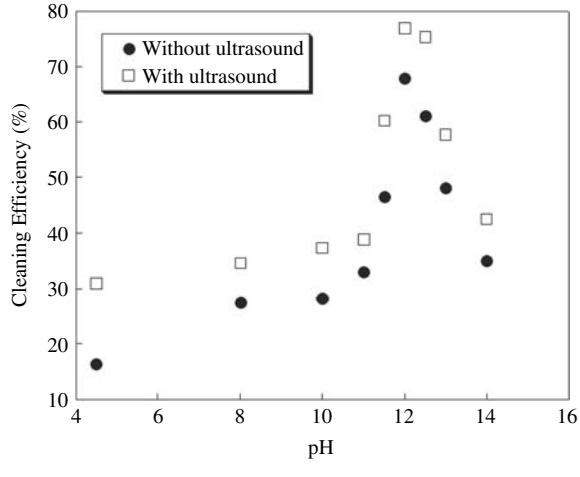


Fig. 3.7 Effect of solution pH on the cleaning efficiency of an ultrafiltration membrane fouled with a dairy whey solution in the presence and absence of ultrasound. Reproduced with permission from Muthukumaran *et al*. (2005b).

Table 3.4 Flux recovery of a polysulfone ultrafiltration membrane fouled with milk after cleaning with various chemicals at 30°C.

Cleaning agent	Flux recovery (%)
Sodium hydroxide (NaOH)	10
Sodium hypochlorite (NaOCl)	40
Ethylenediaminetetraacetic acid (EDTA)	8
Sodium dodecyl sulphate (SDS)	27
EDTA + NaOH	52
EDTA + SDS + NaOH	100

Note: the concentration of all chemicals was identical, but is not defined in the article. Data compiled from Kazemimoghadam & Mohammadi (2007).

beyond 50°C is less valuable. This could be due to reduced solubility of calcium phosphate deposits, as this compound shows an inverse solubility with temperature, or to swollen protein deposits blocking access to the internal pore structure.

Chelating agents, such as ethylenediaminetetraacetic acid (EDTA), are also often incorporated into the alkaline cleaning solutions (Begoin *et al*., 2006a). These agents act to remove the calcium content bound within the protein layer. Recent work by both Delaunay *et al*. (2006) and Kazemimoghadam & Mohammadi (2007) has confirmed the efficacy of a formulated alkaline cleaner that contains surfactants and possibly chelating agents, over that of pure NaOH. As shown in Table 3.4, a combination of chelating agent (EDTA), surfactant [sodium dodecyl sulphate (SDS)] and alkali (NaOH) is more effective than any of these chemicals in isolation. Similarly, Table 3.5 shows that there can be less protein remaining on the membrane after cleaning with a commercially formulated alkaline cleaner, and the flux through the cleaned membrane is also higher. However, these authors note that the surfactant(s) from Ultraclean II tended to adsorb on the polyethersulphone (PES) membrane and decreased the membrane hydraulic resistance by 50%. Consequently, the

Table 3.5 Permeability of membranes measured in water at 25°C and residual proteins (measured by FTIR-ATR) following fouling of a polyethersulfone ultrafiltration membrane with skim milk and cleaning at 50°C.

	Membrane permeability relative to that of a virgin membrane	Proteins remaining on cleaned membrane ($\mu g\ cm^{-2}$)
Fouled and rinsed	0.19	14
Fouled and rinsed then NaOH (pH 11.5) cleaned and rinsed	0.42	7
Fouled and rinsed then Ultrasil 10 (0.1 g 100 g^{-1}, Henkel–Ecolab) cleaned and rinsed	0.73	8
Fouled and rinsed then Ultraclean II (0.3 mL 100 mL^{-1}, Koch) cleaned and rinsed	1.70	4

FTIR-ATR = Fourier Transform Infrared Spectroscopy - Attenuated Total Reflectance.
Data compiled from Delaunay *et al.* (2006).

high value of flux presented in Table 3.5 (1.7 times that of the virgin membrane) did not necessarily reveal the efficiency of the cleaning, but probably only a more hydrophilic membrane surface.

Sodium hypochlorite (NaOCl) can be used effectively as both a cleaning agent and as a disinfectant. The cleaning action of this chemical is maximised at pH 9 (Begoin *et al*., 2006a). Under these conditions, NaOCl acts as a swelling agent and protein solubiliser. In combination with its ability to break the bindings between the foulants and the membrane, this can mean that it is more effective as a cleaning agent than NaOH (Daufin *et al*., 1991, 1992). At pH 11, it acts most effectively as a disinfectant (Begoin *et al*., 2006a, 2006b; Rabiller-Baudry *et al*., 2006). A chemical disinfection step is commonly performed at a concentration of 200 mg L^{-1} NaOH at pH = 11.5 (Begoin *et al*., 2006a). Sodium metabisulphite or hydrogen peroxide can also be used as a disinfecting agent (D'Souza & Mawson, 2005).

While disinfecting agents are necessary, exposure to excessive chlorine levels from the disinfection process is a common cause of polymeric membrane failure. Begoin *et al*. (2006a) examined a membrane following 8000 h of acid whey UF, and they observed extensive damage to both the polypropylene (PP) spacers and PES membranes. Parallel ageing studies, where membrane samples were exposed to cleaning agents continuously for some months at 50°C, showed that the PES membrane damage could be attributed to the use of NaOCl as a disinfection agent. A membrane autopsy also revealed that the mineral content remaining on the used membrane was substantially different from that in the feed of acid whey. The presence of elements, such as Si, Fe and Al, could only be attributed to the water used for rinsing and cleaning. The proportion of chlorine was also high and the proportion of calcium to magnesium had shifted substantially (Fig. 3.8).

Enzymatic cleaning cycle

Proteinaceous fouling can also be removed by enzymatic cleaners. These generally contain proteases that cleave protein bonds and thus facilitate removal. Lipases can also be used to act upon dairy fats. Dairy operators are sometimes nervous about using enzymatic cleaners, as product quality can be affected if there is any residual enzyme remaining at the end of the cleaning cycle; they can also be expensive. In addition, enzymatic cleaners can be relatively slow in their action with optimal exposure times varying from 20 min (Arguello *et al*., 2002, 2003) to 1 h (Munoz-Aguado *et al*., 1996). For these reasons, enzymatic cleaners are generally not preferred unless membrane durability constraints restrict operation to below pH 10 and/or fouling is severe (Coolbear *et al*., 1992; Rucka *et al*., 1996; Turkiewicz *et al*., 2006; D'Souza & Mawson, 2005). However, their use can extend membrane life as they operate under milder conditions of temperature and pH (Arguello *et al*., 2003). Furthermore, the use of enzymatic cleaners reduces the salt load in factory wastewater and this is of increasing importance in many systems. Munoz-Aguado *et al*. (1996) reported that there was an optimum enzyme concentration for cleaning, beyond which the enzyme acts more as a foulant. These authors also found that cleaning with a protease prior to a surfactant cleaning cycle produces better results than either step alone, particularly if the membrane is rinsed at 40°C between the two cycles. They argue that the globular whey proteins have the majority of the sites for binding of detergents hidden in the structural matrix, and that an enzymatic cycle allows these sites to be exposed by the scission of the protein strands.

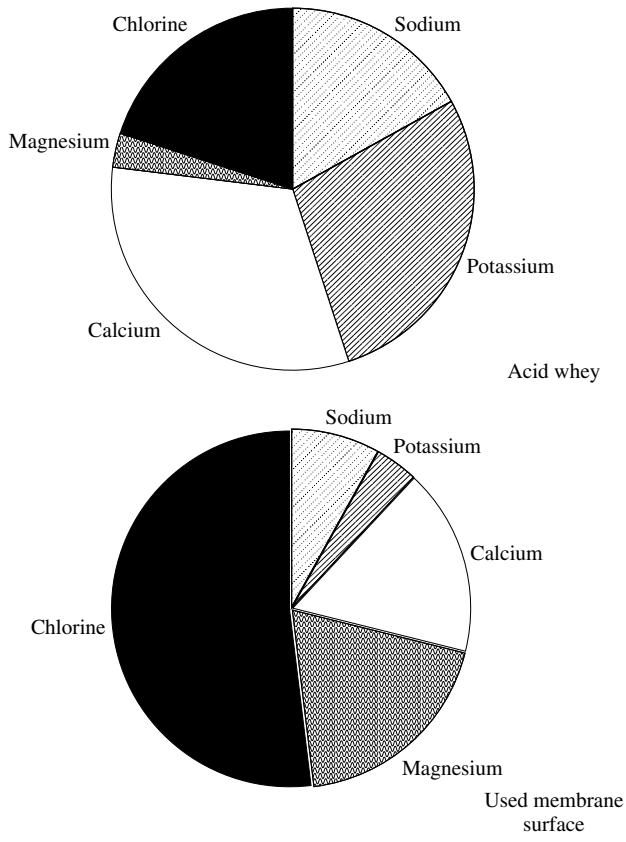


Fig. 3.8 Relative atomic percentage of the minerals in the acid whey feed to a polyethersulfone membrane and observed on the membrane surface after 8 000 hours of operation. Adapted from Begoin *et al.* (2006).

Acid cleaning cycle

Standard acid cleaners contain a mixture of phosphoric and nitric acid, and are chiefly used to remove calcium salts or scale. The pH is typically 1.6 (Begoin *et al.*, 2006a and 2006b). More recently, there has been a trend to use citric acid instead as this is less corrosive and milder on membrane surfaces (D'Souza & Mawson, 2005). Matzinos & Alvarez (2002) also showed that rinsing with a simple sodium chloride solution (0.4 M NaCl) is effective in removing calcium deposits. They argue that this is due to replacement of calcium by sodium ions.

Cleaning chemical reuse

There is an increasing focus on water recycling in dairy operations. Reuse of caustic cleaning solutions results in an increase in suspended solids and chemical oxygen demand (COD), which can decrease the cleaning effectiveness. However, reuse can also cause a decrease in surface tension, which assists with cleaning (Merin *et al.*, 2002; Alvarez *et al.*, 2007; Gesan-Guiziou *et al.*, 2007). Such cleaning solutions can be regenerated using MF,

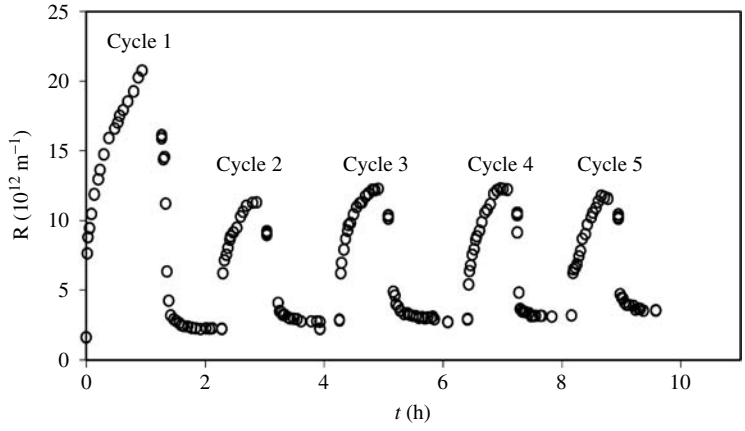


Fig. 3.9 Evolution of the hydraulic resistance(R) of a Tami 150 + 4T membrane fouled with whey protein when reusing Maxatase XL enzymatic cleaning solutions without additional purification. Reproduced with permission from Arguello *et al.* (2003).

UF and NF (Novalic *et al*., 1998; Gesan-Guiziou *et al*., 2002). MF is the least expensive approach, and results in a recovered caustic solution that contains more surfactant. However, the COD concentrations are also highest and the impact of this needs to be considered (Gesan-Guiziou *et al*., 2007). Fleming (2006) suggested that cleaning chemicals can be recovered by a dead-end membrane operation that forces 100% of the water through the membrane, with occasional periods of cross flow to flush out the contaminants that have accumulated at the membrane surface. Alternatively, these workers suggested that a combination of two membrane recovery systems be constructed, with the units alternating between dead end and cross flow. However, Arguello *et al*. (2003) showed that it was possible to reuse enzymatic cleaners for up to five cycles (Fig. 3.9) without any additional purification and this is of obvious economic importance to the use of such systems (see also Chen, 2006).

3.4 Spray drying technology

3.4.1 *Principles of spray drying*

Spray drying is the transformation of liquid food or other liquid product into powder form. It is carried out by spraying the concentrated liquid into a drying chamber simultaneously with hot air that might be introduced concurrently (in the same direction as product inlet), counter concurrently in the opposite direction, or under the product inlet at an angle (mixed flow). This process is continuous and fully automated.

Milk products, produced as spray dried powders, are milk powder, skimmed milk powder, whey powder, dry buttermilk, dry cream (sour and sweet), various dry dairy-based blends, casein and caseinates and others. The main objectives in processing are to achieve good quality and low production costs. The production of dried milks has the advantage of processing and storing all market surpluses of milk, as well as all by-products. World production of dry milk and dairy products increases rapidly each year. In general, spray drying

is the most common method of drying used in the milk processing industry. However, the theoretical principle of spray drying is briefly presented in this chapter. For further details refer to Carić (1994) and Westergaard (2004).

Spray drying in the stream of hot air is performed mostly at a constant rate of drying (~20 g 100 g^{-1} of moisture in the product, the intensity of mass transfer decreases), so the following equation is relevant:

$$\frac{dW}{dt} = AK\,(p_{vk} - p_v^{\circ}s) \quad (kg\ s^{-1}) \tag{3.12}$$

$$\frac{dW}{dt} = Ah\,(T_v - T_{vk})/r \quad (kg\ s^{-1}) \tag{3.13}$$

where

$W = \frac{\pi D^3 \rho}{6}$

D = droplet diameter (m)

ρ = droplet density (kg m^{-3})

$A = pD^2$ = droplet surface (m^2)

p_{vk} = partial pressure of water (at saturation) at the wet- bulb temperature

p_v° = partial pressure of water in the surrounding air

K = mass transfer coefficient $\left(\frac{kg}{sN}\right) = 2D_v/D$ and $hk/D = 2$ (for low air speed)

h = heat transfer coefficient ($W\ m^{-2}\ K$)

k = heat conductivity ($W\ m^{-1}\ K$)

T_v = air temperature (K)

T_{vk} = temperature of wet bulb (K)

r = latent heat of vaporisation of water (J kg^{-1}).

Moist air enthalpy is presented by the sum of dry air enthalpy and vapour enthalpy:

$$i = i_L + i_w x = i_L + i_w \frac{W}{L}\ (J\ kg_L^{-1}) \tag{3.14}$$

where i_L = air enthalpy $\left(\frac{J}{kg_L}\right)$

i_w = water enthalpy $\left(\frac{J}{kg_w}\right)$

x = water content in the air $\left(\frac{kg_w}{kg_L}\right)$

W = water quantity (kg_w)

L = air quantity (kg_L).

Since it may be assumed that the air and vapour act like ideal gases, this relation follows:

$$i = C_{pL}t + x(C_{pw}t + r_o)\ (J\ kg_L^{-1}) \tag{3.15}$$

where C_{pL} = specific heat of the air $\left(\frac{J}{kg_L K}\right)$

C_{pw} = specific heat of the water as a vapour $\left(\frac{J}{kg_w K}\right)$

r_o = latent heat of water vaporisation under normal conditions $\left(\frac{J}{kg_w}\right)$, which means that

$$i = t + x(1.93t + 2.5\times10^3)\ (J\ kg_L^{-1}) \tag{3.16}$$

where $t = T - 273$.

However, diagrams are most often used for drying calculations. Two of the three given variables are taken as coordinates (usually i, x diagram) and the third one (t) is changeable.

Because of the constant temperature, the expression for enthalpy is a straight line. Enthalpy for wet unsaturated air ($x < x_s$) has the following coefficient of direction in the i, x diagram:

$$\left(\frac{\mathrm{d}i}{\mathrm{d}x}\right)_t = 1.93t + 2.5 \times 10^3 \quad (\mathrm{J\ kg_w^{-1}}). \tag{3.17}$$

An isotherm in the super-saturation area (on the right side of the limiting curve $\phi = 1$) changes the coefficient of direction into

$$\left(\frac{\mathrm{d}i}{\mathrm{d}x}\right)_t = \left(\frac{\mathrm{d}i}{\mathrm{d}x_w}\right)_t = c_\mathrm{w} t \tag{3.18}$$

where c_w = specific heat of water $\left(\frac{\mathrm{J}}{\mathrm{kg\ K}}\right)$.

The theoretical drying process, drying at constant enthalpy (i = constant), is carried out in practice when the air for drying is heated to the designed temperature and introduced in the drying chamber. At constant enthalpy (adiabatic drying process), the moisture from the product is brought away into the drying air. The thermal balance for this case is:

$$Li_1 + Gi_{\mathrm{G1}} + Wc_\mathrm{w}t_{\mathrm{G1}} + Q = Li_2 + Gi_{\mathrm{G2}} + Q_\mathrm{R} \tag{3.19}$$

where L = drying air quantity ($\mathrm{kg_L}$)
i_1, i_2 = initial and final air enthalpy $\left(\frac{\mathrm{J}}{\mathrm{kg_L}}\right)$
G = weight of product leaving the dryer (kg)
$i_{\mathrm{G1}}, i_{\mathrm{G2}}$ = enthalpy of drying product G at the beginning and at the end of the process $\left(\frac{\mathrm{J}}{\mathrm{kg}}\right)$
W = moisture removed from the product during drying ($\mathrm{kg_w}$)
c_w = specific heat of water $\left(\frac{\mathrm{J}}{\mathrm{kg\ K}}\right)$
$t = T - 273$
Q = heat gained (J)
Q_R = heat losses (J)
t_{G1} = inlet temperature of the material to be dried (K)
$i_G = C_\mathrm{G} t_\mathrm{G}$.

So, the following equations are obtained:

$$Q = L(i_2 - i_1) - Wc_\mathrm{w}t_{\mathrm{G1}} + G(i_{\mathrm{G2}} - i_{\mathrm{G1}}) + Q_\mathrm{R} \tag{3.20}$$

$$\frac{Q}{W} = \frac{L}{W}(i_2 - i_1) + \frac{G}{W}(i_{\mathrm{G2}} - i_{\mathrm{G1}}) + \frac{Q_\mathrm{R}}{W} - c_\mathrm{w}t_{\mathrm{G1}}. \tag{3.21}$$

By introducing:

$$\frac{G}{W}(i_{\mathrm{G2}} - i_{\mathrm{G1}}) + \frac{Q_\mathrm{R}}{W} - c_\mathrm{w}t_{\mathrm{G1}} = \frac{Q_0}{W}$$

where $Q_0 = Q - L(i_2 - i_1)$ is the difference between the gained heat quantity and the energy absorbed by the drying air, the equation 3.21 changes to

$$\frac{Q - Q_0}{W} = \frac{L}{W}(i_2 - i_1). \tag{3.22}$$

Since water could be removed only into the air, it follows:

$$L(x_2 - x_1) = W \tag{3.23}$$

where x_1 and x_1 ($\mathrm{kg}_W\ \mathrm{kg}_\mathrm{L}^{-1}$) are the initial and final water contents, respectively, in the drying air.

$$\frac{Q - Q_0}{W} = \frac{i_2 - i_1}{x_2 - x_1} = \frac{\mathrm{d}i}{\mathrm{d}x}. \tag{3.24}$$

The total heat quantity is obtained by air passing through the air heater, so

$$\frac{Q}{L} = i_2 - i_1\ (\mathrm{J\ kg^{-1}}) \tag{3.25}$$

and from equations 3.23 and 3.24 follows

$$\frac{Q}{W} = \frac{Q}{L(x_2 - x_1)} = \frac{i_2 - i_1}{x_2 - x_1} + \frac{Q_0}{W}. \tag{3.26}$$

Heat balance depends on the drying system, and material balance is equal, no matter which drying system is used.

The material balance is

$$G_1 = G_2 + W \tag{3.27}$$

where G_1 = quantity of humid product subjected to drying (kg $\mathrm{s^{-1}}$)
G_2 = quantity of product after drying (kg $\mathrm{s^{-1}}$)
W = quantity of removed moisture (kg $\mathrm{s^{-1}}$).
For dried material it is

$$G_1 \frac{100 - w_1}{100} = G_2 \frac{100 - w_2}{100} \tag{3.28}$$

where w_1 and w_2 = initial and final moisture of product in weight (g 100 $\mathrm{g^{-1}}$)

$$\left(\frac{\text{kg water}}{\text{kg mixture}} \times 100\right).$$

From equation 3.27 it follows

$$G_1 = G_2 \frac{100 - w_2}{100 - w_1} \tag{3.29}$$

$$G_2 = G_1 \frac{100 - w_1}{100 - w_2} \tag{3.30}$$

$$W = G_1 - G_2. \tag{3.31}$$

On the basis of equations 3.29, 3.30 and 3.31, W may be expressed as

$$W = G_1 - G_1 \frac{100 - w_1}{100 - w_2} = G_1 \frac{w_1 - w_2}{100 - w_2}. \tag{3.32}$$

By combining the equations 3.29 and 3.31 the quantity of removed moisture is obtained:

$$W = G_2 \frac{w_1 - w_2}{100 - w_1} \tag{3.33}$$

3.4.2 *Spray drying techniques and systems*

The supreme method of drying milk and milk products is spray drying. The ambient air is filtered, heated to 150–300°C and introduced into the drying chamber at a velocity up to 50 m s^{-1}; various drying chamber designs are shown in Figure 3.10. Air cleaning is usually performed by using dry filters that are cleanable or disposable. Since air pollution continues to increase, it is necessary to introduce more rigorous air purification to be used for spray drying. Air is heated indirectly by steam in a tubular heat exchanger (THE) or PHE, liquid phase heating, or indirect oil or gas heating. Due to its high temperature, the inlet air has very low relative humidity. Good distribution of hot air in the chamber, with laminar flow and no dead corners, is very important for successful processing (Carić, 1994; Pisecky, 1997). Two systems for the recovery of heat and/or mass from the spray dryer exhaust are common, using sanitary scrubbers and sanitary venturi scrubbers. Their disadvantage is high bacterial counts, which increase immensely during operation. Another possibility, but only for mass recovery, is to install cloth filters.

Milk atomising devices serve a basic function in spray drying; they provide a high surface-to-mass ratio in the system sprayed milk–hot air mixture enabling quick heat transfer and high evaporation rates. The two most common atomising devices are centrifugal (rotary) and pressure (nozzle) atomisers. In both the cases, milk particles gain a spherical shape during drying because of the surface tension. Occluded air in the droplets results in a low bulk density of the powder. Atomisation parameters influence some important properties of the final product: bulk density, shape and size distribution of powder particles, occluded air content and moisture content.

In order to achieve the versatility in powder production, the dryers are often constructed for both atomising possibilities. Milk is dispersed in the centrifugal atomiser at rotating speeds of 10 000–20 000 revolutions per minute (rpm), or by a pressure of 17–25 MPa in the pressure nozzles. In this way, fine particles, uniform in size, having a diameter of 20–150 μm (most of which are in the range of 50–80 μm) with a large surface area, are obtained. By increasing the milk dispersion rate, the surface area is increased as well; this enables rapid and intensive heat transfer from air to milk and mass transfer from milk to air. The high latent heat of water evaporation (2.26 MJ kg^{-1}) and the increased surface area of sprayed particles cause instant evaporation and an immediate temperature drop of the incoming air to the temperature of outlet air (~95°C in a one-stage dryer).

In nozzle atomisation, the milk is discharged under pressure through an orifice of several millimetres in diameter into the drying chamber. There are nozzle designs in which dispersion is accomplished from many nozzles simultaneously. The mean diameter of the milk droplets and, consequently, the powder particles, is inversely proportional to the pressure

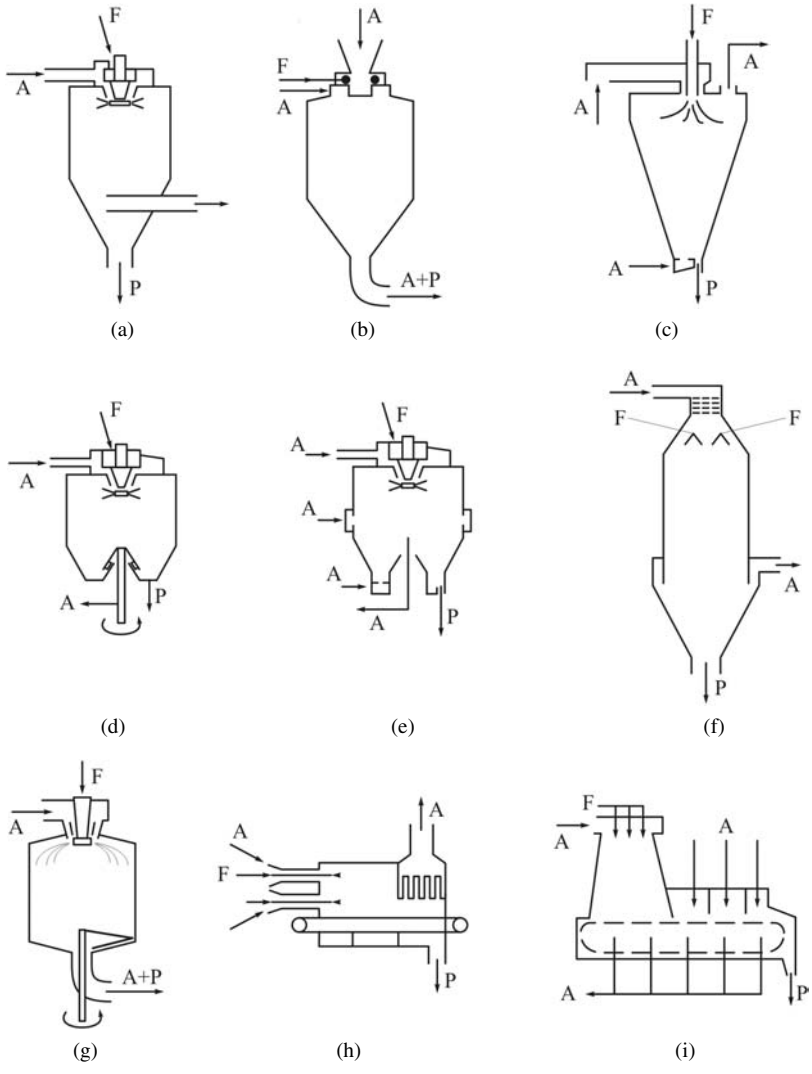


Fig. 3.10 Drying chamber design. Note: (a) conical-based chamber with two-point product discharge; (b) conical-based chamber with single-point product discharge; (c) conical-based chamber with integrated static fluid bed; (d) inverted-based coned chamber, (e) inverted-based coned chamber with integrated static fluid bed; (f) tower-form (nozzle tower) chamber; (g) flat-based chamber with product sweeper; (h) box chamber with integrated screw conveyor; (i) box chamber with integrated conveying band. F = feed, A = air flow and P = product. After Carić (2004).

applied, but is directly proportional to milk viscosity, density and surface tension of the feed. The disadvantages of this kind of atomisation are uneven powder particle size (outer section of the nozzle results in smaller particles than the inner portion) and a relatively low dry matter concentration of the feed (up to 48%).

A centrifugal atomiser is designed as a disc with vanes. The raw milk is transported to the atomiser under normal pressure and sprayed into fine droplets by the centrifugal force

of the rotating disc. With this kind of atomiser, large variation of capacity is possible, for example ±25%. There are controversial data in the literature regarding which type of atomisation consumes more energy *per* unit of product.

When milk is sprayed in the drying chamber, intensive heat and mass transfer take place. The heat, brought by the hot air, is transferred into the milk, while water, in the form of vapour, is transferred from milk droplets to the air. In the beginning, the rate of mass (water) transfer is constant due to the quick evaporation of water from the droplet surface. This (constant) phase is identical to the evaporation from a water surface or from a water droplet. When surface evaporation is completed, the solid phase on the droplet surface is formed, giving certain resistance to further evaporation (falling phase). In addition, evaporation in the advanced phase comprises diffusion of water molecules from the internal part to the droplet surface. After the removal of free water at constant temperature of the air, the removal of bound water by osmosis takes place (temperature increases), and, finally, the water bound by absorption is removed. Heat transfer coefficient is changeable during drying depending on the corresponding water content and varies in the range of about 100–500 $Wm^{-2}{}^{\circ}C$. The time necessary for a milk droplet to be dried in a spray dryer depends mainly on droplet diameter and air temperature (1 s or less). Dry product falls to the chamber bottom and is continuously discharged by air stream, separated from the drying medium and cooled by a cold air. Longer contact with hot air could result in penetration of fat to the particle surface, causing sticking together and overheating of the powder.

Powder can be separated from the air inside or outside of the drying chamber. Cyclone separators are used for external powder recovery. The principle of cyclone separation is based on centrifugal force. Considering that the velocity is higher in narrower cones, the cyclones of smaller diameter have better efficiency, but operate at a higher cost. Currently in application is a system of several cyclones with large diameters and one cyclone with a small diameter at the end that serves to separate fines.

Spray drying has numerous important advantages over the other drying techniques, such as the whole process proceeds very rapidly and the air residence time in the chamber is up to 30 s. In addition, because drying is accomplished at low temperatures, the product has excellent properties. There is no oxidation, loss of vitamins, denaturation of proteins, lactose transformation and other adverse effects of heat. For these reasons, spray drying is also used for drying of different pharmaceuticals, biological substances and thermosensitive materials. The product obtained by spray drying is similar in quality to the product obtained by freeze drying.

During spray drying, it is possible to automatically control the drying parameters and, by this, to control the properties of the final product such as final moisture and temperature, bulk density and powder particle size. The process is fully automated, so that even high capacities with high productivity require minimal labour. Since the product comes into contact with the wall of the closed chamber only in the powder form, there is neither a problem of equipment maintenance or corrosion nor a problem with the microbiological quality of the final product. The spraying device(s) can be used for drying all kinds of products that can be pumped, even if they are amorphous, adhesive or very viscous, such as casein, caseinates, cream and various dairy blends. Spray dried products have fine structure and another advantage of spray drying is that there is no large quantity of the product in

the chamber simultaneously, which is important in case of an eventual breakdown of the system during processing (Carić, 1994, 2002, 2004; Carić & Milanović, 2002).

Certain disadvantages of spray drying include very large dimensions of the drying chamber, expensive equipment, high electricity and steam consumption. By increasing the dispersion rate, it is possible to intensify the drying procedure, thus reducing energy consumption and chamber dimensions. Because of the high investment cost of spray drying, installation is economically justified only for large capacities, like 100 000–500 000 kg d^{-1} of raw milk.

Attempts to develop and introduce techniques other than spray drying in dairy–food technology and pharmaceuticals failed for various reasons, mostly of a technical nature.

3.4.3 *Plant design of spray drying configuration*

The conventional spray drying plant consists of a drying chamber with atomiser, heater, feed tank and cyclones. This configuration is defined as a one-stage drying system (Fig. 3.11). The product is dried to the final moisture content in the spray drying chamber only.

A significant advancement has been achieved in the reconstitution properties of spray dried powder by the introduction of two-stage drying configuration that combines spray drying as the first stage and fluid-bed drying as the second stage (instantisation) (step 6 in Fig. 3.12). There are two different instantisation methods known as '*rewet*' and '*straight-through*' processes. Instantisation markedly improves not only the reconstitution properties like wettability, penetrability, sinkability, dispersibility and rate of dissolving but also the economical aspects of drying technology. The instantisation process has been patented by Peebles in 1955 (Carić & Kalab, 1987; Kalab *et al*., 1991; Carić, 1994, 2002, 2004; Carić & Milanović, 2002), where the main feature was agglomeration during the two-stage drying procedure with entrapped air between powder particles (Fig. 3.13). Agglomeration was achieved by the introduction of the fluid-bed dryer for the two-stage drying system.

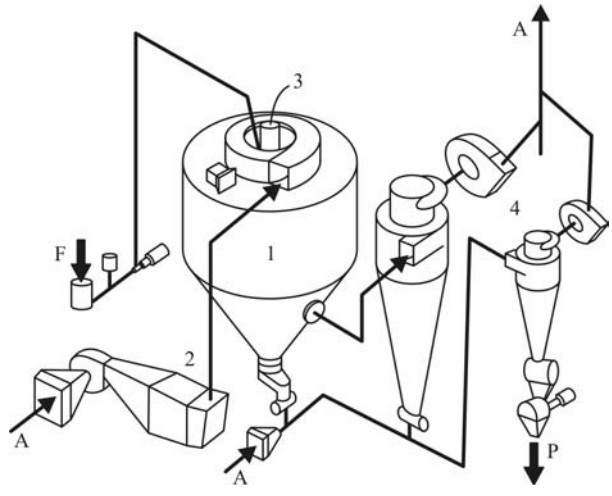


Fig. 3.11 Process line for spray drying of powder (one-stage drying, centrifugal atomisation). Note: 1, spray drying chamber; 2, air heater; 3, atomiser; 4, cyclone system; 5, control panel. F = feed, A = air, and P = product (powder). After Carić (1993). Reproduced with the permission of Niro Atomizer, Soeborg, Denmark.

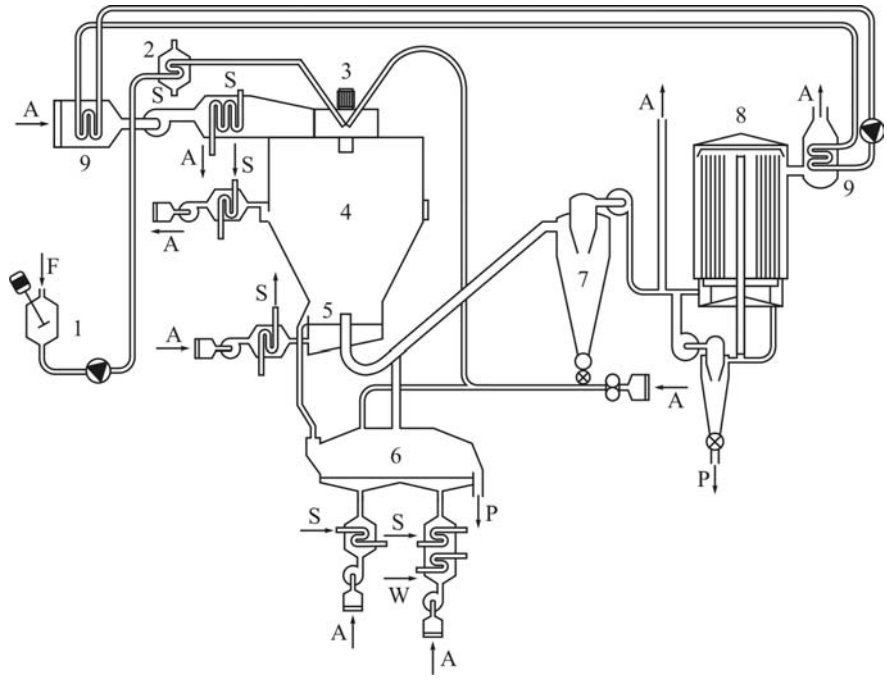


Fig. 3.12 An illustration of a three-stage drying system. Note: 1, feed tank; 2, concentrate pre-heater; 3, atomiser; 4, spray drying chamber; 5, integrated fluid-bed dryer; 6, external fluid-bed (instantiser); 7, cyclone; 8, bag filter; 9, liquid coupled heat exchanger. F = feed, A = air, S = steam, W = water and P = product. After Carić (2004). Reproduced with the permission of APV Anhydro, Denmark.

The agglomerated powder, containing 10–14 g 100 g^{-1} moisture, is transported from the spray drying chamber to a vibrating fluid-bed dryer, where it is finally dried in a hot air stream at 90–120°C, and immediately cooled to 10°C in the same dryer. The product outlet contains no more than 4 g moisture 100 g^{-1}.

During reconstitution, the entrapped air is replaced by water, enabling significant and instant water–powder contact that prevents viscous layer formation around grouped powder particles; this is what happens during the reconstitution of conventional one-stage dried powders (Fig. 3.13) (Carić, 2004).

Research to improve the spray drying procedure has produced significant results recently. In the middle of the 1970s, a two-stage drying system with an external vibrating fluid-bed dryer was developed and in the late 1980s, an alternative option, a stationary fluid-bed dryer, was integrated in the drying chamber. Although both types of fluid-bed dryer may produce agglomerated (instantised) as well as non-agglomerated powders, the dominating aspects that differentiate these dryers are those associated with the characteristics of the instant powders.

The special design of the three-stage spray drying chamber (Fig. 3.10) is called the *Filtermat* dryer, which contains a main drying chamber and three integrated smaller compartments for aggregation, final drying and cooling. The integrated moving belt transfers the powder to the final stage where it has the exact degree of agglomeration. Various designs of spray drying chambers have been developed for special products (high fat,

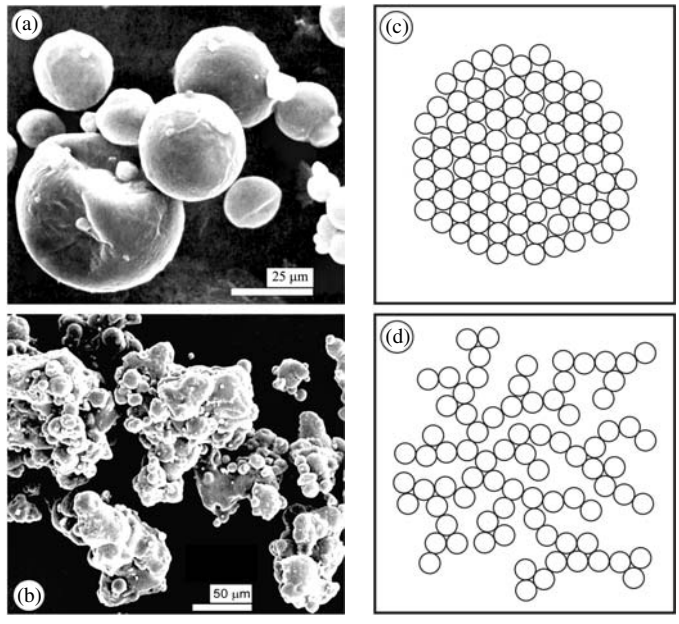


Fig. 3.13 Scanning electron microscopy (SEM) and schematic drawing of spray dried milk powder. Note: (a) one-stage dried (SEM); (b) agglomerated (SEM); (c) one-stage dried (schematic); (d) agglomerated (schematic). After Carić (2004), and reproduced with the permission of Wiley-VCH Verlag GmbH & Co.

infant formula) including Multi-Stage Dryer (MSD) by NIZO Atomizer, Spray Bed Dryer (SBD) by APV Anhydro and Multi-Fluidisation Technology (MFT) by Storck Amsterdam. The last type of dryer comprises a drying chamber, directly connected to the external fluid-bed dryer through a 'well mix' section. This reduces the problem of moist powder transportation. Most recent investigations have been carried out with the aim of further improving and modifying the spray drying devices for particular purposes (Carić, 1994, 2002, 2004; Pisecky, 1997).

3.4.4 *Heat economy of spray drying*

As in any other industrial process, the aim in spray drying technology is to produce the projected powder quality with the best possible process economy. The economy is improved with an increase of inlet air temperature and feed concentration. The increase of these parameters requires the increase of the outlet air and temperature, which leads to higher temperatures of the final product. This problem is solved by the introduction of an instantiser for final drying stage. The drying conditions are less severe and the specific heat consumption is 15–20% lower in the two-stage system than in the single-stage dryer. Further advantages are improved product quality and higher capacity (Carić, 1994, 2002, 2004).

The essential advantage of counter-current air–milk flow is that the hottest (incoming) air comes into contact with the already partially dry product and this enhances heat and mass transfer, reducing energy consumption. However, the resulting milk powder is heated to a higher degree during the last stage of drying, when concentrated milk

components, especially casein, become susceptible to thermal deterioration. In spite of the high heat requirement, concurrent flow is predominantly used in the dairy industry because it enhances product solubility.

Air quantity and thermal losses in the spray dryer are markedly decreased if the following aspects are provided:

- Temperature of the inlet air is high (e.g., inlet air temperature of 240°C, instead of 160°C, decreases steam requirement by 29% or energy consumption by 54%).
- Temperature of outlet air is as low as possible.
- The outlet air is used for heating the inlet air.
- Inlet air is taken from the upper part of the drying plant since it is the warmest.
- The drying chamber is insulated and hermetically closed (Carić, 1994).

An attempt to achieve a better heat recovery by utilising the overheated steam was also made, with recirculation to the evaporator (instead of hot air). In that case, the spray drying expenses may be reduced to one-third of the normal. In addition, the development of the three-stage drying system has made it possible to achieve greater energy savings than the two-stage dryer (Fig. 3.12). Three-stage drying involves a spray dryer as the first stage, a static fluid bed integrated in the base of the drying chamber as the second stage and an external vibrating fluid bed as the third stage. By moving the second drying stage into a drying chamber, it is possible to achieve even higher moisture removal at the end of the 1st drying stage than by two-stage drying. Lower temperatures are applied, which results in a powder of better quality and thermal efficiency (Carić, 1994, 2004). Specific heat consumption in a three-stage drying system is 866 kcal kg^{-1} of evaporated water compared to 972 kcal kg^{-1} in the two-stage dryer, other parameters being equal (Carić, 2004). Talking about energy use and energy efficiency when producing milk powder in four European countries, Ramirez *et al*. (2006) have shown that Germany, the Netherlands and United Kingdom have reduced energy consumption by ~2.1%, ~1.2% and ~3.8%, respectively, thus achieving considerable improvements in energy efficiency, contrary to the developments in the French industry where the same parameter was only 0.4%.

3.4.5 *Cleaning of dryers*

In principle, the cleaning operations of the washing programme of any dryer are similar to those of other dairy processing lines, and consist of the following stages: (a) pre-rinse using heated water, (b) caustic cleaning, (c) intermediate rinse, (d) acid cleaning and (e) final rinse (see Tamime, 2008). However, two other steps are carried out before and after the cleaning programme of any dryer; these include a dry method of cleaning in order to recover any residual powder from the equipment before starting the wet clean and, after the wet cleaning cycle, the plant has to be dried by passing warm air through all the equipment by activating the air heaters and fans before commencing the drying of milk products.

The frequency of cleaning spray dryers has improved over the past decades and, in general, dryers are cleaned less frequently (i.e. after operating the plant for a few weeks or even months) as compared to liquid processing plants, which have to be cleaned after 8–10 h of operation. Prior to the 1980s, the old-type dryers were single-stage units or

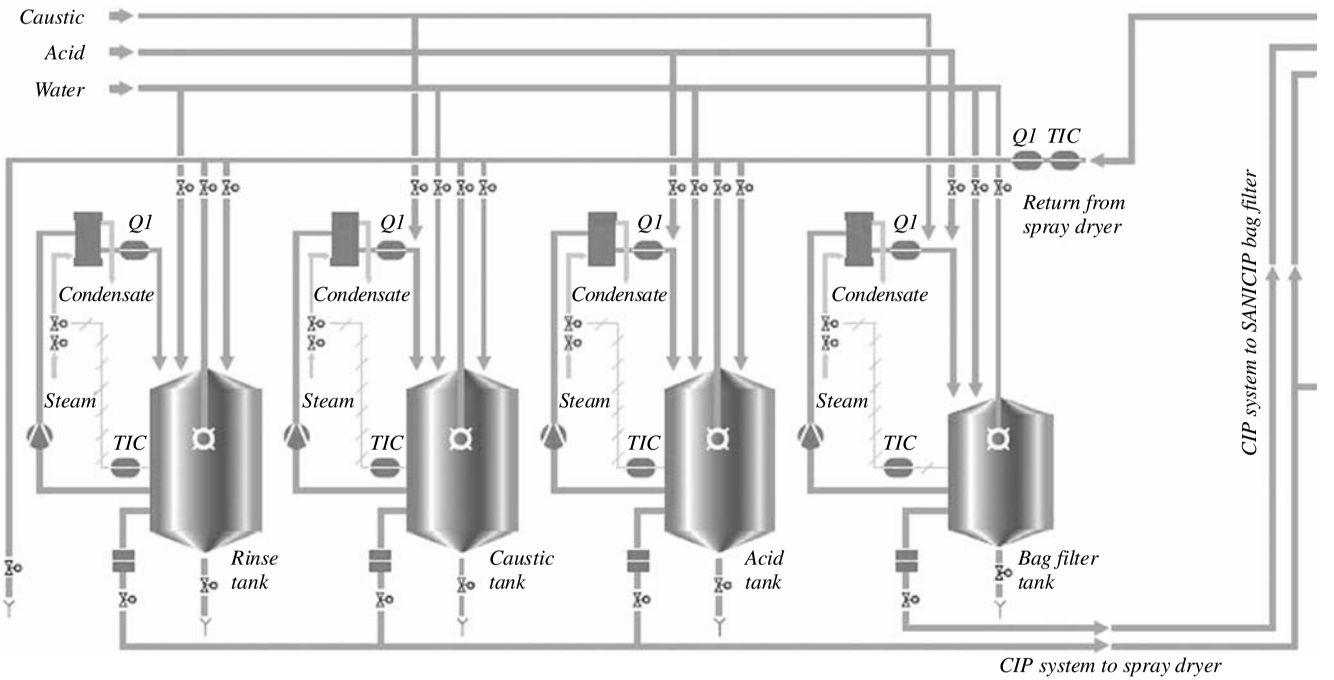
two-stage plants that were separate and not integrated (i.e. a spray drying chamber integrated with a fluid-bed dryer) and these needed to be cleaned after 1–7 days of operation. This frequency was due to the fact that the powder leaving the first-stage dryer, being moist and sticky, tended to build up on the chamber cone before discharging into the second stage dryer, whereas in more recent designs, the powder drops straight into a fluid bed of predominantly already dried powder and this effect gives far less build-up and thus reduces the need for cleaning. Since 2000, two-stage integrated dryers with integrated CIP-able bag filters have been developed (Fig. 3.14). Being a single vessel operation, there are no external ducts and secondary collectors that require cleaning, thus reducing cleaning costs and effluent loads. This general trend of improved hygienic design–engineering of spray drying and fluid-bed drying plants that minimise deposit formation has permitted plants to operate over longer periods of time (Chen *et al*., 1993; Schwartzbach & Masters, 2002; Anonymous, 2006a). In some cases, the equipment is only cleaned after 3 months or more of drying operation.

Few published data are available on the cleaning of dryers and, as most drying plants are 'tailor-made' to suit the product that is dried, the frequency and design of the cleaning programme of drying plants are developed in-house. Nevertheless, Anonymous (2006b) has reported the cleaning of drying plants of one of the major equipment suppliers (e.g. GEA Process Engineering (Niro); A. J. Partridge – personal communication). In brief, the cleaning of the drying and powder handling equipment is achieved by using either dry or wet methods of cleaning or a combination of both, and a summary of the cleaning of drying equipment is discussed in the subsequent text.

Spray drying chamber

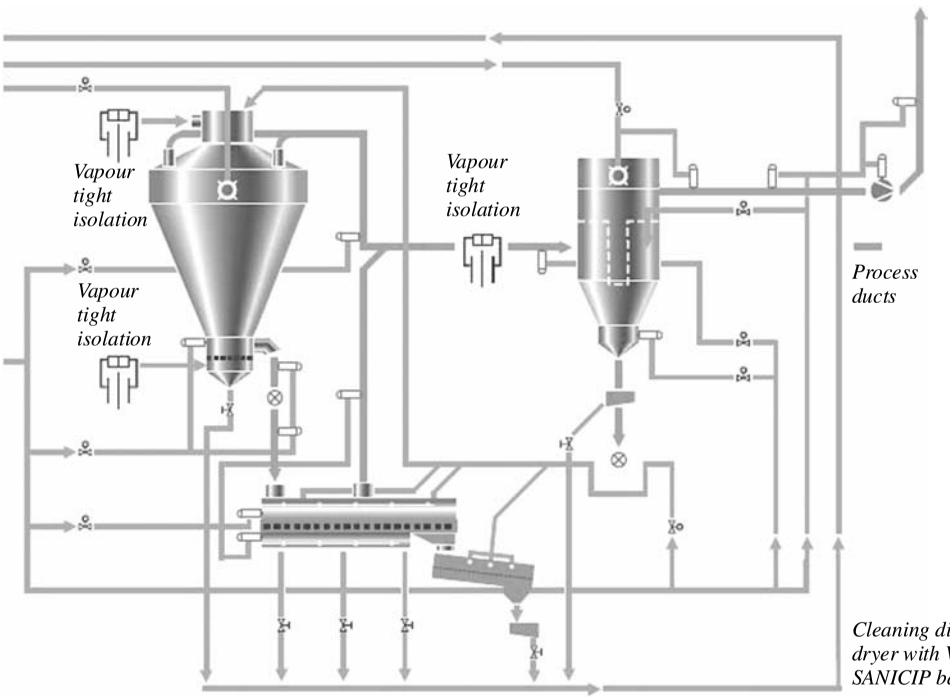
As mentioned elsewhere, the spray drying chamber is sometimes cleaned after a few months of operation. The part of the plant, which requires more frequent cleaning due to fouling, is the atomiser unit (i.e. rotary atomiser or nozzles) and, as a consequence, the dryers are usually supplied with stand-by atomisers. Periodically, the drying of dairy powders, for example, is stopped for a short duration and the rotary atomiser is changed over; if there are high-pressure nozzles, these are removed and are replaced one by one as the dryer continues to run, if this is required, or replaced with a clean set of nozzles, and the process is resumed. It should be noted that evaporators supplying the dryer need to be cleaned daily for up to 4 h and this allows time to attend to the dryer CIP as well. The fouled atomiser unit is positioned in a special stand for cleaning either manually or in a CIP circuit (Fig. 3.15) and, by allowing the atomiser unit of the dryer to be cleaned separately, the running time of the plant is extended until cleaning of the drying chamber (including the fluid-bed dryer) is required (Figs. 3.16 and 3.17).

The dry method of cleaning of the equipment involves manual sweeping of the surfaces in contact with the product using a variety of implements and brushes. Alternatively, air sweeping is used by allowing a high velocity air stream to pass over the surfaces to be cleaned. The latter approach is achieved by passing air through air-sweep slots (Fig. 3.18a, b), which are fitted, for example, along the sides of the drying chamber. Earlier designs allowed for remotely opening air-sweep doors, but this approach is less frequently used since the air introduced to the chamber is not filtered. Correctly designed air sweeps have their own blowing and heating systems where the air is also pre-filtered to the correct



(a)

Fig. 3.14 **a,b** Cleaning illustration for a multi-stage dryer with Vibro-Fluidizer® and Sanicip bag filter. Data by permission of A.J. Partridge (personal communication) and GEA Process Engineering (Niro A/S), Gladsaxevej 305, Soeborg, Denmark.



(b)

Fig. 3.14 Continued.

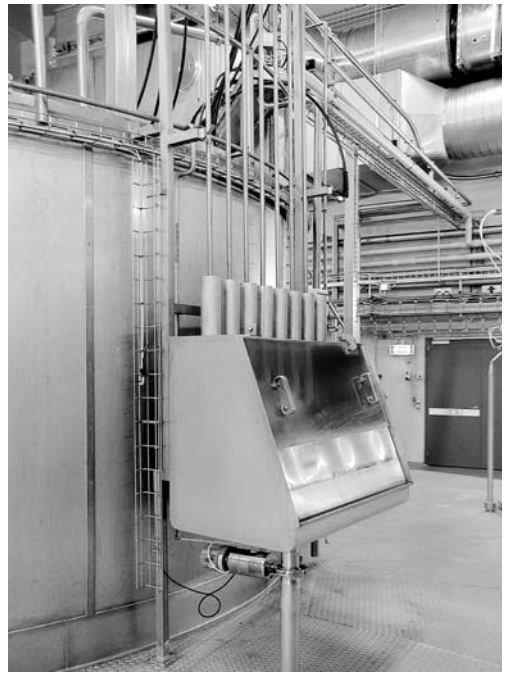


Fig. 3.15 High-pressure atomiser nozzles ready for cleaning-in-place (CIP) nozzles, and all nozzle lances and nozzle heads are placed in a special stand for cleaning. Data by permission of A.J. Partridge (personal communication) and GEA Process Engineering (Niro A/S), Gladsaxevej 305, Soeborg, Denmark.

standard. It needs to be decided locally whether any powder removed using these methods can be included in the production or downgraded. The main purpose, however, is to remove as much powder as possible in order to minimise the amount of powder to be wetted and, therefore, subsequently treated in a liquid effluent treatment plant. After removing the residual powder from the drying equipment and having replaced the atomiser unit with a turbine or tank cleaning nozzle (Figs. 3.19 and 3.20), the CIP programme is activated to clean the drying chamber. It should be noted that many operators take the opportunity of testing the fire extinguishing system at this point since this will serve both the test and the first wetting of the chamber.

Fluid-bed dryers

These are designed by GEA Niro A/S, are known as the Vibro-Fluidizer® (Fig. 3.17), and are cleaned in a similar manner to that described to clean the spray drying chamber using the dry and wet methods (Fig. 3.21). Cleaning nozzles, activated to protrude into the dryer either by the cleaning liquid pressure or by air cylinders, are used for the CIP.

Ducts and cyclones

Any process duct that is installed in the drying plants (e.g. air exhaust ducting from the top of the spray dryer (Fig. 3.22) and the cyclones to recover powder before the air is

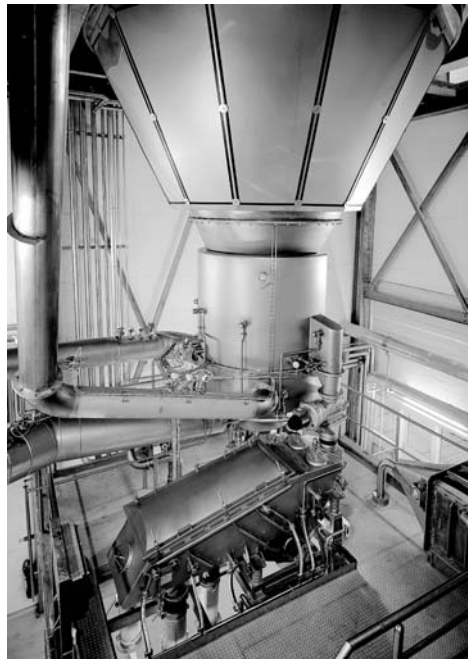


Fig. 3.16 Drying chamber cone with integrated fluid-bed dryer and sieve with cleaning-in-place (CIP) nozzles. Data by permission of A.J. Partridge (personal communication) and GEA Process Engineering (Niro A/S), Gladsaxevej 305, Soeborg, Denmark.

Fig. 3.17 Vibro-Fluidizer and sieve with side-mounted cleaning-in-place (CIP) nozzles. Data by permission of A.J. Partridge (personal communication) and GEA Process Engineering (Niro A/S), Gladsaxevej 305, Soeborg, Denmark.

(a) (b)

Fig. 3.18 Liquid pressure operated cleaning-in-place (CIP) nozzle (a) and pneumatically operated CIP nozzle (b). Data by permission of A.J. Partridge (personal communication) and GEA Process Engineering (Niro A/S), Gladsaxevej 305, Soeborg, Denmark.

exhausted) is cleaned separately using the dry and wet methods. This can be manual or more automated by using the aforementioned cleaning nozzles, which are staggered along the ducts (i.e. ∼2 m apart along the top and side of the duct) to ensure proper coverage of all surfaces during the CIP cycle.

CIP-able bag filters

These contain filter bags to recover powder from the air being exhausted and are cleaned once every month or so. Compressed air is blown inside the bags to remove any residual

Fig. 3.19 Jet liquid nozzle for cleaning of the drying chamber surface. Data by permission of A.J. Partridge (personal communication) and GEA Process Engineering (Niro A/S), Gladsaxevej 305, Soeborg, Denmark.

Fig. 3.20 Spray drying chamber in cleaning-in-place (CIP) mode; the atomiser nozzles are removed from atomisation zone and a CIP turbine cleans the surface of the inner chamber walls. Data by permission of A.J. Partridge (personal communication) and GEA Process Engineering (Niro A/S), Gladsaxevej 305, Soeborg, Denmark.

powder from outside the bags using back pulsing when the plant is in normal production. The same back-pulsing nozzles are used for CIP, and each bag is served by a nozzle to jet the CIP cleaning solutions into the bag (Fig. 3.23). In addition, the plates that hold the filter bags are cleaned using nozzles from the top, underneath and in between the bags to ensure good standards of hygiene. Plants fitted with dry bag filters will generally not have

Fig. 3.21 Preparing the Vibro-Fluidizer® for cleaning-in-place (CIP) by placing a funnel which returns the cleaning liquid to the CIP station. Data by permission of A.J. Partridge (personal communication) and GEA Process Engineering (Niro A/S), Gladsaxevej 305, Soeborg, Denmark.

Fig. 3.22 Air exhaust ducting from top of the spray drying chamber equipped with cleaning-in-place (CIP) nozzle. Data by permission of A.J. Partridge (personal communication) and GEA Process Engineering (Niro A/S), Gladsaxevej 305, Soeborg, Denmark.

the bag filter cleaned until the bags are replaced. Plants fitted with wet venturi scrubbers will need to have the scrubbers cleaned, at the very least, on a daily basis as with any other liquid dairy process.

Vapour proof dampers/butterfly

These are special units fitted in the drying plants (Figs. 3.14 and 3.24), which can seal the bag filter from the rest of the drying chamber when individual sections of the dryer are

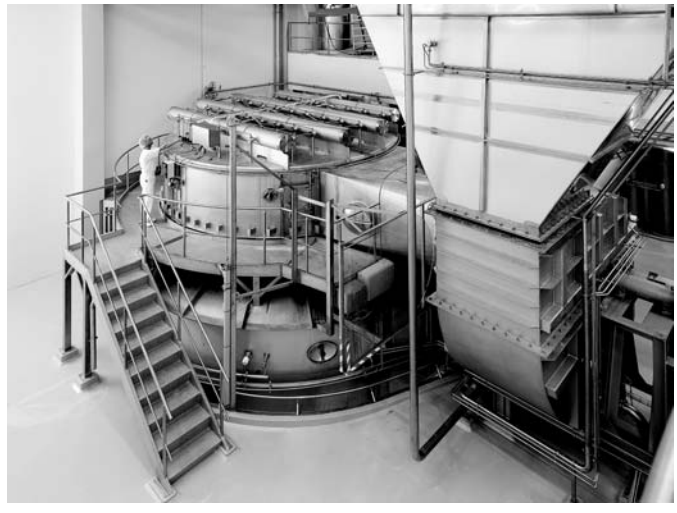


Fig. 3.23 A view of the top of a Sanicip™ bag filter. Data by permission of A.J. Partridge (personal communication) and GEA Process Engineering (Niro A/S), Gladsaxevej 305, Soeborg, Denmark.

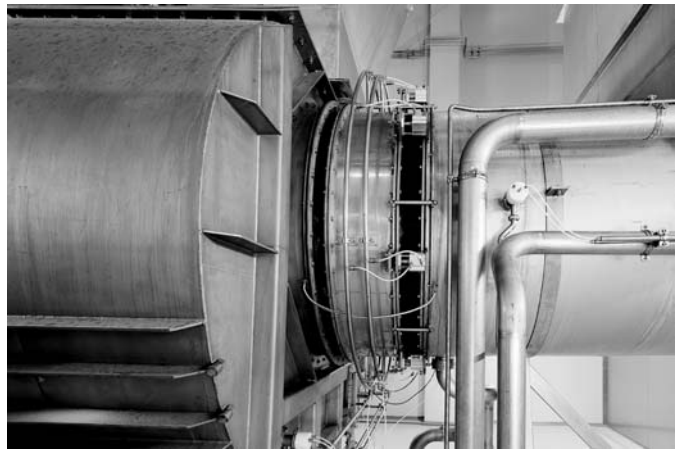


Fig. 3.24 Vapourtight damper that isolates and keeps the Sanicip™ dry during the wet cleaning of other parts of the dryer. Data by permission of A.J. Partridge (personal communication) and GEA Process Engineering (Niro A/S), Gladsaxevej 305, Soeborg, Denmark.

cleaned separately. Rubber balloons are also used but these are generally more troublesome and require manual intervention into the plant.

After the wet cleaning, a visual inspection can take place to ensure there is no residual powder left in the plant. This should be done with the minimum of manual intervention for good hygienic reasons. Repeating of the CIP may be necessary if the plant has not been sufficiently cleaned. The drying period for the plant varies according to the design. Plants with CIP-able bag filters take considerably longer by several hours. The plant drying process needs to be thorough for both hygienic and safety reasons. Surfaces that remain wet will attract deposits, which at best could spoil the product, leading to losses and, at the worst, cause layer smouldering and the potential for fire and explosion. For similar reasons, all CIP nozzles and pipework near to or above the dryer should be checked regularly for leaks to prevent ingress of water into the drying chamber and other equipment when in production.

3.5 Conclusions

Introduction of modern spray drying techniques enabled immense expansion of various concentrated and dried dairy product development. In addition to the main features of spray drying and instantisation technology, the outstanding achievement in concentrating and drying milk or food is the development of multi-stage vacuum evaporation with the thermal and MVR resulting in better economy, and the introduction on membrane methods that allowed numerous combinations of dairy-based powders with different compositions.

In general, for further industrial application, the capacity of membrane processes should be increased. Therefore, appropriate fouling control is also important regarding the economics of the process. Although flux enhancement has got a lot of attention in terms of understanding and practical improvement (with inserts and back pulsing), achievement of a high selectivity (full retention of large components and full transmission of small ones)

deserves more attention. High selectivity is not only dependent on the membrane, but is just as much dependent on the process conditions and interactions between the components in the feed. New types of membranes with narrow pore-size distribution are currently becoming available on the market (Brans *et al*., 2004).

Attempts to develop drying techniques other than spray drying failed for various reasons, most of which are of technical nature. Further developments are expected in improving and modifying the spray drying procedure itself.

While there will always be more work to do, the science of membrane fouling and cleaning is now relatively well understood. The challenge in the future will be to do more with less. As water becomes increasingly scarce and environmental restrictions make disposal of cleaning solutions more expensive, there will be an increasing focus on minimising the use of harsh chemical cleaners and the use of 'green' cleaning solutions. This is likely to include an increased emphasis on enzymatic cleaning cycles and upon cleaning chemical reuse.

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4 Production of Evaporated Milk, Sweetened Condensed Milk and 'Dulce de Leche'

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4.1 Background

The Organisation for Economic Cooperation and Development (OECD) forecasts that the world milk production is set to increase by 121 million tonnes from its current base of approximately 470 million tonnes by 2013. OECD member countries are expected to contribute about 25 million tonnes of this anticipated expansion in production. Hence, the bulk of the expansion is going to take place outside the OECD member states and the spotlight is focused on countries such as China and Argentina as major individual contributors. In line with the past trends, growth in milk production is already being accompanied by the increased manufacture of commodity dairy products, such as butter and skimmed milk powder (Kelly, 2006).

Evaporation has historically been the primary technology for liquid concentration in the food industry. The production of concentrated milk and dried milk products began in the nineteenth century when Nicolas Appert, a French inventor, described his procedure for concentration and drying of milk. Technologies for evaporation have been well established, and considerable background and experience exists for the evaporation of liquid foods to maintain, as close as possible, the initial characteristics of the 'original' food (Heldman & Hartel, 1998).

Fresh milk and dairy products have a high nutritional value but, as these products have a limited shelf life, they should be processed in order to become microbiologically stable. One of the widely used techniques for this is reducing the water content, and thereby the water activity (a_w), by concentration and drying.

Concentrated milks are liquid milks preserved by reduction of water contents to extend the shelf life and/or to improve its value. To achieve these aims, water is removed by evaporation, leading to a product called *evaporated milk*, or by creating conditions that do not allow the growth of micro-organisms. The addition of a large quantity of sucrose and exclusion of oxygen result in a product called *sweetened condensed milk*.

Technology for traditional evaporated milk and concentrated milk products has not seen changes in the last 25 years. The use of bulk condensed whole milk has declined from 1.2×10^5 tonnes to 5.9×10^4 tonnes; however, the use of bulk condensed skimmed milk has grown moderately from 3.2×10^5 tonnes to 4.7×10^5 tonnes since 1981 (Henning *et al*., 2006).

Evaporated and concentrated milk products are manufactured using evaporation technology. The efficiency and capacity of the evaporators have increased substantially in the last 25 years. Modern falling film evaporators are designed to take advantage of the thermal

vapour or mechanical vapour compression systems to reduce the requirement of steam in evaporating water from milk (Henning *et al*., 2006).

Production processes for evaporated or concentrated milk by membrane technology [e.g. ultrafiltration (UF)] have been developed, but the use of such processes is limited on an industrial scale. Concentration by reverse osmosis (RO) would yield a product virtually identical in composition and properties to the one undergoing concentration by evaporation. The use of the RO process allows the separation of water from milk without a phase change in such a manner that water is removed as permeate (i.e. liquid water) rather than as steam by evaporation installations (Henning *et al*., 2006).

'Dulce de leche' is a type of sweetened condensed milk that is very popular in some South American countries, such as Argentina, Uruguay, Brazil and Mexico. It is produced by concentrating milk at atmospheric pressure in the presence of added sucrose. Sodium carbonate could be used to avoid casein coagulation and, to favour the Maillard reactions responsible for its typical brown colour. Mainly, there are two types of 'dulce de leche' products; the first is for household use, which is consumed as a spread or a dessert and the second is for confectionery use and has a higher viscosity (Ares *et al*., 2006). Figure 4.1 shows some types of 'dulce de leche' products available in some South American Countries.

Retail packaging for evaporated milk continues to be the traditional tin or metal can, although a layer of polymer coatings is usually applied to the cans to prevent dissolution of tin and iron into the concentrated product. These cans allow for in-container heating to kill all vegetative micro-organisms and to inactivate bacterial spores. A variety of packaging materials, such as aluminium foil-lined cartons, cups made of aluminium or polystyrene and even translucent polystyrene cups are used in packaging ultra high temperature (UHT)-processed evaporated milk products aseptically (Henning *et al*., 2006).

This chapter discusses the technology of evaporated and sweetened and condensed milk manufacture and covers the main aspects of 'dulce de leche' manufacture using the UF process and the properties of all the products.

Fig. 4.1 Types of 'dulce de leche'.

4.2 Evaporated milk

4.2.1 *Introduction*

Concentration of liquid foods is an important operation of many food processes and there are several technologies available, such as evaporation under vacuum and membrane concentration. Evaporation involves removal of water by boiling, with a concentrated stream of milk, for example, remaining after separation of the vapours generated upon boiling. Concentration by use of permeable membranes is a rapidly developing technology that has found many applications in food processing. The basis for membrane separations is the difference in the permeability of a semi-porous membrane to different molecular sizes. Freeze concentration is another technology that has been developed over the past few decades, although significant applications of freeze concentration of foods are limited to fruit juices, coffee and tea extracts, beer and wine (Heldman & Hartel, 1998).

Evaporation

In the dairy industry, evaporation is used for concentration duties, such as milk, skimmed milk and whey. It is also used as a preliminary step to drying. Evaporation of water from the solution by heating (e.g. at 100°C) is feasible, but usually the products to be evaporated are heat sensitive and heating can change some their physicochemical characteristics at this elevated temperature. Hence, evaporation at low temperatures, such as under vacuum at ~70°C, reduces the thermal degradation of food properties (textural and nutritional), and aroma recovery is designed to allow collection of essential flavours and aroma compounds. To minimise thermal degradation, the evaporator should be designed for the shortest possible residence time.

Requirements for optimal evaporation include: (a) rapid rate of heat transfer, (b) low-temperature operation through application of a vacuum, (c) efficient vapour-liquid separation and (d) efficient energy use and recovery. Proper evaporator design and product handling are also necessary to ensure hygienic operation, since microbial inactivation does not occur to significant extent in an evaporator (Heldman & Hartel, 1998).

Vacuum evaporation

It takes a large amount of energy supplied as steam to boil off water from the solution. For adequate separation between the vapour produced and the remaining concentrate, efficient evaporation techniques are needed to remove fine droplets of concentrate that are entrained in the vapour stream. One of the main advantages of evaporation technology is the range of options available for efficient energy use and recovery. Although steam is no longer an inexpensive resource, a combination of multiple stages and steam reuse schemes allow an extremely efficient use of the energy in the steam. Typically, 0.5 kg of steam can be used to generate a little less than 0.5 kg of vapour through evaporation. Steam economies of up to 25–30% (mass of vapour produced per mass of steam used) can be obtained by using energy recovery technologies (Heldman & Hartel, 1998).

Multiple-effect evaporation

Several methods for improving evaporation efficiency, particularly in reutilising the energy contained in the steam, have been developed. To reduce the amount of steam needed, the evaporation equipment is normally designed as a multiple-effect evaporator. A number of evaporators are combined in a process utilising vapours from a previous stage as a steam to provide heating for evaporation in the next stage. Two or more units operate at progressively lower pressures and, thus, with progressively lower boiling points. In such an arrangement, the vapour produced by evaporation of water from the initial product in the first stage is fed into the steam/heating section of the second stage to provide further evaporation. The vapour produced in the last stage is then condensed. Since the vapour from the first stage is generally at the same temperature and pressure as the feed going into the second stage, the operation pressure of the next stage must be reduced to lower the boiling temperature and to allow the previous-stage vapour to provide a driving force for boiling the milk, for example, in the next stage for additional evaporation (Heldman & Hartel, 1998).

The result is that the amount of steam needed is approximately equal to the total amount of water evaporated divided by the number of effects. Evaporators with up to seven effects are now used in the dairy industry. Alternatively, electricity can be used as the energy source; in this case, an electrically powered compressor or fan is used to recompress the vapour leaving the effect to the pressure needed on the heating side. Although evaporator plants generally work on the same principle, they differ in the details of their design (Anonymous, 2003).

All evaporators are composed of the same principal components. The principal component is the steam section, or other means of introducing heat efficiently into the product to accomplish boiling. The liquid feed is often preheated to the evaporator temperature before being distributed uniformly to the heat transfer surface area. This feed distributor is important for efficient operation with minimal product degradation and evaporator fouling. Once vapour has been produced by boiling, an efficient separator is required to isolate vapours from liquid concentrate. A condenser cools the final vapours removed into a warm-water stream that may be used for energy recovery schemes (Heldman & Hartel, 1998).

The tubes that form the partitions between steam and product can be either horizontal or vertical and the steam can be circulated either inside or outside the tubes. In most cases, the product circulates inside the vertical tubes while steam is applied to the outside. The tubes can be replaced by plates, cassettes or lamellas (Anonymous, 2003).

Circulation evaporation

This method can be used when a low degree of concentration is required or when small quantities of product are processed. This treatment simultaneously deaerates the product and removes any off-flavours present. The milk, heated to 90°C, enters the vacuum chamber tangentially at a high velocity and forms a thin, rotating layer on the wall surface. As it swirls around the wall, some of the water is evaporated, and the vapour is drawn off to a condenser. Air and other non-condensable gases are extracted from the condenser by a vacuum pump. The product eventually loses velocity and falls into the inwardly curved

bottom, where it is discharged. Part of the product is recirculated by a centrifugal pump to a heat exchanger for temperature adjustment and then to the vacuum chamber for further evaporation. A large amount of product must be recirculated in order to reach the desired degree of concentration. The flow through the vacuum chamber is four to five times the inlet flow to the plant (Anonymous, 2003).

Falling film evaporation

In the dairy industry, falling film evaporators are commonly used and have practically replaced all other types. In this method, the milk is introduced at the top of a vertically arranged heating surface and forms a thin film that flows down over the heating surface, resulting in short retention times and gentle heat treatment. The heating surface may consist of a bundle of vertical stainless steel tubes or plates. The plates are stacked together forming a pack with the product with plates on one side and steam on the other. When tubes are used, milk forms a film on the inside of the tube, which is surrounded by steam. In practice, a large number of different evaporator configurations are used in the industry. The number of evaporator effects (tube bundles) varies from one to seven. The actual configuration depends on the desired properties of the concentrate and the state of the art at the time of installation of the evaporation plant. In order to obtain a high thermal efficiency, in a number of cases, the products are first preheated to a temperature equal to or slightly higher than the evaporation temperature in spiral tubes placed in the condenser and the multiple-effect evaporators. From the pre-heater, the product flows to the distribution system (e.g. by distribution plate) at the top of the evaporator. In general, the whole pre-heating trajectory has a great impact on the properties and quality of the concentrate and powder. To meet some quality standards, it is necessary to use a direct heater (e.g. steam injection, steam infusion) to apply a short-time high-temperature treatment (Heldman & Hartel, 1998; Muir & Banks, 2000; Anonymous, 2003). The boiling temperatures in the multiple-effect evaporators vary from 70–80°C in the first unit to 40–50°C in the last unit. It is well known that by increasing the number of vacuum units (effects) the energy consumption decreases (Muir & Banks, 2000).

Thermal vapour recompression

During the manufacturing of milk products, the inclusion of thermal vapour compression to increase the quality of the vapours produced is fairly common because it improves the thermal efficiency of the evaporator. This combined steam is then fed into the steam section to accomplish evaporation (Heldman & Hartel, 1998). High-pressure steam (fresh) to increase the kinetic energy is passed through a nozzle or ejector creating a steam jet before entering the evaporator chamber. As the fresh steam passes through the nozzle, it draws in some of the lower-pressure vapours from the separator. The mixed steam increases its energy content to a higher value than that of the vapour produced, and the quantity of fresh steam is reduced. A two-effect falling film evaporator with thermo compressor requires about 0.25 kg of steam to evaporate 1 kg of water, and a five-effect evaporator about 0.20 kg of steam. Without the thermocompressor they would need about 0.60 and 0.40 kg of steam, respectively. Demand for lower energy consumption has led to the construction of

plants with more than six effects (Anonymous, 2003). Usually, the multiple-effect operation and thermal recombination are often employed together.

Mechanical vapour compression

Unlike the thermocompressor, a mechanical vapour compression system draws all the vapour out of the evaporator and compresses it before returning it to the evaporator. The pressure increase is accomplished by the mechanical energy that drives the compressor. No thermal energy is supplied to the evaporator (except steam from the first effect). There is no excess steam to be condensed. In mechanical vapour compression, the total amount of steam is circulated in the plant (Anonymous, 2003).

4.2.2 *Evaporated milk production*

Several variations of the manufacturing process of evaporated milk are possible and the main steps are be summarised in Figure 4.2. Raw milk is received, selected and submitted to preliminary treatments, such as clarification, fat separation or milk standardisation. Pre-heating is performed to inactivate enzymes and to destroy micro-organisms including any bacterial spores present. Also, it is done to improve the heat stability of the product, and to facilitate the sterilisation process. Currently, UHT treatment is preferable at 115–128°C for 1–6 min even though a longer heat treatment (e.g. below 100°C for 20 min) could be done. The milk is usually concentrated by vacuum evaporation at 45–70°C. The milk could be concentrated by RO, but this is rarely done (Walstra *et al*., 2006). The dry-matter content of the evaporated milk must be known in order to optimise the raw milk supply or the amount of steam employed. This determination should be based on refractive index and should be standardised.

Subsequently, homogenisation is done in order to avoid creaming and coalescence. In general, it is performed in two homogenisation stages at 65°C; the former stage is operated at 15–25 MPa pressure and the latter stage at 5–10 MPa. In the process of in-bottle sterilisation, homogenised milk is packed and after filling, sterilisation is applied batchwise in an autoclave or continuously. In general, the process is conducted at 100–120°C over 15–20 min or at 140°C for 3 s. Sterilised evaporated milk is stored at 20°C. After concentration, the milk could be sterilised by UHT treatment as an alternative process. UHT treatment destroys spores more effectively than in-bottle sterilisation. It is done for 15 s at 140°C followed by cooling to 60°C. The whole process, from the pre-heating stage up to the aseptic packaging, should be proceeded without interruption.

4.2.3 *Product properties*

Table 4.1 shows the chemical composition of different types of evaporated milk produced in different countries. The nutritional value of evaporated milk is different from whole milk due to the heat treatment. In-bottle treatment destroys up to 10% of the available lysine, about half of the vitamins B_1, B_{12} and C, smaller proportions of vitamin B_6 and folic acid (Walstra *et al*., 2006). All the changes are smaller when the UHT process is used.

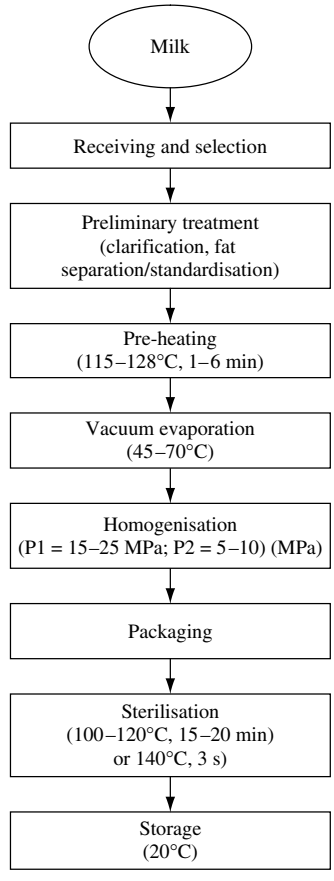

Fig. 4.2 Flow diagram showing the manufacturing stages of evaporated milk.

Table 4.1 Gross composition (g 100 g^{-1}) of different types of evaporated milk and sweetened and condensed milk.

Type	Fat	Solids-not-fat (SNF)	Concentration factor
American evaporated milk	7.8	18.1	2.10
English evaporated milk	9	22	2.60
Low-fat evaporated milk	4	20	2.25
Skimmed evaporated milk	0.1	22	2.35
American sweetened and condensed milk	8	20	4.60
English sweetened and condensed milk	9	22	5.00

Adapted from Walstra *et al.* (2006).

Maillard reactions are very important for the flavour and colour development in evaporated milk. Reducing sugars produce brown colours that are desirable and important in some cases, such as when evaporated milk is used in coffee. Other brown colours obtained upon heating, especially at high process temperatures or during long storage, are undesirable.

The viscosity of evaporated milk is often an important quality attribute. It could be achieved by heat treatment. For example, UHT-evaporated milk is less viscous; sterilisation prevents heat coagulation and often, κ-carrageenan is added (Walstra *et al*., 2006).

4.3 Sweetened condensed milk

4.3.1 *Introduction*

The method of preserving milk by sterilising evaporated milk in sealed containers was developed at the beginning of the 1880s. Earlier, about 1850, the method of preserving evaporated milk by the addition of sugar was developed in America. The manufacture of condensed milk, using these two methods, has developed into a large-scale industry. A distinction is made between the two types; *unsweetened* (evaporated) and *sweetened* condensed milk.

Unsweetened condensed milk (also called *double concentrated milk* or *evaporated milk*) is a sterilised product, light in colour and with the appearance of cream. The product has a large market, for example, in tropical countries for the armed forces. It is used where fresh milk is not available. Unsweetened condensed milk is also used as a substitute for breast milk. In this case, vitamin D is added. It is also used for cooking or as coffee cream. The product is made from whole milk, skimmed milk or recombined milk with skimmed milk powder, anhydrous milk fat (AMF) and water as typical ingredients (Anonymous, 2003).

4.3.2 *Production stages*

Sweetened condensed milk is milk concentrated by evaporation to which sucrose is added. The addition of a large quantity of sucrose creates conditions that do not allow the growth of micro-organisms, and the sweetened product could have a long shelf life. Figure 4.3 illustrates a flow diagram of a typical manufacturing process for sweetened condensed milk.

Initially, milk is clarified and standardised, and heated to inactivate the enzymes and to destroy many of the micro-organisms present. The heating intensity considerably affects the rheological properties, such as viscosity and gelation of the product during the storage period. The actual heat treatment must be considered for these factors and be adjusted. UHT heating is commonly applied about 130–145°C for 5 s. However, creaming and coalescence do not often occur and homogenisation is not always done but, when producing a low viscous sweetened condensed milk, homogenisation is required and is performed at alow pressure such as 2–6 MPa at 70°C.

Sucrose could be added to the original milk, but it causes extensive Maillard reactions during the heating and evaporation stages. Instead, concentrated sucrose solution

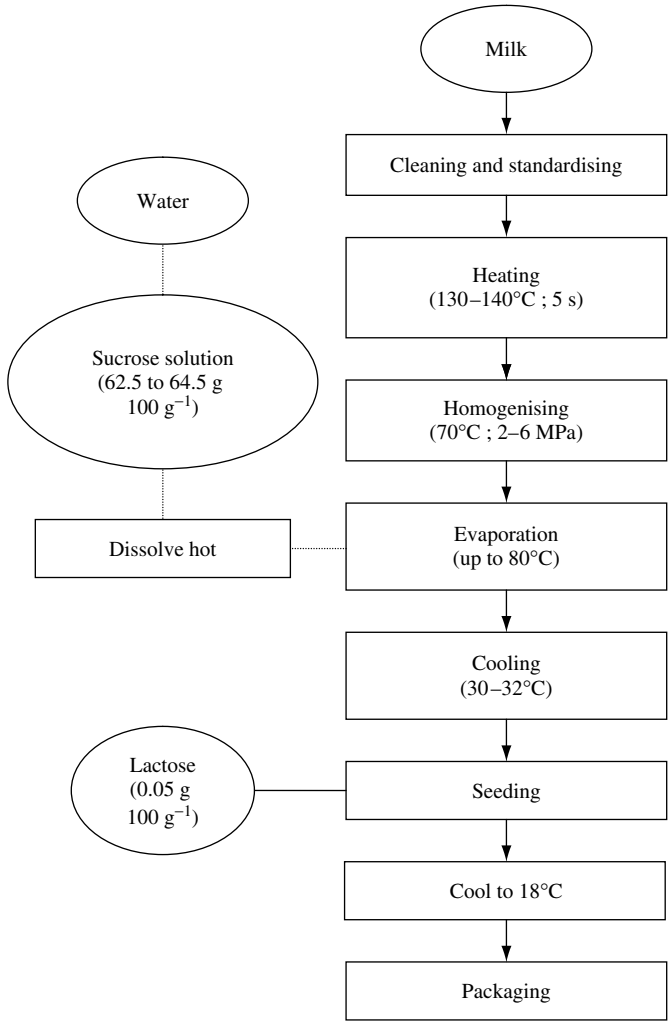

Fig. 4.3 Flow diagram showing the manufacturing stages of sweetened condensed milk.

(62.5–64.5 g 100 g^{-1}) is heat treated and added to the concentrated milk at the end of the evaporation stage. The sugar should be refined and devoid of invert sugar to prevent browning.

The milk is usually concentrated by vacuum evaporation at relatively high temperatures up to 80°C. In the evaporator, the milk is being concentrated in successive stages, and the sucrose solution is added to the evaporated milk in the last effect of the evaporator. At the outlet of the evaporator, the condensed milk has a density of 1.30 kg dm^{-2} in the case of the whole milk and 1.35 kg dm^{-2} in the case of skimmed milk.

In addition, one of the main stages of the process is cooling. Water of the condensed milk can only maintain in dissolution half of its lactose content; the remaining must be precipitated. It is necessary to avoid this precipitation occurring in an uncontrolled way,

Table 4.2 Gross composition (g 100 g^{-1}) of sweetened and condensed milk and 'dulce de leche'.

	Sweetened and condensed milk	Cooked sweetened and condensed milk	'Dulce de leche'
Moisture	28.60	26.83	20.13
Energy (kcal)	309	323	343
Energy (kJ)	1293	1351	1436
Proteins	6.40	5.38	5.25
Total lipids	5.90	6.88	6.73
Total carbohydrates	57.55	59.87	66.52
Ash	1.55	1.04	1.37

Adapted from Anonymous (2006).

which would lead to the formation of thick crystals of lactose, giving a sandy texture to the product. Cooling should be quickly performed under intense agitation to 30°C and the product should be transferred soon to a tank where fine lactose crystals are inoculated. The added lactose serves as a crystallisation seed and avoids the formation of large lactose crystals. If the formed crystals are <10 μm, they will stay dispersed in the mass of condensed milk without giving the sandy texture. Subsequently, the product should be vigorously agitated for a period of 1 h. Condensed milk must be cooled to a temperature at which the lactose is supersaturated so that the lactose seeds do not dissolve. However, the temperature must not be so low that spontaneous nucleation can occur before the seeded crystals are mixed in. After seeding, cooling should be continued to crystallise the lactose, initially to 30–32°C and then to 15–18°C. The product is kept in the tank for 12–14 h so that the crystallisation is completed.

Sweetened condensed milk is normally packed in cans that are first sterilised by flaming. The cans should be filled accurately to avoid any air in the head space, as this could permit the growth of osmophilic yeasts. The product can maintain its nutritious properties for a period of up to 2 years, without the need of refrigeration. Sweetened condensed milk has low a_w (~0.83), and high contents of sugar that restrict the growth of most microorganisms; this product is not sterile. Microbial deterioration of the product is usually caused by osmophilic yeasts, most of which belong to the genus *Torulopsis* that often causes gas formation, a fruity flavour, and coagulation of the protein (Walstra *et al.*, 2006). The composition of different types of sweetened condensed milk is shown in Tables 4.1 and 4.2.

4.4 'Dulce de leche'

4.4.1 *Background*

'Dulce de leche' (milk jam, milk caramel and dairy confectionary) is a typical dairy-based product in Latin America, and it has been consumed in Brazil, Argentina (Pinto, 1979;

Martins & Lopes, 1980; Souza *et al*., 1990; Pavlovic *et al*., 1992) and Mexico for more than a century. This product is mainly produced in Argentina, Brazil and Uruguay (Sabioni *et al*., 1984b). However, Mexico is probably the only country in Central and North America producing this product, which is marketed in Mexico and United States.

The manufacture of 'dulce de leche' in Mexico is done mainly using goat's milk; however, cow's milk is also used in some cases. The production of goat's milk, according to Mercado (1982), provides a very important income for goat farmers and, in 1980, milk production was 279.7 million litres; out of this, 25% was consumed as liquid milk and the rest was used for the manufacture of cheese and 'dulce de leche'. The *Cheese Reporter* of the USA (Anonymous, 1993) reviewed some aspects of the North American Free Trade Agreement (NAFTA) reporting that Mexican goat's milk 'dulce de leche' has an immediate tariff-free access to the United States. It is, therefore, one of the few dairy products that contribute external income for the Mexican dairy industry.

Actually, 'dulce de leche' is also appreciated in other countries for household and industrial uses. It is widely used as an ingredient for the manufacture of confectionary products, filling of crepes, biscuits, cookies and cakes, or as topping for ice cream and fruits cakes, and it is also directly consumed as a dessert or as accompaniment of bread, toast and cheese (Demiate *et al*., 2001; Malec *et al*., 2005). According to Pauletti *et al*. (1990), its consumption is increasing in Europe and the United States.

Traditionally, 'dulce de leche' in Brazil was home made until the intervention of the multinational companies, that secured the production and distribution of the product to all parts of the country. The regional production, mainly in South America (Pavlovic *et al*., 1992), explains the scarce scientific references in literature about this product. Most available papers were published in Brazil or Argentina and they are related to processing or quality characterisation. However, there is no data about the chemical and microbiological properties, physical parameters and microstructure of traditional, light and diet varieties of this product. 'Dulce de leche' does not present a uniform composition, despite the high volume produced.

In Brazil, 'dulce de leche' is used as a dessert or as a confectionary ingredient and, according to the Brazilian Statutory Regulations (BSR, 1997), the product must contain a maximum of 30 g 100 g^{-1} moisture and 2.0 g 100 g^{-1} ash; a minimum protein content of 5.0 g 100 g^{-1} and fat content can vary from 6.0 to 9.0 g 100 g^{-1}. Furthermore, the amount of sucrose that is used for the processing is 30 kg 100 L^{-1} of milk. Starches and modified starches can be used at the maximum of 0.5 g 100 mL^{-1} of milk, as well as monosaccharides or disaccharides that substitute for sucrose at a maximum of 40 g 100 g^{-1}. Cream or dairy solids are also permitted as optional ingredients. The list of allowed additives is huge, but there are maximum limits for most of them. The enzyme β-galactosidase and $NaHCO_3$ (sodium bicarbonate) are considered as coadjuncts. The traditional 'dulce de leche' has a caramel colour with variable intensity (Brasholanda, 1991); however at present, the colour of the product ranges from light cream to dark brown, which is governed by the extent of Maillard reaction intensity and caramelisation (Ferreira *et al*., 1989). Martins & Lopes (1980) report that the product presents high nutritional value because of its protein and minerals, besides its energy value. It is a food less perishable than milk and has high sensory acceptability. However, Pavlovic *et al*. (1992) report that processing conditions lead to a decrease in the nutritive value of the product, possibly by affecting the lysine

residue in the protein. They fed diets to young rats with freeze-dried 'dulce de leche' or spray-dried milk as a source of protein for 4 weeks and studied the nutritive value of 'dulce de leche' using protein efficiency ratio, food efficiency, apparent net protein utilisation and apparent digestibility.

The industrial manufacturing stages of 'dulce de leche' are similar to that of sweetened condensed milk, where the milk solids are concentrated by heating and evaporation (see Section 4.3). 'Dulce de leche' often has a sandy texture because of the high concentration of lactose leading to crystallisation; the lactose crystals can be up to 1500 μm in size (Hough *et al*., 1990). Efforts have been made to resolve this problem by breaking down lactose using bacteria or enzymes, and by seeding with lactose microcrystals. Reliable results have been obtained, but all of the methods are highly costly (Sabioni *et al*., 1984a, b; Martinez *et al*., 1990). The gross chemical composition of 'dulce de leche' is shown in Table 4.2.

UF may have good advantages in reducing the processing time and preventing sandiness by lowering the final lactose content of the milk, which is the most significant technological problem in 'dulce de leche production', having a consequent negative impact in reducing product acceptability (Sabioni *et al*., 1984b). The UF technology is now a well-established process for the separation and concentration of chemical molecules in milk due to the differences in their molecular weights. Using this process to treat milk, an ultrafiltered concentrate product with a reduced concentration of lactose can be obtained. Thus, UF process may be adapted to produce a low-lactose type of 'dulce de leche' and, by reducing the heat processing time, the production costs may be lowered (Carić, 1994). The major advantage of the UF process is that it yields a higher protein and lower lactose milk with excellent nutritional and functional properties (Lee & White, 1991).

During the production of sweetened condensed milk and 'dulce de leche', the evaporation temperature is normally above 93.5°C. Lactose crystals are not present due to the fact that, at that temperature, the concentration of lactose is below the saturation point. Doan (1958) reports that when sweetened condensed milk is cooled to 60 or 65°C, between two-fifths and two-thirds of the lactose present will emerge as crystalline α-lactose hydrate; this is because lactose is soluble to the extent of only about 15 parts to 100 parts of the water as found in the product.

4.4.2 *'Dulce de leche' production*

'Dulce de leche' is prepared by concentrating the milk using heat to ~70 g total solids 100 g^{-1} ($a_w = 0.85$) at atmospheric pressure in the presence of sucrose, $NaHCO_3$ (to increase the pH value to ~6.0 and preventing protein coagulation) and vanilla as a flavouring agent. In some cases, sucrose is partially replaced by glucose and the milk lactose is partially hydrolysed to avoid crystallisation. Due to heating, non-enzymatic browning reaction takes place giving the product its attractive flavour and brown colour. Finally, up to 1000 mg g^{-1} of potassium sorbate (KS) can be added to the final product to inhibit the growth of fungi (Char *et al*., 2005). Due to its characteristics (presence of lactose and ε-amino groups of lysine residues in protein, pH and moisture content) and the processing conditions (i.e. heating for a long time), 'dulce de leche' presents favourable conditions for the occurrence of the Maillard reaction, with possible adverse consequences on the nutritive value of the product (Pavlovic *et al*., 1992) through damage to the essential amino acids.

Though other essential amino acids are also involved, particularly in the advanced stages of the Maillard reaction, lysine is the most affected amino acid as its free ε-amino group can react with carbonyl groups (Hurrell, 1990; Mauron, 1990; Malec, 2005).

The most relevant technological problem during the manufacture of 'dulce de leche' is its physical stability as related to the prevention of lactose crystallisation. Crystallisation causes a sandy texture and lowers the product acceptability (Sabioni *et al*., 1984a; Carić, 1994). Lactose crystals tend to aggregate, which alter the physical character of the product and, under normal conditions for dairy products, the α-lactose monohydrate is the major determinant of the nature and degree of crystallisation (Nickerson & Moore, 1973). According to Hough *et al*. (1990), lactose crystallisation in 'dulce de leche' is inevitable due to the milk composition (g 100 g^{-1}) (i.e. 12 total solids and 4.5 lactose). Lactose concentration in dulce de leche may reach 9.85 g 100 g^{-1} and, considering the water phase, the lactose concentration is 33 g 100 g^{-1} water. In general, the solubility of lactose at 15 and 30°C is 16.9 and 24.8 g 100 g^{-1} water, respectively. Thus, even without the interference, lactose in 'dulce de leche' is initially in a supersaturated solution and this is compounded by the simultaneous presence of sucrose (146 g 100 g^{-1} water), which substantially reduces the solubility of lactose. With lactose crystal sizes below 6 μm, sandiness is not detected, even if all lactose in 'dulce de leche' is in the crystallised form. Above this size, the detection threshold depends on the number of crystals and, in sweetened and condensed milk according to Buyze (1952), the acceptable size of the lactose crystals is 10–20 μm.

In Argentina and Brazil, efforts have been made to control the sandiness problem using different methods, such as seeding the product with lactose or using enzymes; the latter method is costly (Martinez *et al*., 1990; Sabioni *et al*., 1984a, b). Seeding, apparently is a good technique to force crystallisation in condensed milk (Buyze, 1952) but, according to Sabioni *et al*. (1984b) and Martinez *et al*. (1990), 'dulce de leche' manufacturers face certain technical difficulties in the application of this technique, such as controlled cooling and proper seeding techniques. In addition, it increases total operation time and induces air bubble formation in the product due to agitation and product contamination. There are two brief reports (Christiansen *et al*., 1987; Edelsten *et al*., 1987) using UF processing for the production of 'dulce de leche' in which sandiness was prevented; however, no more information was given. Carić (1994) reported that UF can be used to prevent lactose crystallisation. Martinez *et al*. (1990) reported that the effect of sandiness in sweetened condensed milk could be prevented by seeding with lactose microcrystals, but seeding has not been used during the manufacture of 'dulce de leche' due to its high viscosity and possibility of contamination problems at the recommended seeding temperature (30°C). They also reported that UF technology is not economically feasible in Argentina.

In 'dulce de leche' manufacture, the main ingredient supplying the solids is normally either whole or skimmed milk; however, concentrated milk may be used to reduce the processing time. According to Lees & Jackson (1992), the presence of milk solids in caramelised products causes the product to be different in its properties to other types of confectionery, mainly in texture, flavour and colour. Similarly, the higher the level of milk solids present in caramel, the harder will be the product with casein being the component that contributes to the hardness.

The function of milk protein, in 'dulce de leche' – a toffee-like product, is complex according to Stansell (1990). Apart from the reaction with reducing sugars to provide the characteristic flavour and colour, which is apparently specific to milk protein, it also stabilises the emulsion of fat in the sugar phase possibly by binding some of the water. In addition, the function of the fat is to provide the 'chewing' characteristics in the product, good texture, colour and flavour. Low fat levels tend to produce products, which are sticky and difficult to chew and, when high fat is used without the addition of emulsifier, it leads to oiling on the surface of the confectionery product (Lees & Jackson, 1992).

Sucrose is one of the basic ingredients used for classical sugar-based confectionery. It is a disaccharide, which can be broken down into a mixture of two monosaccharides known as dextrose (glucose) and *lae*vul*ose* (fructose) by inversion; the inversion is promoted by the action of acid, heat and mineral matter. Sugar is readily soluble in water and, at room temperature, two parts of sugar (67 g 100 g^{-1}) will dissolve in one part of water. The solubility rises to 83 g 100 g^{-1} at 100°C and, when sugar is present in a solution together with invert sugar and/or glucose syrup, a higher total concentration of the mixed sugars can be achieved than may be obtained with the individual sugars alone (Fabry, 1990). Furthermore, the amount of sucrose that is used for the processing varies from 18 to 28 kg 100 L^{-1} of milk (Santos *et al*., 1977). It is very common to add around 2.0 g glucose L^{-1} of milk in order to improve the brightness and texture of the product. The fat content is also important for yield and texture (Martins & Lopes, 1980). The low a_w of 'dulce de leche' contributes to its preservation; however, this induces sandiness (Santos *et al*., 1977), which is a defect observed during sensory profiling of the product. Alternative sugars such as glucose are generally used to replace a proportion of the sucrose in confectionery products in order to modify the sweetness and/or textural properties (Pepper, 1990). The monosaccharide glucose (dextrose) occurs widely in nature where it is found, together with fructose, in most fruits and in honey. It can be derived from starch by enzymatic hydrolysis or, alternatively, may be produced from sucrose by hydrolysis (inversion) to its constituent's glucose plus fructose, followed by separation.

Glucose is commercially available in either monohydrate or anhydrous form. The monohydrate form, containing about 9 g water 100 g^{-1}, is most commonly used in the confectionery industry and the anhydrous form during the manufacture of chocolate. Glucose has a lower sweetness, lower solubility and lower viscosity than sucrose. It is a better humectant and provides better preservative properties owing to its lower a_w. Since it is a reducing sugar, glucose is more reactive than sucrose. However, glucose solutions have a greater tendency to browning on boiling (particularly between pH 5 and 6), and participate more readily in the Maillard reaction with the proteins. According to Hunziker (1934), the use of glucose and other sugars in sweetened condensed milk has a positive effect in preserving the product because of the osmotic effects, which inhibits microbial growth.

In 'dulce de leche' manufacture, replacement of 5–15 g 100 g^{-1} of the sucrose with glucose will have the effect of lowering the overall crystal size and/or smoothing the confection. However, glucose will also increase the tendency of crystallisation during manufacture (Pepper, 1990). Glucose syrup is widely used in the industry to replace part of the sucrose in the formulations; this is due to the fact that sucrose solubility in the formulations can only give 67.1 g 100 g^{-1} at 20°C, Thus, if the product is intended to be concentrated to >70 g 100 g^{-1}, glucose syrup has to be used. Another reason for using glucose syrup

in 'dulce de leche' manufacture is that, if sucrose is added, the supersaturation point of lactose is then reached (i.e. causing the formation of crystals) at a lower milk solids concentration but, in the presence of glucose syrup, much higher solids can be obtained before saturation of lactose is achieved (Howling & Jackson, 1990). In addition, glucose syrup has an influence on the plasticity of the product. Nevertheless, glucose syrup is used as a 'doctor' to replace some of the sucrose used in order to diminish the development of sandiness in the product (Pepper, 1990). The high solute concentration of 'dulce de leche' results in a_w usually below 0.85 (Ferramondo *et al*., 1984), which constitutes the main preservation factor in this product. The stability of 'dulce de leche' to bacterial spoilage at room temperature is well known, even under household conditions. However, growth of yeasts and moulds may occur when the product is stored at room temperature for long periods of time.

Figure 4.4 shows the flow diagram of the manufacturing stages of 'dulce de leche'. According to Hough *et al*. (1990), a standard initial formulation is 10 parts of milk and

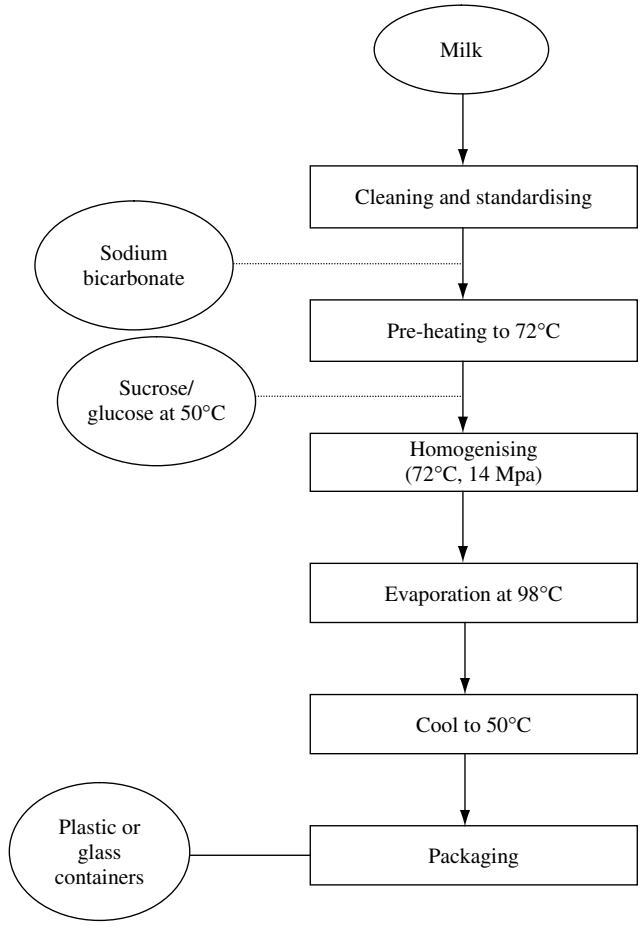

Fig. 4.4 Flow diagram showing the manufacturing stages of 'dulce de leche'.

2 parts of sucrose. A typical Mexican recipe would be a quantity of milk and 20 g 100 g^{-1} of sucrose; if glucose syrup is added, it should be at 10 g 100 g^{-1} of the sucrose weight, but it should be subtracted from the overall original weight of the sugar. According to Hunziker (1934), sucrose and glucose should be added to the milk when it is hot (40 to 50°C) to dissolve these sugars in the solution, and this applies also to 'dulce de leche'. The milk and sugar mixture is concentrated to about 70 g 100 g^{-1} total solids by boiling at atmospheric pressure, then the cooling process should be done very quickly to 50°C in order to promote a very uniform crystallisation, and be followed by the packaging of the product. Carić (1994) reported that, in some instances, $NaHCO_3$ is added to the milk for acidity correction before concentration commences. The use of neutralising agents in 'dulce de leche' manufacture is essential because the acidity in milk may range between 0.14 and 0.18 g lactic acid 100 mL^{-1} and, when the sugar(s) and milk mixture is evaporated, the lactic acid is concentrated causing the protein coagulate. For this reason, the acid in milk should be neutralised.

During the evaporation of the milk, the temperature of the product is continuously monitored in order to know when to add the sucrose and the glucose syrup, and also to monitor the holding temperature, which is in the range of 94–98°C. This is because, if the temperature exceeds 100°C, foaming will occur.

Packaging of 'dulce de leche' must avoid moisture losses and microbial contamination. The most common rigid packages used are glass, plastic pots or metal cans; these types of containers have the advantage of hermetic closing. Semi-hard and thermoform-type packages, mainly polypropylene plastic, have the advantage of lighter weight (i.e. when compared to glass and metal cans), but also give adequate protection against oxygen permeability and moisture losses similar to glass and metal cans (Martins & Lopes, 1980).

4.4.3 *Product properties*

Demiate *et al*. (2001) evaluated the chemical composition of 42 Brazilian commercial samples of 'dulce de leche', and the samples had moisture contents ranging from 19.0 to 37.2 g 100 g^{-1}. According to BSR (1997), the maximum moisture content in 'dulce de leche' is 30.0 g 100 g^{-1}; however, 10 of the commercial samples tested were found to contain higher moisture content than the regulation permits. The lactose content varied from 4.7 to 12.7 g 100 g^{-1}, and only one sample declared the use of lactase on the label but, in general, the low content of lactose in most samples was difficult to explain. The sucrose content of all the commercial samples varied from 32.7 to 56.6 g 100 g^{-1}, but only two samples had low sucrose values, which consequently may explain the high moisture content in these products. In most samples ($n = 30$), the sucrose content varied from 40 to 50 g 100 g^{-1}, and the presence of glucose was detected in all samples – amount varied from 0.4 to 3.9 g 100 g^{-1}. Most of samples ($n = 37$) contained 1.0 to 3.0 g 100 g^{-1} of glucose, but the glucose content in the nutritional table of the product only appeared in 26 samples.

In addition, 'dulce de leche' must contain at least 5.0 g protein 100 g^{-1} (BSR, 1997), but the commercial samples analysed showed that the protein content varied from 1.5 to 14.5 g 100 g^{-1}. Considering that 'dulce de leche' is manufactured from milk and sucrose, it is difficult to explain the low protein content compared to the average protein content

observed in milk (3.2 g 100 g^{-1}). However, the addition of whey protein can contribute to some differences in this balance, as well as the addition of starch.

The fat content of the analysed samples varied from none to 8.4 g 100 g^{-1} in skimmed milk 'dulce de leche', but the fat content of 'dulce de leche' should be around 8.0 g 100 g^{-1} (BSR, 1997). However, most of the samples tested had lower values. Due to health interest against milk fat in dairy products, it is possible to suggest that most of the Brazilian commercial samples of 'dulce de leche' were prepared from low-fat milk. On the other hand, the low levels of other components in 'dulce de leche' (i.e. proteins, ash and fat) may suggest, in part, that the concentrated milk used was highly fortified with starch in order to decrease the manufacturing cost of the product.

Demiate *et al*. (2001) proposed a method for enzymatic determination of starch in 'dulce de leche' using dialysis. In the first step, soluble sugar was eliminated from the samples by using dialysis, followed by starch hydrolysis of the retained fraction with a thermoresistent bacterial α-amylase and an amyloglucosidase. The material was dialysed for the extraction of glucose that was quantified by the glucose oxidase colorimetric reactant.

Char *et al*. (2005) analysed the influence of a_w (0.74–0.85), pH (5.5 and 6.0) and addition of KS on growth of *Escherichia chevalieri*, *Aspergillus fumigatus* and *Propionibacterium brevicompactum* in the 'milk jam' stored at 25 or 35°C for 90 days. In general, after a lag period, the growth profiles showed a non-linear behaviour; this pattern being slightly more pronounced under the most adverse growth conditions (i.e. lower a_w and pH values and the presence of KS). The isolated moulds exhibited different growth curves, but the a_w dependence was similar. They could grow at a_w values as low as 0.74 in the absence of KS at both pH values. A decrease in a_w from 0.85 or 0.83 to 0.74 provoked not only lower growth rates, but also increased the lag periods; the KS greatly influenced the effect of a_w on the growth of the moulds. The three isolated strains of micro-organisms were not able to grow at a_w0.74 in the presence of 1000 μg g^{-1} KS. At a_w 0.83–0.85 and both pHs, the addition of 1000 μg g^{-1} although KS increased the detection time of *E. chevalieri* to a great extent, it introduced only a small variation on the growth rate values. The combination of low a_w and the presence of KS in the studied ranges seemed to determine the growth velocities for the three isolated micro-organisms.

4.4.4 *Rheological parameters*

The knowledge of rheological behaviour, structure and physicochemical changes in 'dulce de leche' will help to predict how the quality of the product will be affected in different conditions, that is, straight after manufacture until consumption or sell-by date. However, limited studies have been published on this subject.

The rheological behaviour of 'dulce de leche' has been studied performing flow curves in rotational viscometers (Hough *et al*., 1988; Pauletti *et al*., 1990; Rovedo *et al*., 1991) or by using dynamic oscillatory tests (Navarro *et al*., 1999). The product shows time-dependent thixotropic behaviour that could be modelled using Weltman's model (Pauletti *et al*., 1990). The pseudoplastic behaviour with a yield stress of 'dulce de leche' has been modelled using Casson's and Herschel-Bulkley's model (Rovedo *et al*., 1991; Pauletti *et al*., 1990). From all the instrumental methods used to characterise the non-oral texture of 'dulce de leche', the yield stress seems to be the most appropriate test to assess the quality of the product

because it shows the highest discriminating ability and it has the advantage of being a quick and direct method of testing (Ares *et al*., 2006).

Alvarez *et al*. (1989) evaluated the rheological behaviour of sweetened condensed milk, and the influence of temperature on thixotropy and viscosity of different samples. They reported that the flow is time dependent and the rheograms presented the hysteresis loop. The modification of shear stress with time was adequately explained by the Weltman model ($r > 0.990$) and the thixotropy increased the consistency index (K), but did not affect the flow index (n). After the time dependence was eliminated, the flow was Newtonian and the thixotropy disappeared at $>30^{\circ}$C. By contrast, Navarro *et al*. (1999) studied the rheological properties of Argentinean 'dulce de leche' and detected differences among the products due to the presence of different types of thickening agents. Rovedo *et al*. (1991) evaluated the effect of pH and temperature on the rheological behaviour of samples of commercial Argentinean 'dulce de leche' products and they categorised them as for home use and confectionary use. The former type is consumed as a dessert, whilst the latter type is preferably used in the confectionary industry due to its higher viscosity, which is due to the addition of starch and thickening agent in its formula. The compositional quality (g 100 g^{-1}) of the Argentinean samples examined by Rovedo *et al*. (1991) was milk solids 80, sucrose 16 and glucose syrup 4 for the home use type, but the confectionary use contained 2.0 g starch 100 g^{-1} as a thickener. In both type of products, the total solids content was 71 g 100 g^{-1}. The apparent viscosity of 'dulce de leche' made in a factory for home use changed as an effect of varied pH values (3.0, 4.7, 8.3 and 9.4) and temperatures (25, 40 and 55°C). The acidification decreased the apparent viscosity, whilst at high pH (i.e. alkaline level) the viscosity increased and, in both cases, the apparent viscosity increased during storage. Confectionary type 'dulce de leche' presented higher consistency as a result of addition of 2 g starch 100 g^{-1} and a higher pseudoplastic behaviour than the other type of product tested.

4.4.5 *Results of a research on 'dulce de leche' using the UF process*

Methodology – 'Dulce de leche' was made following a normal formulation for the control using whole milk (Table 4.3). To produce the ultrafiltered 'dulce de leche', a modified recipe was used and it was brought about from some preliminary trials that were carried out varying the level of ingredients; finally, by replacing some of the sucrose by glucose syrup. In both cases, sodium bicarbonate was added to neutralise the warm milk (30°C) or UF retentate to pH 7.0 in order to avoid protein precipitation during processing. The quantity of milk used for the control and ultrafiltered 'dulce de leche' was initially the same, but the milk for ultrafiltered 'dulce de leche' was subjected to a 55% volumetric reduction by the UF process before use. A higher volume reduction can be achieved, but changes in formulation may be expected since some chemical components are lost during the UF process due to the loss of lactose, minerals and some non-protein nitrogen in the permeate.

The manufacture of 'dulce de leche' was carried out by placing the milk or UF milk retentate in a steam-heated boiling pan. The temperature was controlled throughout the process using a hand-held thermometer. In both cases, glucose syrup was added at 48°C and sucrose at 60°C; thus, avoiding the possible problems with lactose crystallisation at the beginning of the process, as well as achieving adequate solubility of both the glucose

Table 4.3 Formulations for the manufacture of 'dulce de lech'.

	Milk 'dulce de leche'		UF 'dulce de leche'	
Ingredient	(kg)	(g 100 g^{-1})	(kg)	(g 100 g^{-1})
Whole milk	3.5	83.30	–	–
UF milk retentate	–	–	2.1	6.9
Sucrose	0.5	12.50	0.7	23.2
Glucose syrup	0.2	4.20	0.2	7.7
Vanilla	0.004	0.01	0.004	0.1
Sodium bicarbonate[a]	0.003	–	0.004	

UF = ultrafiltration.
[a]For adjustment to pH 7.

syrup and sucrose. The processing temperature ranged from 96 to 98°C with constant stirring.

To determine the final concentration of the product, a hand-held sugar refractometer was used, and then the batches were checked to a final reading of approximately 70 g 100 g^{-1} concentration, as recommended by Hough *et al*. (1990). Although the refractometer was easy to use, it only had an accuracy of about ±2% for measuring the final concentration of the total solids in the 'dulce de leche' when taking the clarity of the scale interface into account. Once the product was ready, and before cooling, 4 mL of vanilla was added for flavouring and the cooling process was continued with a constant stirring to 50°C for packing in 100 g food-grade plastic, screw-top containers.

For the UF-'dulce de leche' process, the UF milk retentate contained 22.8 g total solids 100 g^{-1} at 30°C, but the total solids content (46.70 g 100 g^{-1}) of the product (i.e. mixture of UF milk retentate, glucose syrup and sucrose) was determined from a sample at 60°C. The mixture was then heated to temperatures ranging from 94 to 98°C until the final concentration was reached(~70 g 100 g^{-1}). The time taken for this process was 1 h 40 min.

For the control 'dulce de leche', the process was similar to that of UF 'dulce de leche'; however, the starting temperature of the milk was 34°C with 12.65 g total solids 100 g^{-1}. Glucose syrup and sucrose were added at the same temperature as for UF 'dulce de leche', and the total solids of the partially concentrated milk at 60°C was 27.21 g 100 g^{-1}. The concentration of the mixture was carried out at a temperature ranging from 94 to 98°C to ~70 g total solids 100 g^{-1}. The time taken for the process was 2 h 40 min.

The processing time for UF 'dulce de leche' starting with UF milk retentate was less than that for normal 'dulce de leche'. The production cost of UF 'dulce de leche' may be less than that of normal 'dulce de leche', when the total processing time and energy requirements are considered.

Compositional quality (g 100 g^{-1}) of UF 'dulce de leche' and the control product is shown in Table 4.4 [e.g. the total solids of the control (70.49%) which is 1.78 g 100 g^{-1} higher than that of the UF 'dulce de leche' (69.26)]. The ash content of the control (1.76) is almost 24% higher than that in the UF 'dulce de leche', demonstrating the demineralising

Table 4.4 Chemical composition (g 100 g^{-1}) of 'dulce de leche'.

Sample description	Ash	Protein	Sucrose[a]	Lactose[a]	Other carbohydrate[a]	Total carbohydrate[1]	Fat	Total solids
UF 'dulce de leche'	1.42[b]	7.20[b]	34.90	5.21	5.21	11.63	8.90[a]	69.26[a]
Control 'dulce de leche'	1.76[c]	6.91[c]	32.21	10.52	10.52	10.74	8.35[b]	70.49[b]
SE Difference	0.032	0.122	–	–	–	–	0.102	0.800

[a]All carbohydrates were determined by calculation of ingredient added.
[b,c]Means within the same column followed by the same letter are not significantly different.

effect of the UF treatment of the whole milk. Sucrose levels were similar as required by the product formulation, but the lactose content of the UF 'dulce de leche' (5.21%) was ~50% lower than that of the control (10.52). The lower lactose level in the UF 'dulce de leche' is the main reason for the significantly reduced sandiness in this product. The protein content of the UF 'dulce de leche' was marginally higher (4.0%) than the control and the fat content was 6.2% higher mainly due to variations in the formulations arising from the use of different ingredients.

Mineral content in the dulce de leche was affected by the chemical partition effect during the UF process, separating some of them into the UF milk retentate and some of them into UF permeate giving the product less minerals compared with the milk used to make the control product, with the exception of calcium and phosphorus. The results (Table 4.5) show that UF 'dulce de leche' and the control were statistically different ($P < 0.05$). For instance, the calcium content in UF 'dulce de leche' was higher than the control, that is, 414 mg 100 g^{-1} and 217 mg 100 g^{-1}, respectively, and the phosphorus level was also higher in the UF 'dulce de leche' (285 mg 100 g^{-1}) than the control (174 mg 100 g^{-1}). By contrast, the magnesium content was higher in the control than the UF 'dulce de leche' with 34 mg 100 g^{-1} and 22 mg 100 g^{-1} of products, respectively. Similarly, the potassium and sodium contents were higher in the control than the UF-type product, which were 274 mg 100 g^{-1} and 107 mg 100 g^{-1}, respectively.

Consistency of 'dulce de leche' samples was measured in terms of the penetration resistance of a given probe in Newtons. The UF 'dulce de leche' showed more resistance to penetration by the probe but, the warmer the temperature, the softer the product became and *vice versa* in each case. However, comparing the two samples, the UF 'dulce

Table 4.5 Mineral contents (mg 100 g^{-1} on dry basis) of 'dulce de leche'.

Sample description	Calcium	Phosphorus	Magnesium	Potassium	Sodium
UF 'dulce de leche'	414[a]	286[a]	23[a]	189[a]	88[a]
Control 'dulce de leche'	218[b]	174[b]	34[b]	271[b]	108[b]
SE Difference	30.22	17.28	0.78	13.43	3.18

UF = ultrafiltration; SE = standard error.
[a,b]Means within the same column followed by the same number are not significantly different.

de leche' was always firmer than the control. This may be due to the microstructure of the UF product being built up with slightly more proteins, sucrose and fat, as well as 50% less lactose and about 8% more of other carbohydrates than the control. This created a plastic compact glassy matrix that would give more resistance to penetration by a probe. The protein–carbohydrate ratio was about 7.7% higher in the UF 'dulce de leche', and this may have enabled the protein to establish a firmer network in the matrix. Overall this affected the organoleptic characteristics, giving the UF product a more sticky texture.

Microbiological enumeration – The UF 'dulce de leche' is a microbiologically safe product, which is stable during storage. The microbiological quality of the 'dulce de leche' is examined, even though it is supposed to be a low bacterial growth product due the high sugar concentration. Yeasts and moulds are the only micro-organisms that may grow in 'dulce de leche' and, in most cases, due to external contamination. One day after processing, the products stored at room temperature were analysed for yeast by checking for the presence of gas being produced. The results showed that yeasts could not be detected at the dilution tested in all the products. However, after eight months storage, the products were analysed for coliforms, total viable counts and yeasts and moulds using two media. One medium was prepared with Ringer's solution containing 20 g sucrose 100 g^{-1} and another one was prepared with only Ringer's solution with no added sucrose. The reason for this was to check whether the micro-organisms are affected by changes in sugar concentration of their habitat. The total bacterial, coliforms and yeast and mould counts in all the samples tested (e.g. control and UF products) using Ringer's solution with added sucrose were $<10 \times 10^1$ colony-forming units (cfu) g^{-1}, but the yeasts and moulds in one of the duplicate sample of UF 'dulce de leche' tested were 20×10^2 cfu g^{-1}, which may be due to external contamination during packing. Although similar counts were observed for all the products using Ringer's solution without sucrose, on some occasions the counts were 2×10^1 cfu g^{-1}. According to the results for both the Ringer's solutions, micro-organisms are largely affected by changes in the sucrose concentration, and false results can be obtained if this is not considered. Thus, Ringer's with 20 g 100 g^{-1} sucrose should be used for routine microbiological analysis of 'dulce de leche'. In addition, coliforms were checked using the most probable number (MPN) method, and in all cases they gave results for <3 cfu g^{-1}.

According to the microbiological examinations, 'dulce de leche' is a safe product, but control of the growth of osmophilic yeasts and moulds has to be considered in the manufacture of the product.

Microscopy plays a very important role in the food industry. In this case, 'dulce de leche' samples were analysed using light microscopy (LM) and transmission electron microscopy (TEM) in order to characterise the microstructure. LM of 'dulce de leche' was used to analyse the number, rate of growth and structure of the lactose crystals in six fields using $\times 100$ magnification. The number of crystals present in UF 'dulce de leche' and the control products were kept under inspection during 125 days of storage at 4 and 30°C. Table 4.6 shows the average results of 6 fields taken every 5th day up to 65 days and then monthly. In UF 'dulce de leche' stored at 4°C no lactose crystals were found in the fields from the first to the last day. The same product stored at 30°C showed only one crystal in the fields initially, but this did not increase in size during storage, and no new crystals

Table 4.6 Number of lactose crystals in 'dulce de leche'[a].

	UF 'dulce de leche'		Control 'dulce de leche'	
Day	4°C	30°C	4°C	30°C
1	0	1	0	0
5	0	1	0	0
10	0	1	9	3
15	0	1	21	6
20	0	1	30	10
25	0	1	35	13
30	0	1	38	17
35	0	1	39	19
40	0	1	39	20
45	0	1	39	20
50	0	1	39	21
55	0	1	39	21
60	0	1	39	21
65	0	1	39	21
95	0	1	39	21
125	0	1	39	21

UF = ultrafiltration.
[a]Results are an average of 6 fields in every slide and were taken from the average of every 5th day.

were formed. The control samples stored at 4°C did not show crystals initially, but after the 10th day crystals started appearing with 39 visible on the 35th day when the number was constant until the end of the trial. The control 'dulce de leche' stored at 30°C showed a similar pattern, but to a lesser extent; 3 crystals appeared on the 10th day, and these increased to 21 crystals by day 50, and then remained constant to the end of the trial.

The microstructure of UF 'dulce de leche' at the 10th day at 4 and 30°C after processing is shown in Figure 4.5; no lactose crystals were found at any temperature. However, in the control 'dulce de leche' (Fig. 4.6), the numbers of lactose crystals were different for the product stored at 30 and 4°C. Figures 4.6a and 4.6c appear to show slightly fewer lactose crystals compared with Figures 4.6b and 4.6d (product stored at 4°C), and this agrees with the average values shown in Table 4.6. The storage temperature of 4°C at this stage seems to promote the growth of crystals (Figs 4.6a and 4.6b). This may be due to the low-lactose solubility at this temperature, since at higher temperatures lactose increases in solubility.

After 65 days of storage at the same temperatures (4 or 30°C), the UF 'dulce de leche' had no observable lactose crystals at ×100 magnification at either temperature of the

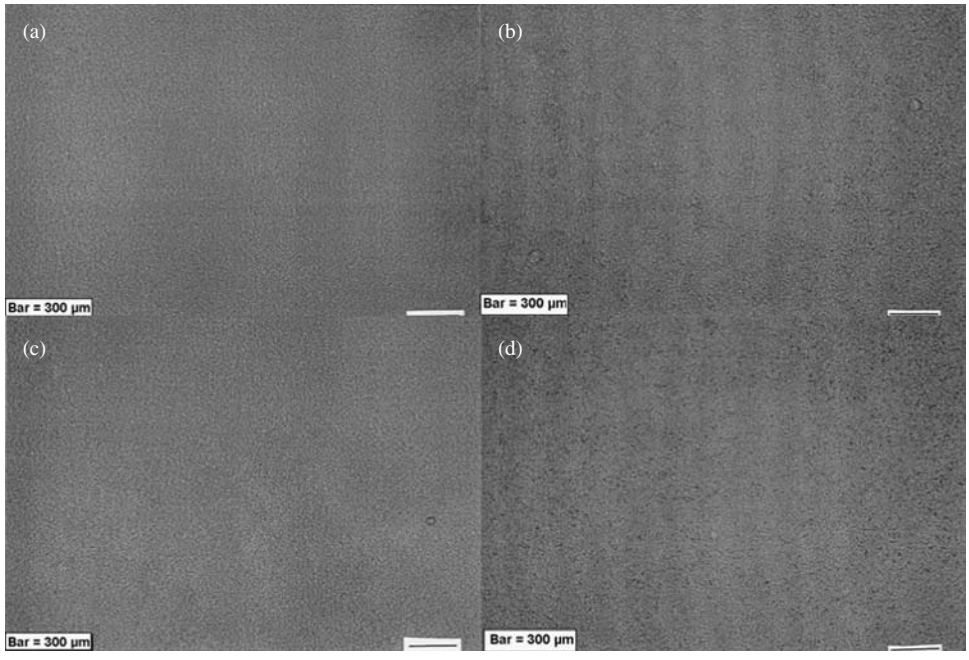

Fig. 4.5 Ultrafiltered 'dulce de leche'. Note: (a) after 10 days of storage at 4°C, (b) after 65 days of storage at 4°C, (c) after 10 days of storage at 30°C and (d) after 65 days of storage at 30°C.

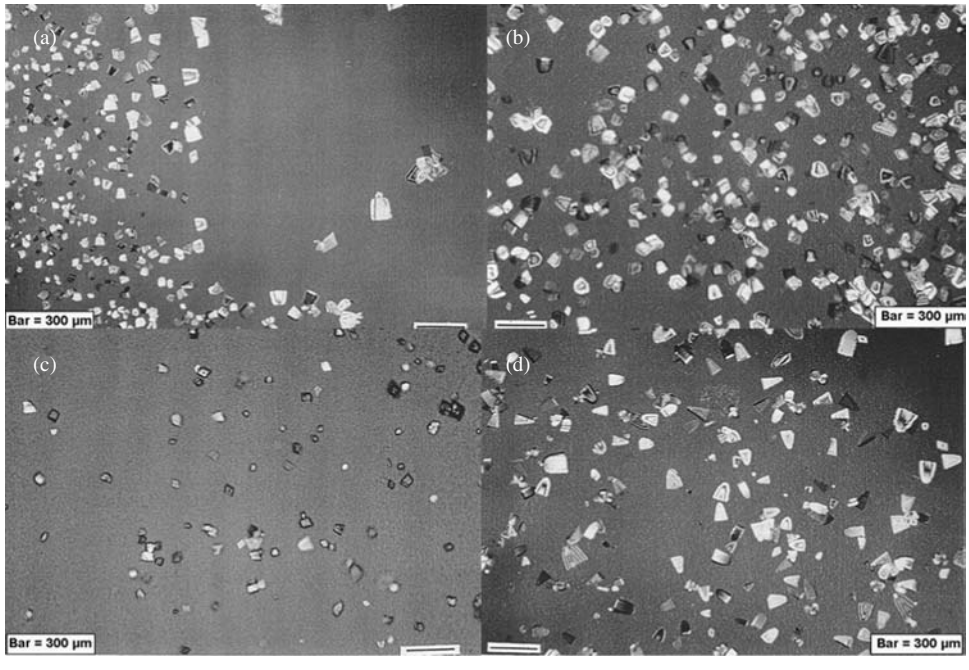

Fig. 4.6 Control 'dulce de leche'. Note: (a) after 10 days of storage at 4°C, (b) after 65 days of storage at 4°C, (c) after 10 days of storage at 30°C and (d) after 65 days of storage at 30°C.

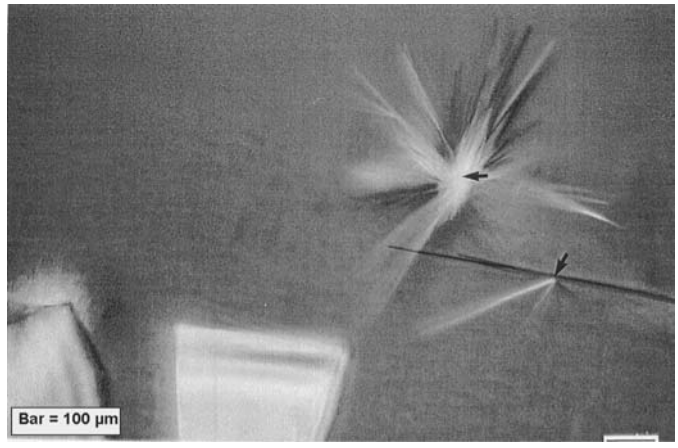

Fig. 4.7 The new form of crystal in control 'dulce de leche'.

storage (Fig. 4.5). However, the control 'dulce de leche' images presented an increase in the number of lactose crystals (Fig. 4.6); at 4°C, crystals of similar size were formed, whereas at 30°C a wider range of lactose crystal sizes were produced. This may be due to the higher solubility of lactose at 30°C, leading to preferential growth of larger crystals rather than formation of new, smaller crystals. In general, conditions favouring slower crystal growth will result in fewer but larger crystals.

A new form of lactose crystal was found after 30 days of storage at 30°C for the control 'dulce de leche'. This new form has a spherulite shape (see arrows in Fig. 4.7), which has a central intercept point for all the elongated components. This new form does not follow any specific pattern with respect to the distribution and the size of the elongated components. The only common characteristic is the central intercept point. The new crystal form does not correspond to any other carbohydrates because they do not achieve saturation levels in the 'dulce de leche'. One possible explanation is that, during the crystallisation of lactose, α-lactose monohydrate is the usual crystalline form obtained from aqueous lactose solutions. However, if crystallisation takes place at high concentrations of lactose, the solution could be supersaturated with respect to both α- and β-lactose. Theoretically, both forms of lactose may crystallise, irrespective of the temperature (Roetman, 1981). When crystallisation takes place, it goes from a saturated solution to a glass state, increasing the viscosity of the solution. Warburton & Pixton (1978) have reported that there are several shapes of lactose α-hydrate crystals and, which one is formed depends on the conditions of growth. They have also reported that when precipitation pressure is high and crystallisation is forced, prism shapes are produced. The form changes with decreasing pressure to diamond, pyramid, tomahawk and 13-sided crystals, but irregular crystals may be found due to the presence of impurities in some dairy products. However, there is no information about β-lactose crystal forms.

The sizes of the lactose crystals were inspected by detecting randomly the crystals in the microscope slide using ×100 magnification. The number of useful fields of view on the slide varied when measuring crystals in UF 'dulce de leche', particularly when the incidence of crystals was rare; hence, the crystal size was more stable than in the control

Table 4.7 Size (μm) of lactose crystals in 'dulce de leche'[a].

	UF 'dulce de leche'		Control 'dulce de leche'	
Day	4°C	30°C	4°C	30°C
1	10	10	0	0
5	10	10	0	28
10	10	10	59	69
15	10	10	89	119
20	10	10	129	208
25	10	10	158	247
30	10	10	178	297
35	10	10	198	317
40	10	10	198	327
45	10	10	208	327
50	10	10	228	327
55	10	10	228	327
60	10	10	228	327
65	10	10	228	327
95	10	10	228	327
125	10	10	228	327

[a]Results are an average of 6 fields in every slide and were read every 5th day.

product (see Table 4.7). When samples of UF 'dulce de leche' product were stored at 4 and 30°C, in both cases the average result was that one crystal with a size of 10 μm from the first day was constant to the 65th day. The presence of a lone crystal may be due to the presence of a dust or gas bubble nucleus and/or the application of mechanical shock or ultrasonic vibrations (Brennan *et al*., 1976). On the other hand, the control 'dulce de leche' stored at 4°C showed no crystal growth until the 10th day with an average size of 59 μm and kept growing until the 50th day with a final size of 228 μm. Afterwards, the size remained constant until the end of the trial. In the control product stored at 30°C, the first crystals were detected on the 5th day averaging 28 μm, and they grew until the 50th day to 337 μm, thereafter remaining constant to the final day. These results, for both the number and the size of the crystals, illustrate the use of UF milk retentate as an ingredient in 'dulce de leche' manufacture, which helps to prevent the presence and formation of lactose crystals.

Transmission electron microscopy – The stability of the casein micelles is influenced by several treatments, such as acidification, heating and addition of Ca^{2+} and, in particular, during heat treatment various physical and chemical changes occur in casein micelles, whey proteins, lactose and salts, affecting their functionalities in milk products. When milk is heated in the temperature range of 90–140°C and at pH values below 6.7, denatured whey proteins complex on to the micellar surfaces, involving κ-casein but, at higher pH values,

denatured whey proteins remain in the intermicellar fluid as fibrous strands (Creamer & Matheson, 1980). Dalgleish *et al*. (1987) suggested that the increase in the casein micelle diameter on heating milk is thought to be due to the deposition of denatured whey proteins on to the micellar surfaces and precipitation of calcium phosphate. Carroll *et al*. (1971) have noted a doubling of casein micelle size in sterilised concentrated milk (26 g solids 100 g^{-1}) compared with fresh milk, and this implies increased aggregation of casein micelles. They have also suggested that the increased level of calcium in concentrated milk may lead to calcium bridging between micelles with a subsequent increase in micelle size. In this study, the calcium content in UF 'dulce de leche' was almost double than in the control 'dulce de leche'.

The increase of casein micelle size in the UF 'dulce de leche' does not affect the stability of the product, since it is formed mainly by a glassy sugar matrix, where the casein micelles take a secondary role in the microstructure of the product. Figures 4.8, 4.9 and 4.10 show the structure of UF 'dulce de leche' at ×7500, ×20000 and ×50000 magnification, respectively. In all illustrations, the proteins are the main components (see arrows 'C' in Fig. 4.8), probably consisting of denatured whey proteins on the surface of the casein micelle (see arrow 'W' in Figure 4.10). However, in Figure 4.10 as indicated with arrow 'W', faint fibrous strands are seen on the surface of the casein micelle. In this case, the proteins are slightly more prominent, forming more extensive clusters. In neither the UF nor the control 'dulce de leche' were the carbohydrates, fat or minerals evident because the samples were prepared for protein fixation.

Final considerations – In the manufacture of 'dulce de leche', a large quantity of water is removed by evaporation during a certain time period. This process is normally costly since evaporation is carried out by applying steam, and this increases the production costs in 'dulce de leche' manufacture. The UF process, as a means of providing a dairy concentrated product, can decrease the time taken by evaporation of water, since UF retentate can be concentrated to different degrees. UF retentate as a dairy ingredient in 'dulce de leche' manufacture will produce a low-lactose product, so preventing the formation of large lactose

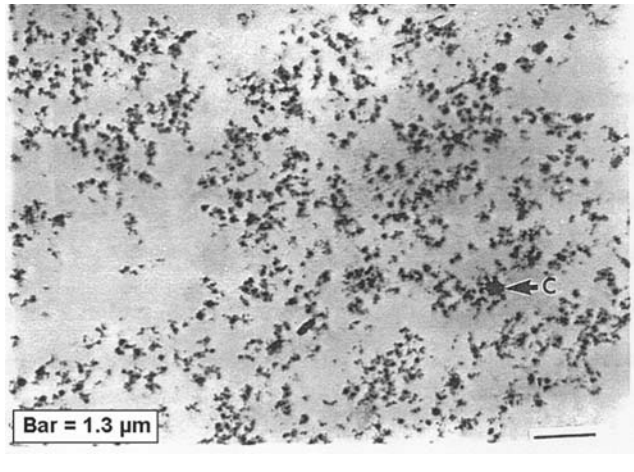

Fig. 4.8 Transmission electron micrographs of ultrafiltered 'dulce de leche' at ×7500 magnification.

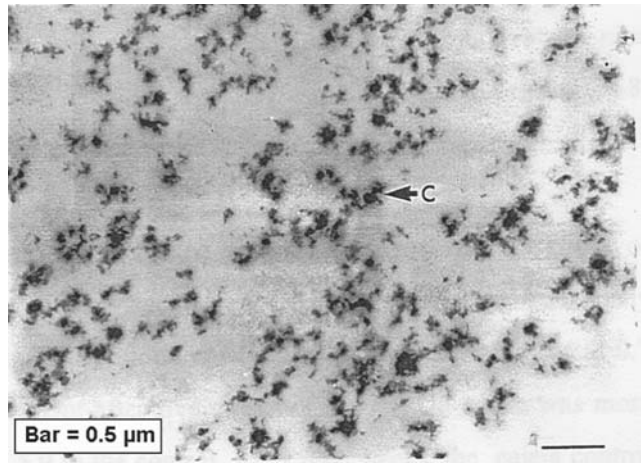

Fig. 4.9 Transmission electron micrographs of ultrafiltered 'dulce de leche' at ×20 000 magnification.

crystals which cause sandiness. As 'dulce de leche' is subjected to a heat concentration process, the lactose is also concentrated. In addition, lactose crystals in 'dulce de leche' start appearing if the concentration of lactose exceeds its solubility in solution. Once sandiness is present in the product, it tends to reduce consumer acceptance as a result of the presence of a grainy texture. In this product, important roles of the milk are in the development of colour and flavour, and in holding moisture within the sugary matrix; UF milk retentate appears to perform well in both aspects.

The overall organoleptic attribute of 'dulce de leche' is strongly influenced by sandiness. UF milk retentate in 'dulce de leche' manufacture is an ingredient, which supplies a low amount of lactose and which improves the organoleptic characteristics of 'dulce de leche' even after being in storage for some months. These preliminary results give a base for

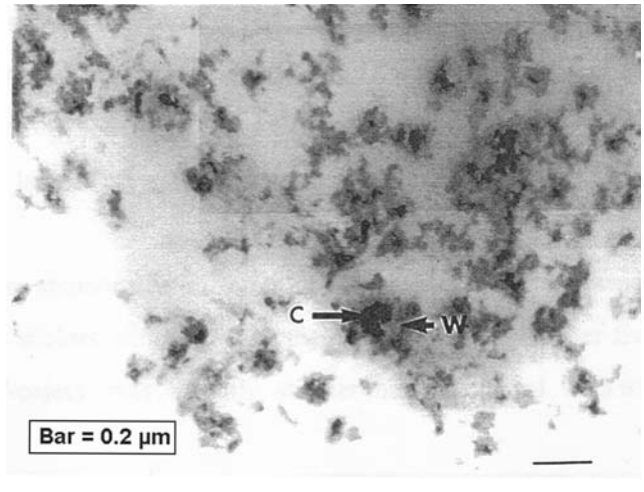

Fig. 4.10 Transmission electron micrographs of ultrafiltered 'dulce de leche' at ×50 000 magnification.

'dulce de leche' manufacture, although individual manufacturers will need to undertake some product development to achieve their individual product requirements. In particular, some attention will need to be given to establish the levels of volume reduction. Furthermore, the use of UF milk retentate as an ingredient for the production of 'dulce de leche' may offer the possibility of adjusting the mass ratios of different milk constituents without adversely affecting their physicochemical characteristics and the functionalities of the processed product. The relative levels of milk proteins, lactose and minerals in the retentate depend on the extent to which milk is processed by UF process and the conditions used. The general effects of UF milk retentate on the functionalities of 'dulce de leche' are mainly related to the concentration of proteins and the reduction of lactose; however, the main advantages are a reduction of the processing time and a reduction of sandiness in the product. Furthermore, lactose crystallisation is prevented in low-lactose 'dulce de leche'. Microscopic analysis corroborates the absence of lactose crystals in UF 'dulce de leche' and the use of UF milk retentate will improve the quality and shelf life of the product without the necessity of using enzymatic methods to hydrolyse the lactose.

In conclusion, UF milk retentate is suitable for 'dulce de leche' manufacture, and the research work has provided pointers to the alternative uses of UF retentate, although the value of some of these suggestions still needs to be demonstrated. Polarised LM clearly showed lactose crystal growth, and this related well to increased granularity on storage. Not unexpectedly, lactose crystal growth was effectively prevented by the use of UF milk retentate in 'dulce de leche', whilst in the control 'dulce de leche', some interesting observations were made in connection with storage at 4°C and 30°C. The consistency of 'dulce de leche' is such that crystallisation will not be delayed to any great extent by the viscosity of the matrix (as might be the case in high sugar boiling or in milk powders) and, consequently, lactose crystallisation is inevitable over a relatively short timescale. The fact that crystallisation occurred more rapidly at 4°C confirms the view that lactose insolubility is the rate determining process, while the fact that samples stored at 30°C produced large crystals is consistent with a lower driving force for crystallisation. It is curious, however, that the 30°C storage temperature produced a second type of crystal; the majority of crystals at both temperatures of storage were of the characteristic truncated tomahawk shape associated with α-lactose monohydrate. At 30°C and 30 days' storage, a spherulite-type crystal form was also seen, which may imply that a change in composition of the matrix as the lactose is removed by crystallisation has produced conditions where a higher hydrate of lactose, or possibly some β-lactose, has crystallised. In technological terms, this is of little immediate significance, but it indicates the complexity of crystallisation phenomena in complex mixtures and illustrates how fairly small changes in composition can affect the behaviour of these systems. Lactose removal is a key role for UF in terms of controlling the functionality of products. This research on 'dulce de leche' using the UF process has shown that, in addition to the anticipated changes, the complexity of mixed systems retains some mysteries.

4.5 Conclusions

New developing markets for dairy products, particularly in Southeast Asia, and the emergence of new entrant countries with rapidly expanding milk production is changing the

dynamics of the world dairy industry. Concentrated milk products have several advantages, including (1) storage – requires small space under regular storage conditions and retains high quality at the same time; (2) economy – because mass and volume are reduced, transportation costs are less; and (3) versatility – can be used under adverse conditions, such as wars, epidemics or earthquakes when fresh milk is unavailable. However, the energy consumption for concentration is high and costly. Besides the processing of the milk products, an important criterion for preservation by concentration and drying is the quality of the (recombined) product. Modern technologies are focused on minimising the loss of nutritive value and improving microbiological quality of the milk product.

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5 Dried Milk Products

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and D.D. Muir

5.1 Introduction

The purpose of this chapter is to provide the reader with a relatively short but thorough introduction to milk powder manufacturing (see Figure 5.1), with reference to key processing tools and parameters as well as quality aspects for raw materials and final products. After powder definitions and classification, the chapter covers heat treatment of the milk and its resulting effects on powder properties, agglomeration and instantising methods and the resulting products, typical processes and parameters for ordinary, instant and other milk powders, and finally, quality aspects together with methods of analysis for milk, milk concentrate and milk powder.

The reader is expected to be familiar with the basic functionalities of the main processing equipment included in the manufacturing process, such as evaporators and spray dryers. If this is not the case, Chapter 3 gives an introduction to this equipment (see also Westergaard, 2004).

The legal aspects of milk powder specifications are not included as they are dealt with in Chapter 2. The products covered in this chapter are the main products from the milk powder industry only. Therefore, secondary products, such as powders of buttermilk, cream and yoghurt, products made to imitate milk products; and products with major additions of non-dairy ingredients, are not included. Other exclusions are dried milk products with a significantly changed composition, such as casein and whey products and their derivatives; these products are reviewed in Chapters 6 and 7, respectively.

5.2 Definitions

5.2.1 *Composition*

Internationally agreed definitions for the origin, composition and permitted additives to milk powders are detailed in the Codex Alimentarius Commission - *Milk and Milk Products* (FAO/WHO, 2003). The main components are limited to water, milk fat and milk solids-not-fat. Milk and cream powders are defined as 'milk products which can be obtained by the partial removal of water from milk or cream. The fat and/or protein contents of the milk or cream may have been adjusted, only to comply with the compositional requirements in Section 3 of the standard, by the addition and/or withdrawal of milk constituents in such a way so as not to alter the whey protein-to-casein ratio of the milk being adjusted.'

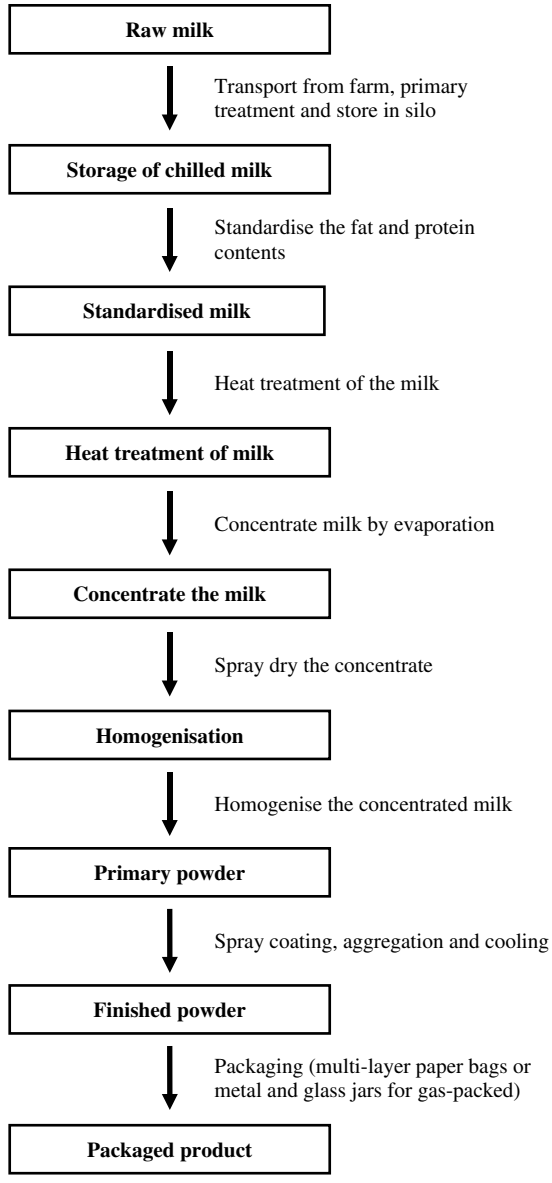


Fig. 5.1 Generalised scheme for the production of whole milk powder (WMP).

The standard specifies that only milk and cream are permitted as raw materials for milk powders. However, the protein content of the powder may be adjusted by addition of lactose, skimmed or partially skimmed milk or by addition of the retentate or permeate obtained from the treatment of milk by ultrafiltration (UF).

The standards also list other permitted additives from within the following groups: stabilisers, emulsifiers (for instant products), acidity regulators, anti-caking agents (free-flowing agents) and antioxidants. No other additives are sanctioned.

Standards are also provided for thresholds for contaminants, production hygiene and product labelling.

5.2.2 *Heat classification*

Milk powders may be heat classified, that is classified according to the severity of the heat treatment they have received. Essentially, heat classification is a measure of the extent of denaturation of whey protein in the powder that, in turn, is indicative of functionality. Two separate indices of the level of whey protein denaturation are employed - whey protein nitrogen index (WPNI) and casein number (CN). Generally, five levels of heat classification are recognised: ultra low-, low-, medium-, high- and high-heat heat stable. The production and specific end uses of these different classes of powder will be discussed later in the chapter.

5.2.3 *Dispersion properties*

Powders manufactured by the simplest of spray drying processes comprise small particles that reflect the size of the fluid drops formed by the atomiser. Such powders are difficult to handle as the fine particles readily form dust. In addition, most powders of this type are particularly difficult to hydrate–an essential part of many applications. Powders in which the small particles have been deliberately removed are referred to as 'dust-free'. Furthermore, some end-uses are even more demanding, for example, some powders are expected to wet easily and disperse with complete dissolution with the minimum of physical effort. This type of powder is referred to as instant. To achieve instant status, the primary particles in powders are agglomerated into clusters which may be further treated by coating the agglomerates with surface-active material. The production of dust-free and instant powders is discussed in detail later in the chapter.

5.3 Microbial quality

The microbial quality of milk powder is determined by the quality of the raw material, the nature and extent of processing and by the extent of post-production contamination. Processing of milk powder follows a complex series of operations (see Figure 5.1). Although processing follows this general pattern, the exact sequence may differ considerably between factories. Because each step in the process can have significant effects on the quality of the milk powder, ultimate powder quality is site-specific. For this reason, the overall effect of the drying of milk is most appropriately considered as the sum of a sequence of unit processes.

5.3.1 *Raw milk*

Raw milk quality plays an important role in powder quality. The initial bacterial load of high-quality raw milk is surprisingly low provided that

- the herd has a good health status;
- the housing of cows is appropriate;

- the milking operation is carried out using best practice;
- the raw milk is stored in clean, well-refrigerated tanks/silos.

Factors affecting quality - The microbiological quality of raw milk is well defined and comprises bacteria that have their origin in the cow and in the environment. The flora can be divided into two groups according to their functionality. First, there are pathogens and potential pathogens. Second, there are spoilage bacteria that can further be divided into Gram-negative and spore-forming types. Subsequent processing operations can affect the survival and growth of these micro-organisms in different ways.

Pathogens found in milk are usually the result of poor udder health, that is, mastitis (mostly subclinical). Nevertheless, contamination from the soil, faeces or bedding is also a potential source of food poisoning bacteria. Among the organisms of concern are *Bacillus cereus*, *Campylobacter jejuni*, *Clostridium* spp., *Escherichia coli*, *Listeria monocytogenes*, *Staphylococcus aureus*, *Salmonella typhii* or *Salmonella typhimurium* and *Yersinia enterocolitica*. These organisms can result in serious illnesses and, in the most severe cases, death. The organisms enter the milk as a result of udder disease, or from faecal contamination of the udder, or from milking equipment. Recent concern has also been expressed over the incidence of *Mycobacterium avium* subsp. *paratuberculosis* in raw milk, but its status as a human pathogen remains to be proven.

Primary control involves prevention of disease and, as a result, of pathogenic bacteria from contaminating raw milk. Udder health is routinely assessed by the measurement of the level of somatic cell counts (SCC) in milk. Financial penalties are routinely imposed on milk with elevated SCC. This encourages milk producers to treat infections promptly, and to adopt prophylactic measures, such as stringent udder cleaning and antibiotic treatment during the 'dry' period between lactations. Adventitious contamination from the environment is controlled by ensuring that the cow, especially its udder, is clean.

It is equally important that once the milk is harvested it only comes into contact with clean and sanitised equipment. This reduces the number of pathogens derived from the environment and inhibits spoilage bacteria from contaminating the milk. Spoilage bacteria comprise mainly of Gram-negative rods and some spore-forming bacteria, mainly *Bacillus* spp.

Control of pathogens in milk is achieved by a combination of refrigerated storage and heat treatment. The effect of pasteurisation (72°C for 15 s) and of storage below 6°C for the major organisms posing a threat to human health is shown in Table 5.1.

Of the major pathogens found in raw milk, only spore-forming bacteria (*Bacillus* spp. and *Clostridium* spp.) survive pasteurisation. However, *Clostridium* spp. are unable to grow at refrigeration temperature. Although spore-forming *Bacillus cereus* is potentially pathogenic (via the excretion of diarrheogenic and emetic toxin), the circumstances in which these toxins are expressed are seldom found in milk. As a result, provided milk is stored in the cold, pasteurised and recontamination is prevented, there is no health risk associated with pathogenic bacteria.

However, no matter how effective disease control and the standards of cleanliness within the farm are, some bacterial contamination of the raw milk is difficult to avoid. The organisms of concern are the spoilage bacteria; these are well adapted to grow in cold temperatures and comprise true psychrophiles and psychrotrophs. Even using best practice, it is difficult to attain counts below 500 colony-forming units (cfu) mL^{-1} in milk *ex* the farm, bulk tank. To avoid subsequent growth of psychrotrophic bacteria, it is essential that

Table 5.1 Control of pathogenic bacteria.

Micro-organism	Survival after pasteurisation[a]	Growth at <6°C
Bacillus cereus	Yes	Yes
Campylobacter jejuni	No	No
Clostridium spp.	Yes	No
Escherichia coli	No	Yes
Listeria monocytogenes	No	Yes
Salmonella typhii	No	?
Salmonella typhimurium	No	?
Staphylococcus aureus	No	No
Yersinia enterocolitica	No	Yes

[a]Heat treatment at 72°C for 15 s or equivalent.
?organism(s) may grow.

milk is promptly cooled to <4°C immediately after milking, and that it is subsequently stored under refrigeration until it is ultimately heat treated during powder manufacture.

The temperature of refrigeration is critical because the growth rate of spoilage bacteria is highly dependent on temperature – at 2°C, most psychrotrophic bacteria grow slowly with generation times in excess of 12 h. However, at 8°C the average generation time is less than 4 h. Typically, after storage for only 24 h, the bacterial count in milk may rise by factors of ×2 and ×64 for storage at 2 and 8°C, respectively. These increases in count may not be significant when the initial counts are low and milk is processed promptly, but assume importance if the initial counts are high, and when the time between milking and processing is extended. For example, it is not unusual for milk to be stored for a further 2 days at the factory before it is converted into product(s). Under this scenario on storage at 6°C (where the average generation time is *ca*. 6 h) the population of psychrotrophic bacteria would be expected to increase by a factor of 4×10^3. Given an initial count of 1000 cfu mL^{-1}, by the time of manufacture the count would have risen in excess of 4×10^6 cfu mL^{-1}.

The psychrotrophic bacteria found in cold stored milk comprise mainly of *Pseudomonas* spp. (of both fluorescing and non-fluorescing types) and, although not pathogenic in nature, they commonly produce extracellular lipase and protease that degrade the milk fat and protein, respectively. Moreover, although most psychrotrophic bacteria are readily destroyed by pasteurisation, the extracellular enzymes they produce are remarkably heat resistant. Typical activity and heat resistance of extracellular enzymes associated with bacteria found in cold milk are shown in Tables 5.2 and 5.3.

Pseudomonas spp. comprise the majority of bacteria in refrigerated silo milk, and clearly they are frequently associated with both lipase and protease activity. In addition (Table 5.3), these degradative enzymes are surprisingly heat resistant. As a result, it is imperative that the count is not allowed to reach a high level where the residual enzyme activity is expressed in the heat-treated milk or in products manufactured from it.

In milk powder, this residual enzyme activity can cause problems when the powder is incorporated in finished goods. For example, soapy defects in chocolate have been

Table 5.2 Extracellular enzyme activity in silo milk.

Type of micro-organism	Frequency (%)	Proportion isolates with stated activity (%)	
		Lipase only	Lipase + protease
Pseudomonas spp. (fluorescing)	40	32	11
Pseudomonas spp. (non-fluorescing)	30	5	71
Enterobacterium spp.	8	2	31
Alcaligenes spp.	1	0	92
Gram-positive bacteria	7	10	12

traced back to defective milk powder. No exact guideline is extant for the acceptable threshold for psychrotrophic bacteria in milk for processing. However, counts in excess of 5×10^6 cfu mL^{-1} have been associated with defects in ultra-high temperature (UHT) milk (gelation associated with proteolysis) and in cheddar cheese (rancidity linked to excessive lipolysis). To provide a margin of certainty, it is prudent to specify that the count of psychrotrophic organisms in milk should not exceed 10^6 cfu mL^{-1} at the point of heat treatment dairy manufacture into powder.

Shelf-life extension - The sensitivity of psychrotrophic bacteria to small increases in temperature and their relatively rapid growth at low temperature may pose a problem if raw milk must be stored for extended periods. In this case, treatment of raw milk is necessary to avoid the threshold bacterial count above which there is a danger from enzyme degradation. Two options are available – thermisation and deep cooling of the incoming raw milk – at the factory. Thermisation involves a heat treatment under conditions less severe than pasteurisation, typically at 65°C for 15 s; whilst deep cooling to 2°C also has potential for shelf-life extension. A combination of these treatments can ensure a safe extension of the shelf-life of raw milk by up to 3 days. It should be noted that thermisation

Table 5.3 Heat stability of extracellular enzymes from psychrotrophic bacteria.

Micro-organism	Proportion of initial activity after heating (%)			
	Lipase		Protease	
	Pasteurisation[a]	UHT[b]	Pasteurisation	UHT
Pseudomonas spp. (fluorescing)	50–80	15–55	58–100	16–48
Pseudomonas spp. (non-fluorescing)	60–78	0–75	22–72	4–45
Enterobacterium spp.	30–98	20–80	5–82	0–58
Alcaligenes spp.	60	36	36–65	8–32
Gram-positive bacteria	–	–	2–40	0

[a]Heat treatment at 72°C for 15 s or equivalent.
[b]Ultra-high temperature – typically 135–150°C for a few seconds.

is not a substitute for pasteurisation because not all pathogens are destroyed. Therefore, even when milk is thermised (or deep cooled) the milk must be pasteurised as an early step in the manufacturing process.

5.3.2 *Effects of milk processing*

Clarification, bactofugation and microfiltration

To reduce contamination by particulate matter and bacteria, milk may be clarified or treated in a bactofuge. Both treatments utilise high-speed centrifugation to separate material of higher than average density from the milk. Mineral soil, somatic cells and bacterial spores – all undesirable contaminants of raw milk – may be removed. However, even under optimum conditions only 95% of the bacterial spores are removed in a single pass through the bactofuge and a second treatment only results in a combined reduction of 99% of the initial spore count.

Microfiltration (MF) is now routinely used for manufacture of pasteurised milk with an extended shelf-life. The membranes are designed to remove almost all spore-forming bacteria (and other bacterial cells), but function effectively only on previously skimmed milk – fat globules blind the membrane pores. The successful application of MF is due to the availability of ceramic microporous membranes with tightly controlled pore size and to the advent of cross-flow geometry in membrane plants. Cross-flow plants control transmembrane pressure and inhibit occlusion of the pores. Although MF is too expensive for routine application to milk powder production, it opens a route to the manufacture of low-heat powders with very low bacterial loads.

Standardisation of the fat and protein contents

In the next step in the processing sequence, the fat content of the raw milk is adjusted or removed completely for the manufacture of SMP. Cream is separated from whole milk using a high-speed centrifugal separator. This device separates the milk constituents/components on the basis of the density difference between the 'light' milk fat globules and the relatively dense serum. The two liquid streams are then recombined to yield milk with the required fat content. In contrast to bactofugation, the overall effect of the fat standardisation on the microbial population of milk is modest.

Standardisation (reduction) of the protein content in the milk involves the addition of permeate from the UF of skimmed milk. For increased protein content, retentate is added. UF partition the bacteria in skimmed milk into the retentate. However, because of relatively small amount of either retentate or permeate used for standardisation, the small effect in bacterial load in the standardised milk is minor ($\pm<20\%$).

Heat treatment

During milk powder production serves two distinct purposes. Not only does it control the microbial quality but it also influences 'functionality'. For example, it is usual to apply a severe heat treatment to milk destined for manufacture of WMP or as an ingredient of

Table 5.4 Heat-resistant genera in milk.

Heat-resistant (survive 60°C for 20 min)	Thermoduric (survive 63°C for 30 min)	Spores (survive 80°C for 30 min)
Streptococcus faecalis	*Microbacterium* spp.	*Bacillus* spp. spores
Lactobacillus spp.	*Micrococcus* spp.	*Clostridium* spp. spores
Corynebacteria spp.	*Alcaligenes* spp.	

bread. Such heating results in denaturation of the whey protein. The presence of denatured whey protein reduces the rate of lipid oxidation during subsequent storage of the powder. Pasteurisation (63°C for 30 min or 72°C for 15 s) kills most pathogens and all Gram-negative bacteria, psychrotrophic and spoilage bacteria. However, a residual population of heat-resistant bacteria remains (Table 5.4).

Heat-resistant *Streptococcus faecalis*, *Streptococcus brevis* and *Streptococcus thermophilus* and *Micrococcus* spp. grow slowly, if at all, at refrigeration temperature. Their presence at high counts is indicative of a fault in the cold chain under which it is stored. Organisms, such as thermoduric bacteria that survive pasteurisation, comprise mainly of Gram-positive organisms. *Alcaligenes tolerans* is the only Gram-negative organism usually found in properly pasteurised milk, albeit infrequently.

The predominant organisms found in heated milk are *Bacillus* spp., which survive the heat treatment in the spore form, and *Corynebacteria* (*Clostridium* spp. cannot grow at the high redox potential of milk). *Corynebacteria* can form a significant proportion of the thermoduric population in milk, but they grow very slowly at refrigeration temperature, and are not implicated in milk spoilage and seldom with pathogenesis. On the other hand, *Bacillus* spp. degrade milk readily and are noted for their phospholipase activity. Spoilage due to these organisms is often associated with damage to the milk fat globule membrane and is characterised by the defect known as *bitty cream*. As described elsewhere, the population of spores in milk can be reduced by clarification, bactofugation or MF. If very low spore counts are required in the product, then severe heat treatments must be applied to the milk – typically 110–120°C for 30 s.

Concentration and homogenisation

Milk is concentrated in an evaporator to 45–55 g 100 g^{-1} total solids before spray drying. Apart from the expected increase in count caused by the concentration process, there is a potential hazard in multi-stage evaporators. The concentrate may be held for extended periods in the temperature range 45–55°C. Some heat-resistant bacteria can grow under these conditions and, as a result, the bacterial count of the concentrate can increase disproportionately.

Pasteurisation of the milk at 72°C for 15 s directly before the evaporation will naturally influence the bacterial count in the final powder and the higher the temperature and the longer the holding, the more efficient the process to inactivate micro-organisms.

The last few years have seen much focus on the content of spore-forming thermophilic bacteria in milk powder. It has been found that after only 12–16 h of operation of a plant,

Table 5.5 Optimal growth and inactivation conditions of some spore-forming bacteria.

Spore-forming bacteria	Growth temperature (°C)			Inactivation in milk by heating	
	Minimum	Optimum	Maximum	Vegetative cells	Spores
Bacillus species					
B. stearothermophilus[a]	30–45	55–60	60–70	12 s at 85°C	8–15 min at 121°C
B. cereus	5–20	30–37	45–48	10 s at 72°C	0.5 min at 121°C
B. coagulans	15–25	35–50	55–60	20 s at 72°C	3–5 min at 121°C
B. licheniformis	15	30–45	50–55	20 s at 72°C	3–5 min at 121°C
B. subtilis	6–20	30–40	45–55	20 s at 72°C	3–5 min at 121°C
Clostridium species					
C. botulinum	3	25–40	48	20 s at 72°C	3–4 min at 121°C
C. perfringens	8–20	45	50	20 s at 72°C	1–4 min at 121°C
C. tyrobutiricum				20 s at 72°C	1–4 min at 121°C

[a]Now known as *Geobacillus stearothermophilus*.

the number of these bacteria starts to increase, exponentially. Spore-forming bacteria are bacteria that under adverse growth conditions, such as too high or too low a temperature or lack of nutrition, transform themselves into a dormant state. They sporulate and become extremely heat resistant. When growth conditions become favourable again, they germinate and develop. The vegetative cells can be killed by heat treatment. Thermophilic bacteria typically grow between 45 and 70°C (Table 5.5).

After concentration of the milk solids, the product is homogenised to reduce the fat globule size and inhibit creaming. However, homogenisation may cause an increase in bacterial count as a result of the disaggregation of bacterial clusters.

Spray drying, agglomeration and coating

Powder is formed by atomising the concentrated milk in a stream of very hot air (typically 180–220°C). However, as a result of evaporative cooling, the temperature of the atomised milk droplet remains low during the drying process. As a consequence, the bacterial load of the concentrate largely determines that of the powder. Provided that the ultimate moisture content of the powder is below 4 g 100 g^{-1}, bacteriostatis is assured. In the case of dried WMP, the moisture content may be as low as 2 g 100 g^{-1}, to inhibit fat oxidation during extended storage. Operations on the powder downstream of the dryer have little further effect *per se* on the bacterial load. Nevertheless, serious deterioration of powder quality can occur from environmental contamination.

Environmental and process monitoring

Serious problems can arise when powder comes in contact with contaminated air or surfaces. Air used for drying and for conveying powder must be filtered through highly

efficient (>90%) fine filters or high-efficiency particulate air (HEPA) filters. Intact surfaces constitute a minor risk to the product because they are effectively cleaned and sanitised using sophisticated cleaning-in-place (CIP) (refer to Chapter 3 for further information). Nevertheless, where surface damage has occurred, such as cracks in a spray dryer wall, a reservoir of bacteria may develop within the material insulating the spray dryer. Such reservoirs of bacteria are resistant to normal cleaning and disinfection procedures, and can harbour pathogenic or spoilage micro-organisms.

Prevention of powder recontamination involves careful separation of raw from heated product, tight control of environmental hazards and scrupulous attention to cleaning and disinfection of surfaces that come into contact with the dried milk. Furthermore, the dryer will often have its own CIP satellite station to prevent contamination from the non-pasteurised side of the process.

It is apparent that limited information on the microbiological status of a spray drying plant can be deduced from the examination of the quality of the powder alone. Multi-point sampling is more effective, especially if the bacterial load of (a) the raw milk, (b) the stored milk from the balance tank of the heat exchanger, (c) the concentrate *ex* evaporator, (d) the powder *ex* primary cyclone and (e) the packed product are monitored. It is prudent to include routine swabs from drains, walls and/or floors in the monitoring operation because these can be valuable indicators of potential hazards.

5.4 Functionality and certain technical aspects

5.4.1 *Heat treatment*

The temperature of the milk concentrate obtained from the last pre-heater in multi-effect evaporators is lower than the boiling temperature in the first effect. Additional preheating is, therefore, necessary to obtain the required 2–3°C above the boiling temperature of the first effect. A separate pre-heater, which is heated by live steam – usually via a thermo-compressor, is then used.

The heating of the milk can be carried out in different ways: (a) indirect heating in plate, spiral or straight-tube heat exchangers or (b) direct steam injections into the milk or milk into a steam atmosphere.

Indirect heating systems

These heaters – the plate, tube or spiral-tube type – are working as ordinary heat exchangers. If temperatures up to 110°C are wanted, it is recommended to use two heaters, where one is in operation while the other one is being cleaned (Fig. 5.2). One of the advantages of the indirect heating is that the product will not be mixed with the condensing steam, neither will the product be diluted. However, the disadvantage of the indirect heating is that it takes a long time for the product to be heated in the interval from 80°C to 110°C resulting in a concentrate with high viscosity. This is because the whey proteins, when unfolded, will denature and interact with each other before forming a complex with the κ-casein. For improved efficiencies, one or more regeneration systems can be incorporated in the heating equipment (Fig. 5.3).

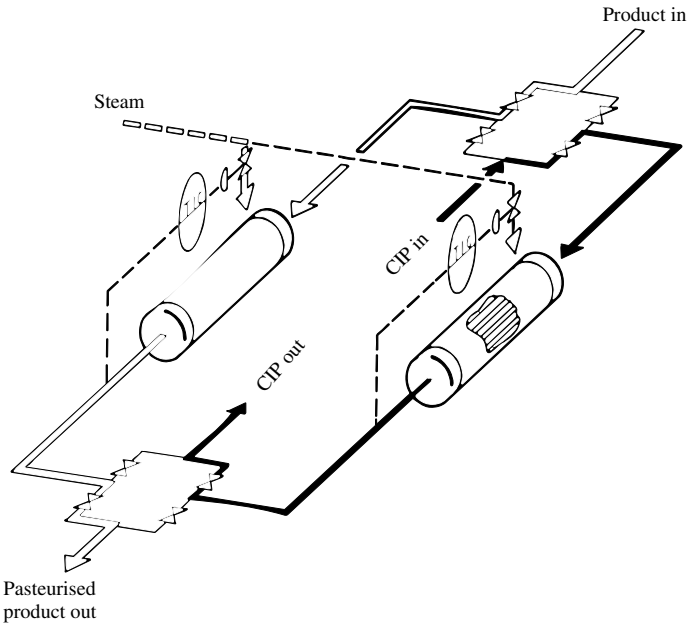


Fig. 5.2 Switchable indirect heat exchangers.

Direct heating methods

This is done in two different ways. The first is by direct steam injection, where the live steam is mixed into the milk using a tangential swirl heater (TSH) (Fig. 5.4). The TSH offers a controlled and short residence time with no mechanical impact, even at temperatures $>120^{\circ}$C. It can operate for 20 h or more without intermediate cleaning. The second is where milk concentrate is sprayed into a steam atmosphere (infusion) at a sufficient pressure. The steam must be of a good quality, that is, it is fit for use in products for human consumption. Culinary steam boilers, where milk condensate is heated up in an indirect coil-type heater by means of live steam, can be used. The advantage of direct heating of the milk concentrate is the short time it takes to reach the desired temperature. This means that both unfolded whey proteins and κ-casein are available, and they can denature/interact with each other, resulting in a milk concentrate of low viscosity. Furthermore, the direct heating will have a less pronounced effect on the denaturation of the whey proteins at the same heating temperature/time. A typical example is as follows.

Degree of whey protein denaturation	Thiamin loss (%)	(g 100 g^{-1})
Direct heating system	35	0.5–0.8
Indirect heating system	65	1.4–4.4

As for the indirect heating system, regenerative flash chambers are used if high temperatures are needed. The temperature of the milk concentrate will drop due to the evaporation in the flash vessel and the resulting vapours are then used for preheating prior to the plate heat exchanger. The regenerative flash chamber can be either indirect as shown in

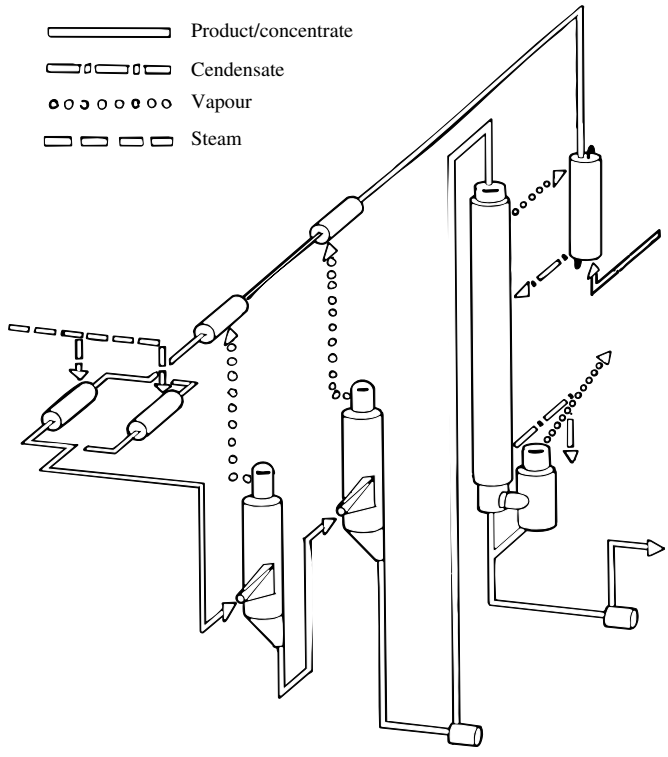


Fig. 5.3 Indirect spiral-tube heat exchanger working in series that is heated by steam and flash vapour.

Figure 5.3 or direct contact as shown in Figure 5.4. The direct contact regenerative system is preferable, as there is no heat contact surface, where deposits (biofilm) can develop.

The holding of the milk concentrate is practically always done in holding tubes of specific length and diameter to give the desired holding time. Holding vats, the so-called 'hot well', have been used, if holding times as long as 30 min are required. It is difficult, however, to control the exact time because some of the product might pass through in 5 min, whereas some would take longer.

The heating temperature will, of course, have a direct influence on the total steam consumption. For the same heating temperature, the direct heating system will result in a higher steam consumption compared with the indirect heating method due to the need to evaporate the extra water formed by the condensation. However, the additional steam used is – after flashing off – utilised as a heating medium in the subsequent calandrias and some of the applied energy is recovered.

The primary purposes of heat treatment in an evaporator are to achieve the following aspects in the final powder.

5.4.2 *Whey protein denaturation*

Heat classified SMP is often produced according to a fixed degree of denaturation of the whey proteins and is classified according to the whey protein nitrogen index (WPNI) that

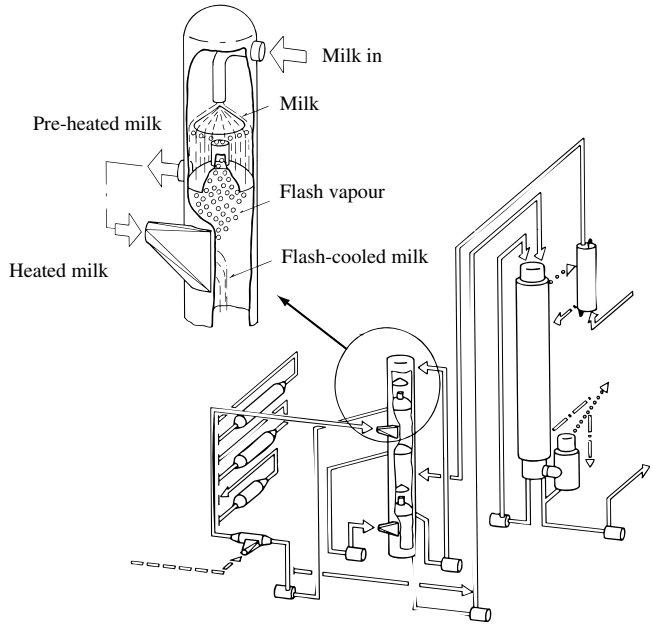


Fig. 5.4 Direct heating system with direct contact flash regenerative system.

expresses the content of un-denatured whey proteins (mg WPNI g^{-1} powder). Different temperature and time combinations have an influence on the WPNI (Fig. 5.5).

Heat classification of SMP by casein number (CN) – Seasonal changes in milk protein content render the heat classification of SMP using WPNI imprecise. An alternative method is based on the *casein number* (CN), that is, the proportion of total nitrogen that is precipitated at pH 4.7 (Sweetsur, 1976). In contrast to the WPNI, CN is largely independent of protein content.

The CN of good-quality raw, that is unheated, milk lies in the range of 80–82, and lower values indicate partial opening of the tight junctions of the secretory cells in the mammary gland allowing degradation of casein by plasmin (Sorensen *et al*., 2008). Values of CN in excess of 82 are indicative of whey protein denaturation, and fully denatured milk has a CN typically in excess of 92. Practical applications of CN have been reported by Sweetsur (1976) for predicting the stability of instant SMP when added to hot coffee and by Muir *et al*. (1991) for optimisation of SMP for use in white sauce.

Keeping the quality of WMP – When producing WMP, one problem is the shelf-life as the fat easily becomes oxidised if the powder is not packed using an inert gas. As most of WMP is shipped in bags, it is not possible to protect the powder effectively, and antioxidants are in most cases not permitted. However, by direct heating the milk prior to the evaporation to 90–95°C for 0.5–1 min, some natural antioxidants will be formed, such as the sulphydryl (–SH) groups, originating from the amino acids (e.g. cystine, cysteine and methionine), which are liberated and will act as antioxidants. Higher temperatures will form more – SH groups, but they will react with casein, and are not to be found in free form (Fig. 5.6). The free – SH groups will at the same time give the milk a cooked flavour, which is liked by many consumers.

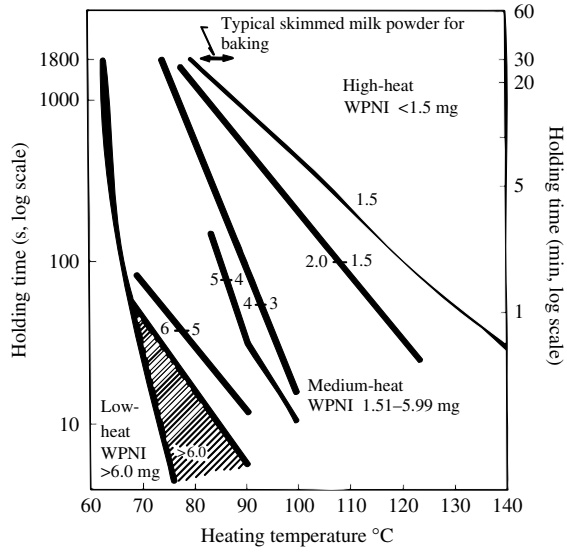


Fig. 5.5 The amount of whey protein nitrogen index (WPNI) in powder (mg g^{-1}) as a function of the heating intensity of milk, a relation between temperature and time.

High-heat heat stable milk powder is used for reconstitution for making evaporated and/or sterilised milk. After reconstitution to 25–27 g total solids 100 g^{-1}, the product has to be sterilised using temperatures of 120°C or higher for 20 min. Furthermore, the use of dried milk for recombination into in-can sterilised milk requires to withstand severe heat treatment equivalent to a lethality index (Fo) of 5, that is 5 min at 120°C. The heat stability

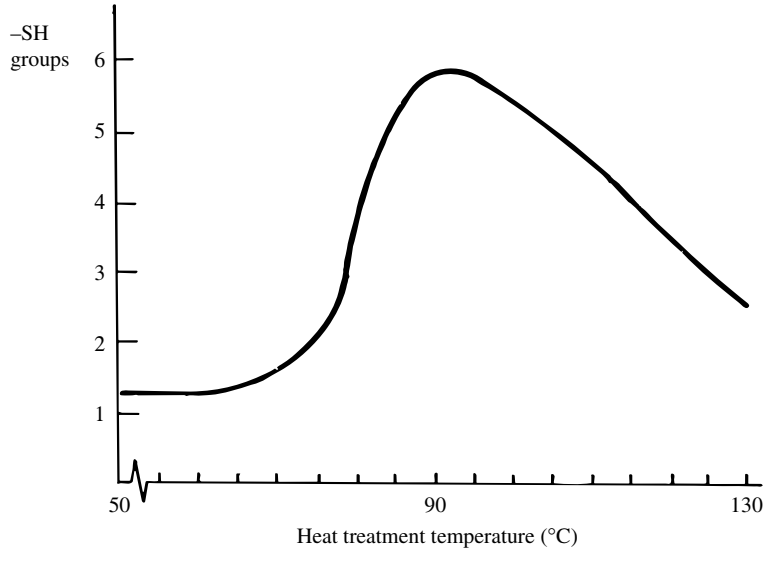


Fig. 5.6 Development of free – SH groups as a function of heat treatment temperature of the milk.

of the recombined product is controlled by the heating temperature–time combination prior to the evaporation and drying. A direct contact heating system gives a better result, and some heating examples where the holding time is 2–4 min are tabulated below.

Heating system	Temperature range (°C)
Indirect	60–80
Direct	80–110[a]
Direct	110–125
Holding time	2–4 min

[a]In the heating interval from 80 to 110°C, a very fast heating is important to avoid the interaction between the whey proteins, in order to produce low viscous products with good heat stability.

Instant quality of WMP – To produce instant WMP with good reconstitution properties in cold water and at the same time with a good 'coffee stability', that is, no coagulation should take place when the powder is added to hot coffee as a 'whitener'. It is recommended to use a temperature–time combination to achieve a WPNI of >3.5 mg g^{-1}, which corresponds to ~45% denaturation of β-lactoglobulin.

5.4.3 *Agglomeration and instantisation*

Agglomeration has a significant influence on the physical properties of the milk powder because the bigger particles will make the powder more free flowing. This is often in demand for powders for use in retail sale as well as in the industry. The agglomeration will positively affect the dissolution properties of the powder. The main disadvantage is a lower bulk density, which means higher logistical costs inclusive of packaging. Both SMP and WMP are, therefore, produced in non-agglomerated, agglomerated and instant versions according to market demands Agglomeration means getting smaller particles to adhere to each other to form a powder consisting of bigger conglomerates/agglomerates, which are essential for an easy reconstitution in water.

During the spray drying process, the aim is to produce particles with a big surface–mass ratio, that is, small particles. The reconstitution in water of a powder consisting of small particles is difficult and requires intensive mixing in order to disperse the powder before it is totally dissolved. Bigger particles exhibit a better dispersion, but the solubility is negatively affected during the drying operation. By agglomeration both a good dispersion and a complete solution can be obtained.

In spray drying, there are two methods of agglomeration: first, the spontaneous and second, the forced; both methods of agglomeration are in a primary and secondary form (see Table 5.6).

The spontaneous primary agglomeration is a result of a random unprovoked collision of particles in a single atomiser cloud due to particles of different diameter having different deceleration paths. It takes place in nozzle as well as rotary atomisers (Fig. 5.7).

The forced primary agglomeration is a controllable means for production of an agglomerated product with certain properties. For example, collision of particles from two or more

Table 5.6 Definition of different agglomeration processes.

Type	Definition	Examples
Spontaneous primary	Random, unprovoked collision of primary spray particles	All atomisation devices
Forced primary	Intended collision between primary spray particles from different atomisation devices	Collision of sprays from different nozzles
Spontaneous secondary	Random, unprovoked collision of primary spray particles and fines	Multi-stage or integrated filter dryers
Forced secondary	Intended collision between primary spray particles and fines returned to the atomisation zone	Normal type when fines return is applied

atomisation clouds, typically in a multi-nozzle unit, where the sprays from the individual nozzles are forced into each other (Fig. 5.8).

The spontaneous secondary agglomeration is a result of a venturi effect at the drying air inlet to the chamber, whereby dry single particles are sucked into the wet atomiser cloud. Moist particles collide with airborne dry particles contained in the exhaust air on its counter-current way out of the multi-stage dryer (MSD) with exhaust air outlet through the drying chamber ceiling (Fig. 5.9).

The forced secondary agglomeration is a controllable means of agglomeration by returning the fines to the atomiser cloud via the fines return. The spontaneous agglomeration, which will always exist, is enforced by the agglomeration applied by returning the fines to the atomiser cloud. By definition, fines are the cyclone or bag filter fractions, and consist of the smallest particles, which are returned to the process. The small dry particles are introduced into the dryer near the atomising device, where they will meet and

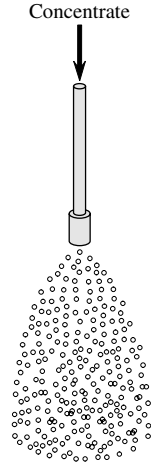


Fig. 5.7 Spontaneous primary agglomeration.

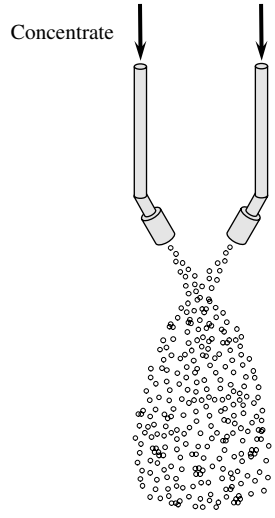


Fig. 5.8 Forced primary agglomeration.

collide with atomised wet droplets, thus forming agglomerates consisting of many particles stuck together having a size of 100–500 μm, depending on the parameters selected (Fig. 5.10).

Combination of agglomeration methods

Due to the special air flow pattern in a MSD, a considerable *spontaneous secondary* agglomeration takes place. For the production of high-quality instant WMP and SMP, this

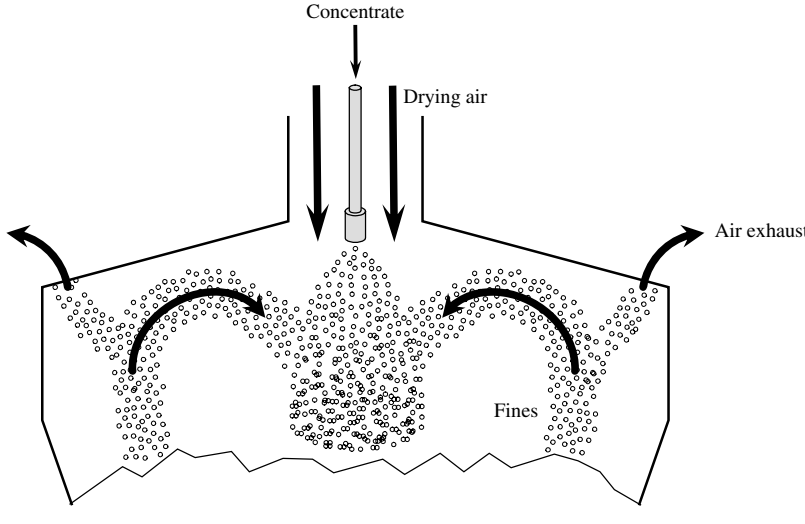


Fig. 5.9 Spontaneous secondary agglomeration.

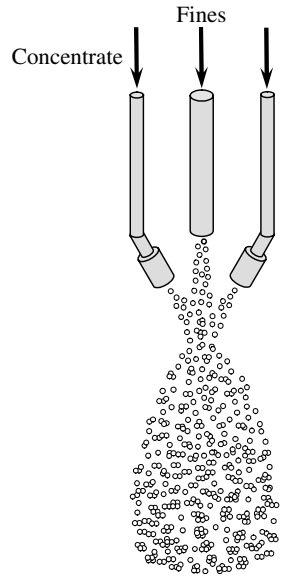


Fig. 5.10 Forced secondary agglomeration.

spontaneous agglomeration suffices, and the fines are simply returned to the integrated fluid bed from where they will get airborne again and reach the atomising zone again. However, the agglomeration may be further enhanced by *forced primary* agglomeration (collision of sprays overlapping each other from different nozzles in a multi-nozzle atomisation unit) and/or by returning the fines to the atomisation zone (*forced secondary* agglomeration). Further flexibility can be gained by designing the atomisation unit in a way that allows the distance between the single nozzles or between the nozzles and the fines return tube to be altered.

Fines return systems

Depending on the atomisation device the fines return is designed in different ways.

First, for rotary atomisation, the aim is to bring the fines as close as possible to the atomiser wheel. This can be done from below (Fig. 5.11) via a pressure conveying system using a 7.5–10 cm diameter dosing pipe with a fines distributor at the end inside the drying chamber. However, deposits are easily formed on this pipe if the air disperser is not adjusted to avoid it. This adjustment is, however, not necessarily optimal from the drying point of view. In modern dryers, fines are introduced from above through the air disperser (e.g. fines return air disperser – FRAD) via four fines pipes situated just above the atomiser cloud. Deflector plates at the end of each fines pipe ensure a correct introduction and distribution of the fines (Fig. 5.12).

Second, for nozzle atomisation, the fines return is an integral part of the nozzle unit, with the fines duct in the centre surrounded by nozzles at the periphery. The fines are introduced tangentially into the fines distribution duct or through a centre pipe (Fig. 5.13).

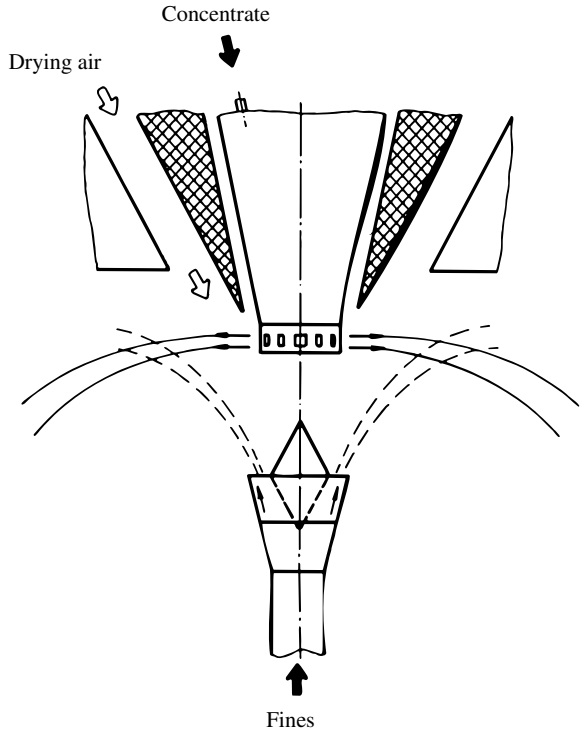


Fig. 5.11 Fines return rotary atomiser – 'old type'.

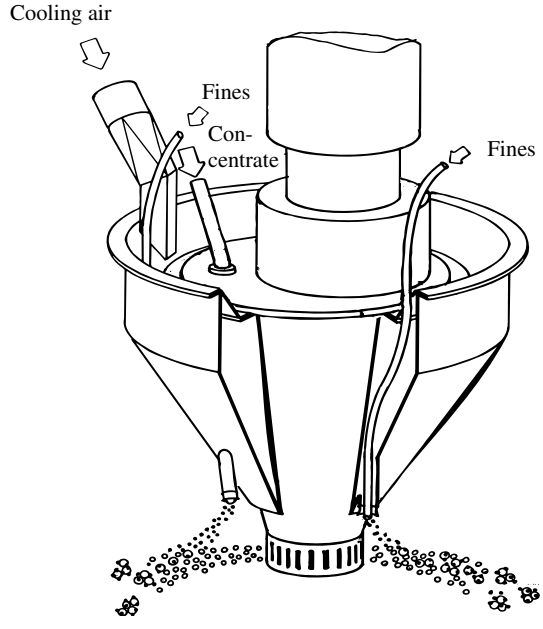


Fig. 5.12 Forced secondary fines return for rotary atomiser FRAD. Note: FRAD = fines return air disperser.

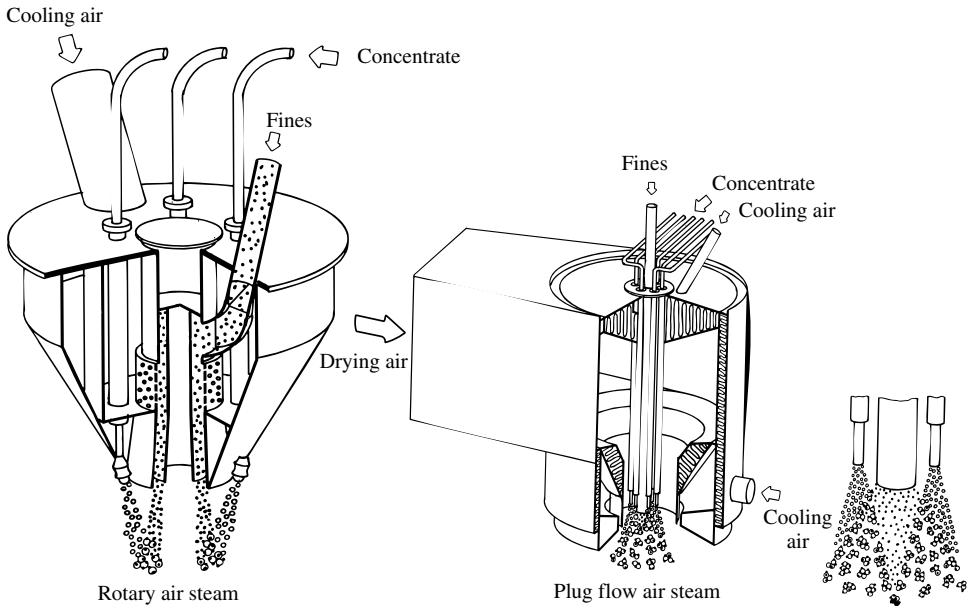


Fig. 5.13 Forced secondary fines return for nozzle atomiser.

The nozzles can be welded to the nozzle rod at a certain angle, so that by turning the nozzle rod around its axis the collision point can be altered. Furthermore, the distance between the single nozzle and the centre pipe can often be adjusted.

Sources of fines

First, separation is the process separating the part of fines entrained in the main drying air leaving the drying chamber. The efficiency of separation is determined by the air flow pattern and air velocities in the drying chamber; it is closely related to the chamber design and can only be marginally affected at normal running conditions, for example by air disperser adjustments and variations in drying air rates. The agglomerated powder leaves the chamber at the base or from the integrated fluid bed and enters the Vibro-Fluidizer. During the passage down the wall of the chamber cone, some stabilisation of the already induced agglomeration takes place. In the static fluid bed and/or the Vibro-Fluidizer, the powder is met by a warm airstream evaporating the excess moisture content, as was the case in the two-stage drying process.

Second, attrition is defined as the partial breakdown of agglomerates in the fluid bed dryer, or powder conveying systems resulting in the creation of either fines and smaller agglomerates (abrasion), or of a number of smaller-sized particles (fragmentation). This often-overlooked phenomenon is the result of mechanical motion between the agglomerate and another body, which may be the walls of the fluid bed dryer or another particle. The most likely cause of attrition in fluid bed dryers is particle–particle interaction, as interparticle impact velocities can be very high, caused by high air jet velocities out of the holes in the perforated plate that forms the bottom of the fluid bed. Factors affecting

the extent of attrition are the jet velocity, determined by the pressure difference across the perforated plate, the fluidisation velocity and the actual design of the perforated plate.

Third, classification is defined as the separation of fines in fluid beds. The efficiency of classification is mainly determined by the fluidisation air velocity, but fluid bed dryer design features are also of importance in ensuring that separated fines are kept airborne and entrained in the exhaust fluid bed air.

After the final drying, the powder enters the cooling section where the powder is cooled by means of air at ambient temperature followed by cooled, dehumidified air. The powder is finally passed over a sifter where any over-sized particles are removed. It is also possible to install a sifter with two screens, thus removing any remaining particles/agglomerates of small diameter. Together with the fines, this fraction may be returned to the atomising device thus producing a powder with a well-defined agglomerate size distribution. The fines removal in the fluid beds is, however, regarded as sufficient from a product point of view and plants with the above-mentioned sifter are only used when particular product specifications have to be met. Figure 5.14 shows a plant set-up featuring milk reception and processing equipment, evaporator and spray dryer.

Agglomerate structure and powder properties

Depending on the design and adjustment of the fines return system – particularly the location of the introduction of the fines in relation to the atomisation device – different agglomerate structures result, which influence certain powder properties, such as bulk density, mechanical stability, dispersibility and slowly dispersible particles (SDP). The relation between agglomerate structure and certain powder properties is illustrated in Figure 5.15.

If the fines are introduced close to the atomising device, the moisture content of the primary spray particles is high and thereby their plasticity and stickiness, and the fine particles may penetrate primary particles or be completely covered by concentrate (Fig. 5.16). Such agglomerates have been termed *onion structured*.

When collision takes place at a progressively longer distance away from the atomising device, less compact agglomerate structures are obtained. Such structures have been termed *raspberry* structures and *grape* structures in decreasing order of compactness.

Onion-structured agglomerates are characterised by a high mechanical stability and a high bulk density, but they will often appear as SDP after reconstitution. They may also be collected during the different dispersibility tests in use and jeopardise the general quality evaluation of the product.

With progressively looser agglomerate structures, the bulk density and mechanical stability decrease gradually, and the overall instant properties improve. However, if a *loose grape* structure is eventually obtained, the mechanical stability may be so low that the powder becomes very susceptible to attrition, resulting in deteriorated instant properties. A *compact grape* structure (Fig. 5.17) is regarded as the ideal compromise, where the powder has simultaneously good instant properties and sufficient mechanical strength to enable necessary transport and packaging.

The agglomeration process is improved by the following:

- High solids content in the concentrate.
- Bigger quantity of fines returned to the atomising device.

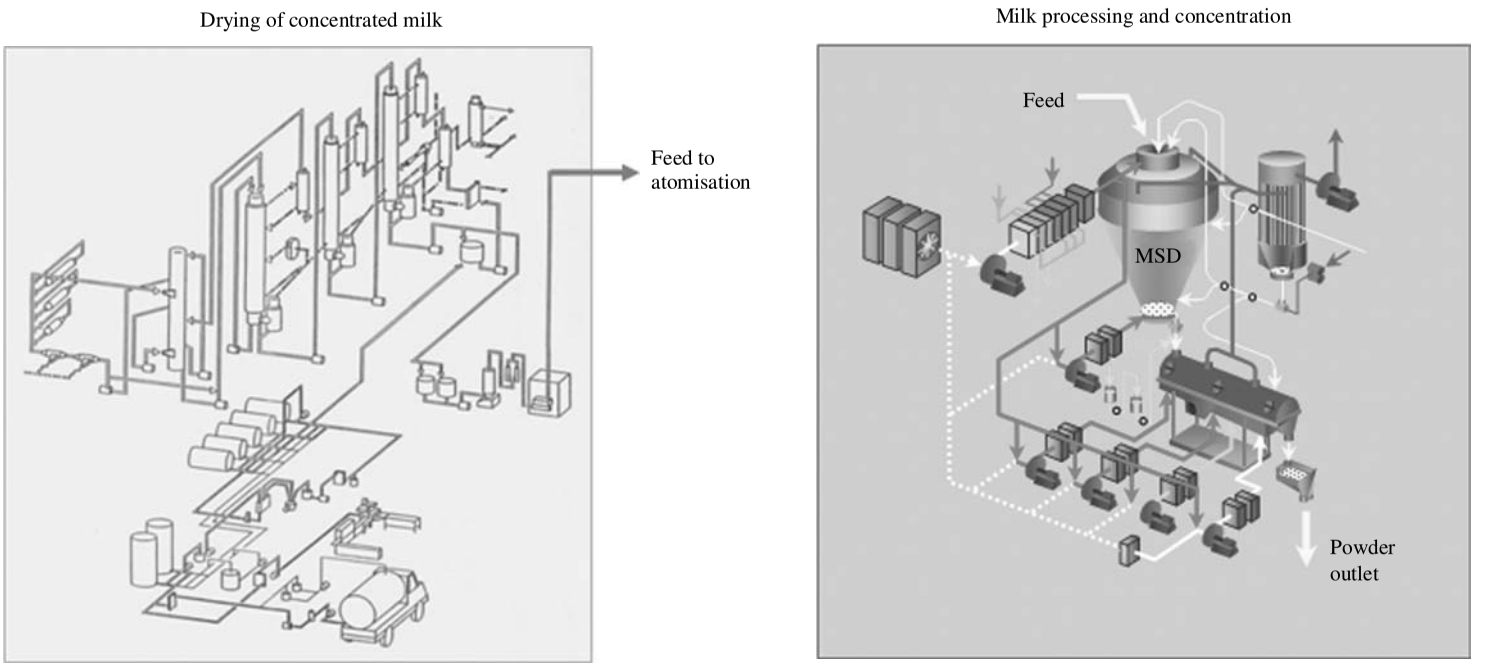


Fig. 5.14 Complete multi-stage dryer (MSD) for the production of agglomerated powders.

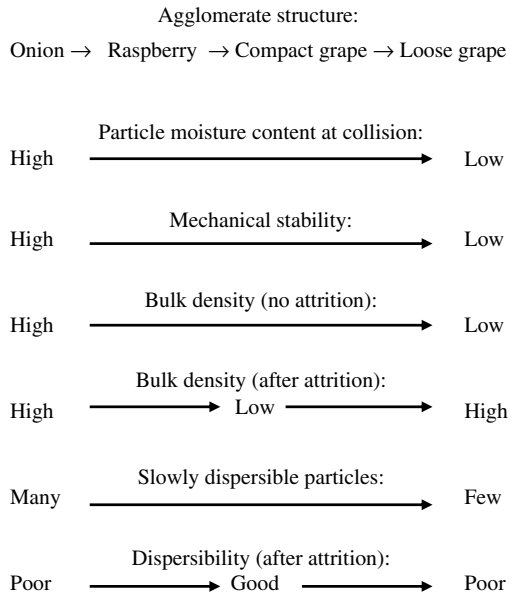


Fig. 5.15 Agglomerate structure/powder properties relationship.

- Introduction of fines closer to the atomising device.
- Shorter distance from the nozzle to the fluidised layer in a static fluid bed dryer.
- Higher moisture content from the primary drying stage.
- Bigger primary particles.
- Lower heat-treatment temperature of the milk prior to evaporation.

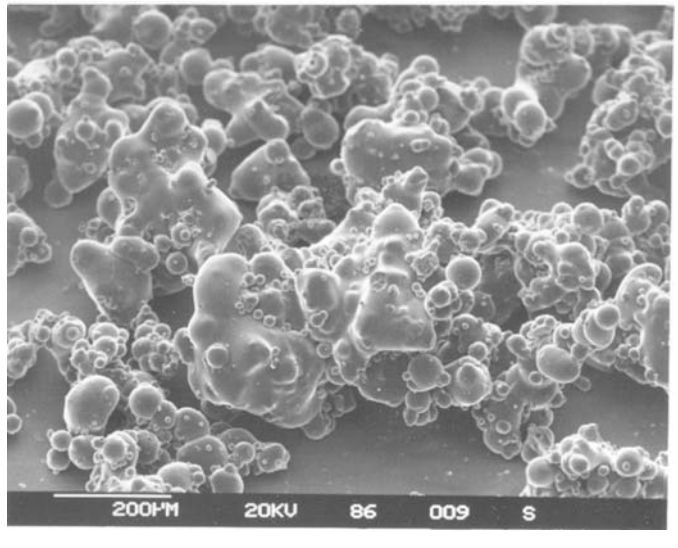


Fig. 5.16 Onion-structured agglomerate.

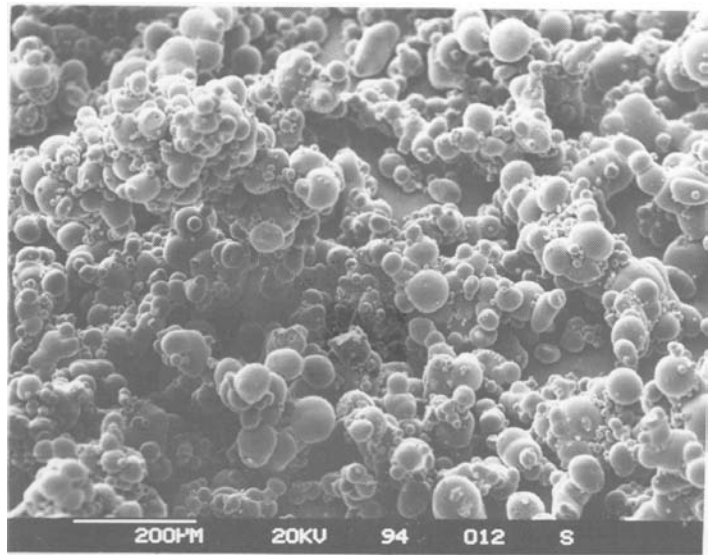


Fig. 5.17 Compact *grape* structured agglomerate.

When leaving the sifter, the powder should not be exposed to strong mechanical conveying, neither by means of air nor by fast moving mechanical screws. However, today's lenient vacuum–low-speed air systems are used without too much damage to the agglomerates. The best approach, however, is to install the plant so high that filling into bags or tote bins is possible by gravity.

5.5 Specific processes

5.5.1 *Ordinary milk powders*

Non-agglomerated milk is usually referred to as ordinary milk powder. By far the most common ones are powders of skimmed milk and whole milk. The applied drying process may be either one-stage or two-stage drying.

Prior to the evaporation, the milk is clarified in a separator to remove dirt and other undesirable materials (see Section 5.3) that may be present in milk and, if SMP is produced, the fat is also removed. If the protein content is high, it may be standardised to 3.4 g 100 g^{-1} by the addition of lactose or UF milk permeate. In the evaporator, the milk is heated at 70–100°C, depending on the powder specification to be complied with. The milk is then condensed to 48–52 g total solids 100 g^{-1}, depending on the milk quality and the drying system used.

Between the spray dryer feed tank and the atomiser (wheel or nozzles), the milk concentrate is normally preheated to 60–70°C, depending on the feed characteristics and atomiser type, and afterwards filtered to avoid any problems with particles in the atomiser. The pre-heating keeps the viscosity of the concentrate low and brings 3–5% extra evaporation capacity to the spray dryer. If nozzle atomisation is used, the concentrate should always

Table 5.7 Processing conditions in different types of dryers and milks.

Type of dryer	One-stage dryer				Two-stage dryer			
Type of atomiser	Nozzles		Wheel		Nozzles		Wheel	
Type of milk	Skimmed	Whole	Skimmed	Whole	Skimmed	Whole	Skimmed	Whole
Drying temperature (°C)	180	180	200	180	210	200	210	200
Solids content (g 100 g^{-1})	45	42	48	48	47	47	48	48

be preheated. Whole milk concentrate is normally homogenised before atomisation. When using nozzle atomisation, the homogeniser and the high-pressure pump are typically built as one unit.

The spray dryer is operated at temperatures of 180–200°C for WMP and 180–230°C for SMP. In one-stage drying, the spray dryer is operated in such a way that the powder has reached the final moisture content when leaving the drying chamber. In the two-stage drying process, the primary drying is carried out to about 2–10 g 100 g^{-1} above the final moisture content. The excess moisture is evaporated in a static fluid bed and/or a Vibro-Fluidizer, and the cooling is done in a pneumatic cooling system, or in a Vibro-Fluidizer. Typical drying conditions are shown in Table 5.7 and the powders can be characterised by the following:

- Consist single particles
- High bulk density
- Dusty if it is skim milk powder (SMP)
- Non-instant

5.5.2 *Instant milk powders*

During the last few decades, the market for powders that are instantly soluble in cold water has grown due to growing market demands in countries, where milk powder is sold in retail for home consumption. Ordinary non-agglomerated powders tend to lump when mixed with water and, if strong mechanical stirring is not applied, may result in an inhomogeneous mixture, which is not attractive to the consumer. It has for a long time been known that an agglomerated powder, that is, a powder where the single particles have formed bigger granulates or agglomerates, possesses completely different properties when mixed with water than is the case with ordinary powder.

The plant is operated so that the powder leaves the primary drying stage with 2–10 g 100 g^{-1} higher in moisture than wanted in the final product. The cyclone–bag filter fraction is returned to the atomising device, where the dry fine particles will collide with the primary particles, thus forming agglomerates. The powder leaving the chamber is warm, moist and consists of stable agglomerates. Consequently, the integrated fluid beds and/or external fluid beds are dried gently (Vibro-Fluidizer) so that the agglomerated product structures are maintained. The cooling should always be done in a fluid bed.

The powder obtained by this process can be characterised by the following:

- Agglomerated product structure
- Non-dusty
- Lower bulk density than for non-agglomerated powder
- Good flowability

The decreased drying air outlet temperature and, consequently, lower product temperature will result in the following:

- Improved solubility because of less thermal damage
- Low content of occluded air, because in the critical stage of the drying, with a water content of 30–10 g 100 g^{-1}, the blowing up of the particles is avoided.

If the above process is limited to SMP, it will, by the mere agglomeration, obtain a product with good instant properties.

Besides the manufacturing equipment, it is necessary to have control of the quality of the final powder, especially regarding the instant properties. It is not surprising that the first method, which was introduced to distinguish instant products, was based on determining the property called *wettability*, as this is the most conspicuous feature of an instant product compared with an ordinary product. It was measured as the time necessary for wetting of a given amount of powder, that is, the time from the first contact with water until the powder had completely passed the water surface.

It has later been realised that wetting is only the first step of a rather complicated reconstitution process, and that this process consists of a number of phenomena, which can be described as follows.

Reconstitution phenomenon	Milk powder property
Wetting	Wettability
Dispersing	Dispersibility
Dissolving	Solubility

Splitting the reconstitution process into the above steps helps to understand the process, for instance, to be able to find the reasons for the lack of instant property. On the other hand, one must be aware that there is no sharp borderline between these individual reconstitution steps, and it is impossible to determine the individual properties independently of the others. For a good evaluation of product properties all these qualifications have to be considered.

Instant SMP – In order to obtain SMP with good instant properties, agglomeration plays the most important part. Wetting and dispersing of a single SMP particle in water is no problem, as the particle will dissolve quickly. If bulk non-agglomerated SMP is poured into water, the first powder to come into contact with the water will dissolve, forming a concentrate film impervious to water, thus stopping any further wetting. The resulting mixture will be difficult to disperse leaving wetted lumps with dry powder inside on the surface of the water.

In order to avoid too quick a wetting of the particles, these are agglomerated thus reducing the specific surface. The specific surface can also be reduced by bigger primary particles, but with the risk of a high insolubility index. Furthermore, the instant properties, especially the wettability, are improved; if the agglomerates are so compact that the moisture absorption and dissolving process are prolonged enabling a dispersion of the powder agglomerates in the water, after which the final dissolution can take place. Needless to say, the powder should also have a good insolubility index.

The agglomeration process is improved by the following:

- Reduction of the heat treatment temperature of the milk before evaporation
- Higher solids content in the concentrate
- Bigger primary particles
- Increased recycled amount of fines
- Fines introduction closer to the wheel or nozzle
- Increased moisture content in the powder from the drying chamber

Typical operation conditions for the spray dryer when producing a first-class instant SMP, depending on the type of dryer are as follows:

- Drying temperature between 180°C and 220°C
- Solids content in the concentrate between 48 and 50 g 100 g^{-1}

Instant WMP – In the case of reconstituting WMP, it is required that the water is >40°C, as WMP particles are always covered by a thin layer of fat making the powder repellent in cold water. However, there has in recent decades been an ever-increasing demand for cold water instant WMP. Thus, besides the agglomeration, it is necessary to apply a surface active agent. For this purpose lecithin (originating from soya beans) dissolved in pure butter oil in order to make a liquid, may be used. Lecithin is superior in its functional performance, that is, the achieving of instant properties. The butter oil is chosen also in order to use a natural milk component, as using a vegetable fat, even when it is done in many cases, could be considered a falsification. The amount of lecithin in the total free fat (i.e. original free fat + added butter oil + lecithin) in the final powder may vary from 0.1 to 0.3 and 1 to 2 g 100 g^{-1}. However, variations within these limits result in rather big differences in the desired properties.

One must be aware that a high amount of total free fat together with a high amount of lecithin improve the wettability, but on the other hand, they affect the flowability and may seriously affect the dispersibility. At lecithin levels >0.5 g 100 g^{-1}, it is possible to detect the characteristic soy flavour. The structure of the powder and the degree of agglomeration are of importance too, as poorly agglomerated powders require higher amount of wetting agent than well-agglomerated products.

The processing stages for the production of instant WMP are shown in Figure 5.18. It can be seen that the whole process, that is the manufacture of a basic powder in a plant, has been split into two subsequent process lines, as shown in Figure 5.19, followed by the lecithination and packing line (Fig. 5.20). The splitting of the process line into two is the

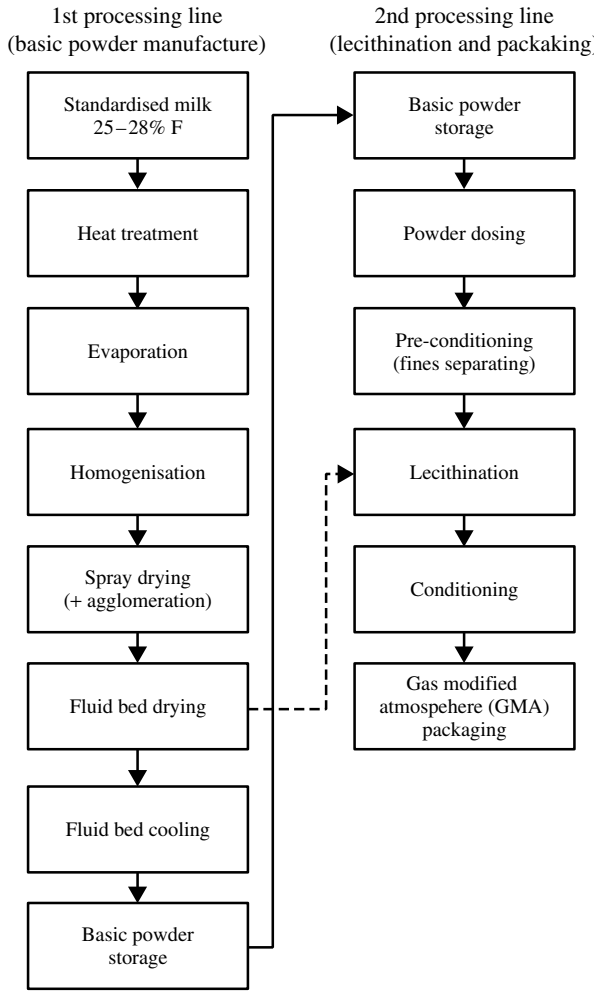


Fig. 5.18 Production lines for instant whole milk powder.

most advantageous way of operation, as will be explained in subsequent sections. In the two-stage process, the basic powder is collected for intermediate storage. It is important to prevent any damage to the powder by mechanical treatment. The intermediate storage is preferably accomplished in tote bins or similar containers of 1–2 m^3 capacity. The basic powder is then transferred from the tote bins into the supply silo and is metered into the first Vibro-Fluidizer by means of a dosing screw. The powder is heated and at the same time, any fines are blown off.

The lecithin-dosing equipment consists of two vessels, dosing pump, powder trap with two-fluid nozzle and control panel. The first vessel serves for the preparation of wetting agent and the second one as supply vessel, from which the wetting agent is metered to the two-fluid nozzle for spraying on to the powder.

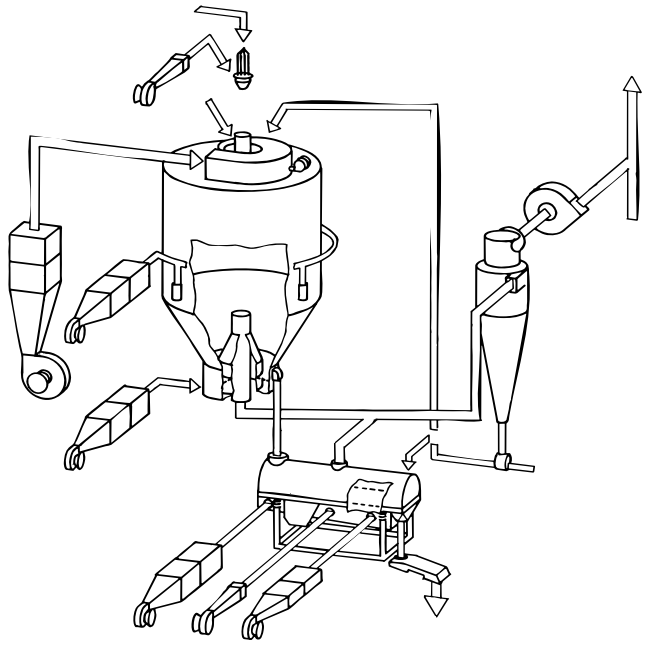


Fig. 5.19 Compact spray dryer with Vibro-Fluidizer as agglomerator/instantiser known as *CDI*.

The flows, temperatures and pressures of the wetting agent and of the atomising air are recorded. Interlocking in the control panel ensures that the flow of powder will stop automatically, if for some reason no lecithin is applied. Consequently, no powder will leave the plant without a proper lecithin coating. The second Vibro-Fluidizer, also supplied with warm air, ensures a gentle but proper mixing of the powder to obtain a uniform distribution of the lecithin mix over the particle surface. Afterwards, the powder leaving the lecithination unit is packed into retail packages. The filling machine is placed preferably directly below the lecithination unit with a hopper for short intermediate storage to avoid any unnecessary transport.

As shown in Figure 5.18 (i.e. the dotted line), the powder production and lecithination can be done in one continuous process. In this case, the powder trap with lecithin nozzle is placed between the integrated fluid bed and the Vibro-Fluidizer. On this flow sheet reception, pre-treatment, standardisation and evaporation equipment are shown. The product quality can be compared with the one achieved by the split process operation described above. However, there are many reasons for preferring the split process operation, as retail packing of milk powder is never a fully continuous operation, since there is always a natural break between the powder production and the packing. During this break, the powder must be stored in bulk for one to several days, preferably in tote bins to avoid damage. For quality reasons it is better to store un-lecithinated powder. Therefore, the lecithination process fits best as a part of the packing line forming one continuous operation.

The intermediate storage of the powder after production makes it possible to analyse the product to classify it, and to calculate the composition and quantity of wetting agent in order to achieve the desired properties.

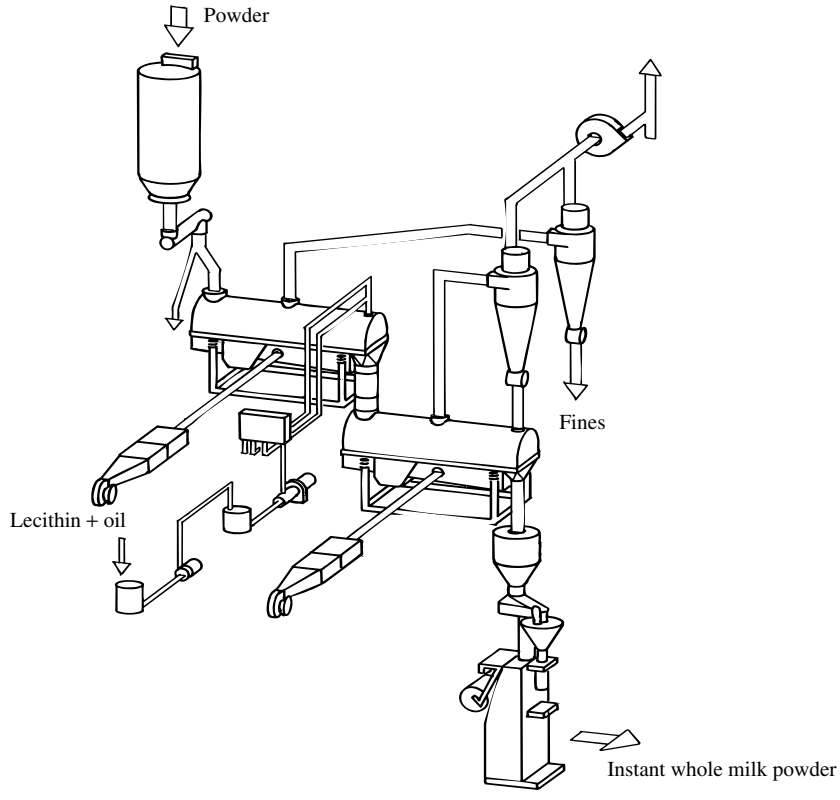


Fig. 5.20 Two-stage lecithination of whole milk powder (WMP).

A further advantage of the split process operation is that fines created during storage and transport can be blown off in the first Vibro-Fluidizer of the lecithination unit. The quality of this rejected fines portion corresponds to ordinary (non-agglomerated) WMP and can be sold as such. The amount is usually less than 5 g 100 g^{-1}.

Today, however, most of the instant WMP is produced in MSD plants equipped with lecithin-dosing equipment, placed between the integrated fluid bed and the Vibro-Fluidizer, in one processing step. The final powder is conveyed to silos by lenient low-speed vacuum conveying systems – being very gentle to the agglomerated product – before packing either in retail packs or in 25 kg bagging lines. The conveying lines are often equipped with pre-gassing by N_2/CO_2 for prolonging the shelf-life of the product. Figure 5.21 shows lecithin-dosing equipment with dosing nozzle situated between two Vibro-Fluidizer.

5.5.3 *Other types of milk powders*

WMP with high free-fat content

The WMP processing attempts to produce a powder with as low a content of free fat as possible in order to prolong the shelf-life of the product. However, the chocolate industry is interested in WMP with as high a free-fat content as possible, as it reduces the viscosity of

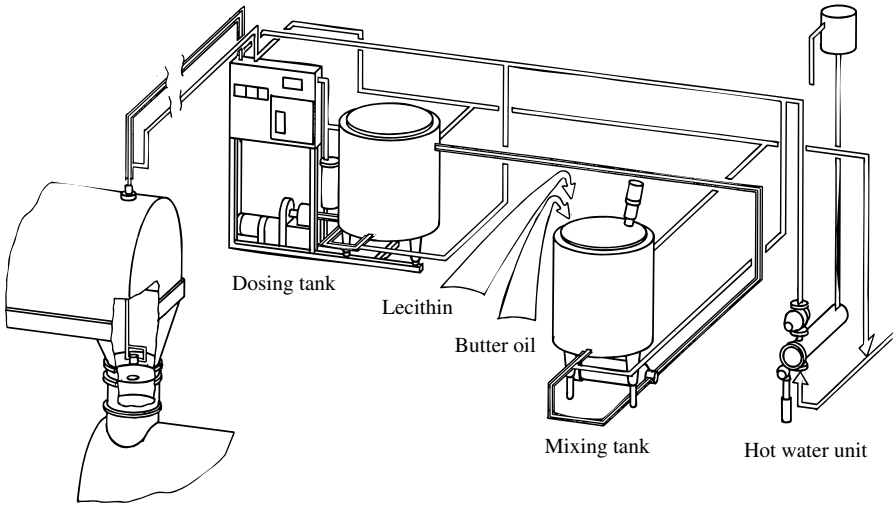


Fig. 5.21 Lecithin-dosing equipment.

the chocolate paste. It is, therefore, possible to use less cocoa butter and thereby save costs. The product is produced by pre-crystallising the lactose in the whole milk concentrate or skimmed milk concentrate, which is then mixed with cream in the correct proportion.

The spray dryer selected for this powder is the MSD type (i.e. integrated fluid bed and air outlet in the top of the drying chamber). This plant offers many advantages, as it is designed for products with high fat content that are difficult to dry. Another advantage of this dryer is the integrated fluid bed, in which the powder is fluidised at a high moisture content. Due to the drying, the lactose becomes supersaturated again, and the crystallisation can proceed further resulting in a high free-fat content; as the lactose crystals 'grow', they destroy the protein membrane protecting the fat globules. Addition of up to 20 g 100 g^{-1} lactose, based on solids, to the milk before evaporation improves the crystallisation (up to 90 g 100 g^{-1} of the total fat).

The high free fat causes more problems in the cyclones due to smearing compared with drying of normal WMP, but as the MSD plant is equipped with a special pre-cyclone or a CIP-able bag filter, the problem is partly solved. The final drying and cooling is done in the Vibro-Fluidizer. It is important that the final powder moisture is below 2 g 100 g^{-1} to maintain the good colour of the chocolate.

Low-heat SMP for cheese production

During the past decade, great efforts have been made to produce SMP that is suitable for cheesemaking, the reason being to counteract seasonal variations in supplies of raw milk for cheese production. However, it also opens the possibility of sending powder to countries with no milk production of their own for making special soft cheese with a short shelf-life, or of producing powder for stock, if prices for cheese are low, with a later reconstitution and cheesemaking in mind because the keeping quality of the powder is better than that of cheese.

Before going into details about the drying technique, it should be mentioned that unless raw milk of top quality with respect to hygiene is available, a suitable powder for making cheese of good quality cannot be obtained.

SMPs are classified according to the WPNI content (mg g^{-1} powder), such as low-, medium- and high-heat powders that contain ≥ 6, > 1.5 but < 6, and ≤ 1.5, respectively. The following are recommended for further reading about critical reviews and predictions of shelf-life of dairy-powder specifications, including an update of standards (Sjollema, 1988; Kjaergaard-Jensen, 1990; Nielsen *et al*., 1997; Stapelfeldt *et al*., 1997; Masters, 2002; Kelly, 2006). As it is the amount of denatured whey proteins we are interested in, and as there is no practical analytical method for the determination of the degree of denaturation, it is necessary to determine the amount of total undenatured whey protein in the raw milk. Seasonal variations and the nature of the whey proteins in the raw milk, however, make it necessary to measure the amount at least once a week. However, only local knowledge about the heat stability of the whey proteins at a particular time of the year makes it possible to choose the correct heat and time combination in order to meet a specific heat classification.

As the amount of whey proteins generally varies from 6 to 13 mg g^{-1} (mainly 8–9 mg g^{-1}), it is not, however, enough just to obtain a WPNI of 6 mg g^{-1}; this could mean up to 7 mg un-denatured whey proteins g^{-1} SMP with the risk of bitter taste in the cheese. Therefore, it is necessary to obtain as high WPNI values in the powder as possible (meaning low temperatures when heating the milk).

As mentioned elsewhere, the requirement to the milk quality is high, but there is a possibility for heating the milk with direct steam injection immediately before entering the first effect, where the milk is flash-cooled to the temperature in the calandria. This way of heating has little or practically no influence on the whey proteins, whereas the effect of killing the bacteria is increased. Special attention should be paid to the content of thermophile spore-forming bacteria, as they will not be destroyed by the 'low' heat treatment temperature. They may even start to develop in the preheating system after 14–16 h, if special attention in the form of design of the evaporator is not taken. But what happens to the whey proteins in the evaporator and the spray dryer? Not very much in the evaporator, and practically nothing in the spray dryer, provided the temperatures are not too high. It is the case that the higher the concentration the better the whey proteins are protected against heat denaturation.

Typical production data are as follows:

- Preheat the milk to 65–70°C – no holding time (possible heat treatment to 90°C with flash cooling to 65–70°C before entering the first calandria, but watch out for the thermophile bacteria and especially their spores).
- Evaporation to 44–46 g total solids 100 g^{-1} (the temperature in first effect is not higher than 65°C) and possible storage at 40°C for 1–2 h).
- Spray drying at 180°C or 200°C.

Single-pass evaporators are recommended and two-stage drying is to be preferred. This provides a more gentle drying, because the particle temperature is much lower, especially during the critical drying phase from 20 to 10 g moisture 100 g^{-1}, which results in a

powder with no protein denaturation. The fines fraction is recycled into the Vibro-Fluidizer, where, together with the chamber fraction, it is finally dried to the wanted moisture content and cooled before bagging off. The gentle drying and after-drying mean that the rate of hydration, that is, the time for complete dissolution when reconstituted, is improved. Besides giving a better product quality, the drying costs are reduced and the evaporation capacity is increased, which is an advantage because of the recommended lower solids from the evaporator. This means that a greater part of the evaporation is transferred to the spray dryer.

5.6 Quality assessment

5.6.1 *Introduction*

Milk powder produced for commercial use must comply with certain microbiological, chemical and physical standards. Obviously, the quality of the final powder is a function of the raw milk quality. Therefore, it is vital for a profitable production to have milk of first-class quality, and to be able to analyse both milk and the powder to know if they meet the requirements, which are very often determined by official export authorities or directed by the governments as national or international legislation. Examples of milk powder specifications are shown in Table 5.8. In this chapter, the most common analytical methods are described, and how to alter the relevant processing parameters in order to change the particular property that fails to meet the standard is discussed.

5.6.2 *Milk*

Specifications

Skimmed milk should be fresh and stored at 5°C (maximum) for 48 h (maximum) after milking, and should not have had any pre-treatment other than (1) separation of cream/desludging by means of a centrifuge and (2) one heat treatment at 72°C (maximum) for 15 s. The milk should be of 'class A' quality and be free from any additives and suspended solids (i.e. filtered 100 μm), with a typical specification shown in Table 5.9.

Whole milk must be fresh and stored at 5°C (maximum) for 48 h (maximum) after milking, and must not have had any pre-treatment other than (a) separation of cream/desludging by means of a (b) standardisation of fat and protein content by separation of cream and/or addition of skimmed milk, milk permeate or lactose and (c) one heat treatment at 72°C (maximum) for 15 s. Similarly to skimmed milk, whole milk should be of 'class A' quality and be free from any additives (apart from fresh cream for standardisation) and suspended solids (filtered 100 μm) (Table 5.9).

Some routine analyses of milk are described in the subsequent text.

Measurement of pH

The pH is a measure of the hydrogen ion (H^+) activity in aqueous solutions. The most exact measure of pH is obtained electrometrically by means of a pH metre. The pH value in normal fresh milk from healthy cows is 6.5–6.7. Values above 6.7 indicate mastitis

Table 5.8 Typical specification of milk powders.

Parameter	Type of powder				
	SMP	Agglomerated SMP	WMP	Agglomerated WMP	Instant WMP
Chemical composition (g 100 g^{-1})					
Moisture[a]	3.7	3.7	3.0	3.0	3.0
Fat	1.0	1.0	26	26	26
Protein	38	38	27	27	27
Lactose	50	50	38	38	38
Ash	7.5	7.5	6.5	6.5	6.5
Physical properties					
Bulk density[b] – tapped × 1250 (g mL^{-1})	0.67–0.70	0.43–0.52	0.58–0.64	0.43–0.50	0.42–0.49
Solubility index (mL)	≤0.1[c]	≤0.2[c]	≤0.2[c]	≤0.2[c]	≤0.2
Scorched particles (disc)	A	A	A	A	A
Free fat (g 100 g^{-1})	–	–	≤2.0	≤2.0	≤2.5
WPNI (mg g^{-1})	1.5–6.0	2.5–4.5	2.5–3.5[d]	2.5–3.5	3.5–4.5
Wettability (s)	–	≤30	–	–	≤15
Dispersibility (%)	–	>98	–	–	>95
Sludge (at 25°C)	–	≤0.1	–	–	≤0.1
Microbiological analysis (cfu g^{-1})					
Total plate count	$\leq 10 \times 10^3$	$\leq 10 \times 10^3$	$\leq 10 \times 10^3$	$\leq 10 \times 10^3$	$\leq 10 \times 10^3$
Coliforms	Absent	Absent	Absent	Absent	Absent
Escherichia coli	Absent	Absent	Absent	Absent	Absent
Staphylococcus aureus	Absent	Absent	Absent	Absent	Absent
Yeasts	<20	<20	<20	<20	<20
Moulds	<20	<20	>20	<20	<20
Salmonella spp. (cfu 100 g^{-1})	Absent	Absent	Absent	Absent	Absent

SMP = skimmed milk powder; WMP = whole milk powder; WPNI = whey protein nitrogen index; cfu = colony-forming units.
[a]Maximum levels.
[b]The bulk density reading(s) will be affected by the degree of heat treatment of the milk, type of atomiser and type of dryer.
[c]These figures will be slightly higher for high-heat powders.
[d]For high-heat powders, the value will be <1.5.

infections, whereas values below 6.5 indicate the presence of colostrum or deterioration of certain components due to microbial activity. Milk with deviating pH should be rejected from milk powder production, as the heat stability most probably will be inferior.

Table 5.9 Specifications of 'class A' raw milk (skimmed and whole)[a].

Total plate count (cfu mL^{-1})	25×10^4 (maximum)
Thermophilic count (cfu mL^{-1})	100 (maximum)
Thermophilic spore count (cfu mL^{-1})	10 (maximum)
Sediment count (mg 100 g^{-1})	0.5
pH	6.6–6.8
Titratable acidity (mL lactic acid 100 mL^{-1})[b]	0.15
True lactic acid (mg 100 g^{-1})	10
Protein (g 100 g^{-1} dry matter)	37–38

cfu = colony-forming units.
[a]The only components, which are different, are (a) the maximum fat content in skimmed milk is 0.05 g 100 g^{-1}, whilst in whole milk is 28 g fat 100 g^{-1} dry (b) the total solids content of these milks should be ~9.0 and 12.0 g 100 g^{-1}, respectively and the maximum non-condensable gases in skimmed milk is 0.02 g 100 g^{-1} and in whole milk is 0.01 g 100 g^{-1}.
[b]IDF (2005a).

Titratable acidity

The acidity in milk is measured, for example by titration with a 0.1 N NaOH solution, which indicates that the consumption of NaOH is necessary to shift the pH value from 6.5–6.7 (corresponding to fresh milk) to a pH value of 8.2–8.4 (phenolphthalein). Lactic acid is an organic acid with one carboxylic acid, CH_3-CHOH-COOH, having a molecular weight of 90. One millilitre of 0.1 N NaOH corresponds to

$$\frac{90 \times 0.1}{1000} = 0.009 \text{ g of lactic acid.}$$

If the titration requires, for example, 14.5 mL of 0.1 N NaOH, the result is often expressed as:

$$14.5 \times 0.009 = 0.13 \text{ mL lactic acid } 100 \text{ mL}^{-1}$$

However, fresh milk contains practically no lactic acid, and the consumption of NaOH is used to change the pH value of the following components (mL lactic acid 100 mL^{-1}): carbon dioxide equivalent to 0.01, citrates 0.01, casein 0.07, albumin/globulin 0.01, phosphates 0.03 and titratable acidity equivalent to 0.13.

The determination of 'acidity' in fresh milk by means of titration is, therefore, more a measure of the buffer action of milk than anything else. Thus, it is necessary to talk about the developed acidity, which is the result of bacterial activity, producing lactic acid during milk collection, transportation and processing. The developed acidity will, needless to say, be more pronounced if the milk is not cooled.

In order to avoid uncertainties about the degree of titratable acidity or developed acidity, it is necessary to use direct determination of lactic acid during the processing. This is done in order to find whether any of the installed equipment is responsible for developing acidity

that expresses activity of not only the bacteria alive after heating the milk but also previous activity of bacteria killed during the heat treatment.

5.6.3 *Concentrate*

Content of air

Content of air in the concentrate should be avoided by any means, as it upsets the whole concentrate pre-treatment and drying. It is possible to measure the content of air in the concentrate by using the equipment shown in Figure 5.22. The apparatus is set under vacuum with a glass stopper; valves B, C and D are closed and A is open. The system is filled up with NaCl solution by sucking it into the apparatus with a vacuum pump, leaving the 100-mL concentrate container empty and 1–2 mL of air in the graduated glass tube on the 'air side'. The concentrate to be tested should now be filled into the 100-mL container, which is then closed. The amount of air (which should be 1–2 mL) in the 'air container'

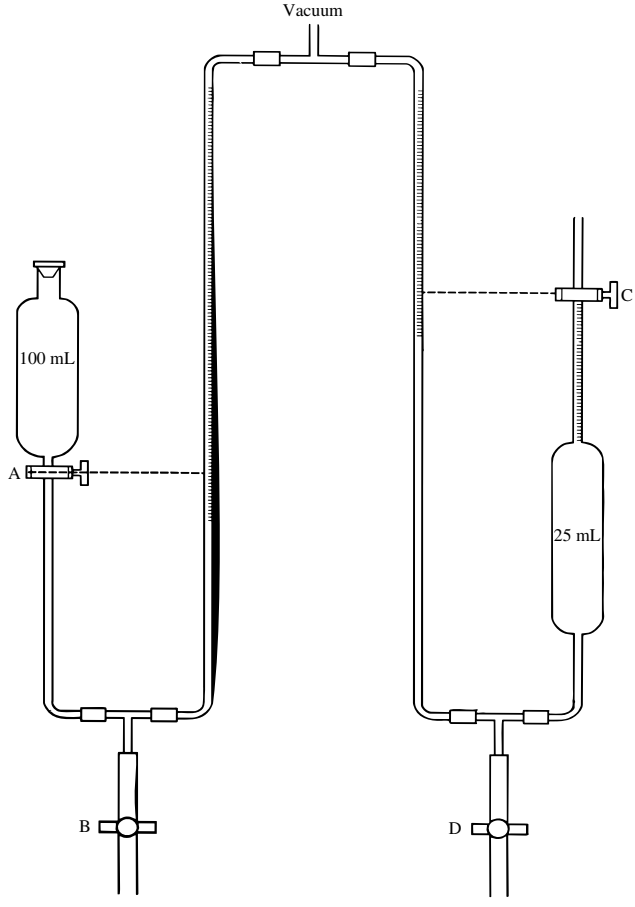


Fig. 5.22 Laboratory equipment for measuring the amount of air in the milk concentrate.

is noted. The vacuum pump is then started again and valve A is opened slowly. By a proportional calculation of the expansion (shown in mL in the graduated glass pipes) of the 'known' amount of air in the 'air' container, compared to the expansion of the unknown amount of air in the concentrate, it is possible to calculate the content of air in millilitre in 100 mL of concentrate (Haugaard Sørensen, 1978).

Solubility

Measurement of the insolubility index (II) is usually performed on milk powder. Should it, for example, not be possible to trace the problems with too high II by changing the drying parameters, it is recommended to perform the test on the concentrate. The amount of concentrate to be used is calculated as follows:

$$\text{Concentrate (g)} = \frac{\text{Powder (g)} \times 100}{\text{TS of concentrate (g 100 g}^{-1})}.$$

Powder = 10 g skimmed milk or 13 g whole milk; the rest of the test procedure is as described for powder.

Scorched particles

Measurement of these is usually performed on milk powder. Should it, for example, not be possible to trace the problem in the spray dryer, if there are too many scorched particles, it is recommended to look at the filter pad under a microscope. If the brown particle has not got a 'spray particle' structure, but rather that of a jelly lump, then the problem is most likely from deposits in the tubes in the evaporator. The scorched particle test is performed on the concentrate, and the amount of concentrate to be used is

$$\text{Concentrate (g)} = \frac{\text{Powder (g)} \times 100}{\text{Total solids of concentrate (g 100 g}^{-1})}.$$

Powder = 25 g skimmed milk or 32.5 g whole milk; the rest of the procedure is as described for powder.

5.6.4 *Powder*

Moisture

All milk powder has to meet a requirement for residual moisture. For skimmed milk, it is usually 4 g 100 g^{-1} and for whole milk usually 2.5 g 100 g^{-1}. There may naturally be differences from country to country.

The moisture content will have an influence on the keeping quality of the powder. High moisture content (i.e. high water activity a_w) will decrease the keeping quality, as the proteins will denature and the lactose that is found in an amorphous stage will crystallise causing the free fat to increase in WMPs and oxidation of the fat will be the result. The Maillard reaction, which is a reaction between the NH_2 group in the amino acid lysine and lactose, becomes more pronounced, and the powder may even become brown and lumpy.

The Maillard reaction is directly proportional to the storage time, temperature and residual moisture content. The moisture can be controlled by the outlet temperature of the dryer or by applying more heat to the Vibro-Fluidizer. Moisture absorption should be avoided and dehumidification of the cooling air is recommended in humid areas.

The packing material should be of such a quality that very little moisture vapour will penetrate the bag or container. As there will always be some vapour diffusion (and the diffusion direction is determined by the water vapour pressure) it is recommended to store the powder in a dry and cool place, where the water vapour pressure will be low.

Residual moisture in the powder is determined by a simple drying oven method. The powder is dried at 102–105°C for 3 h. The difference in weight (i.e. weight loss) is determined and the moisture is calculated as a percentage of the powder weight (Fig. 5.23).

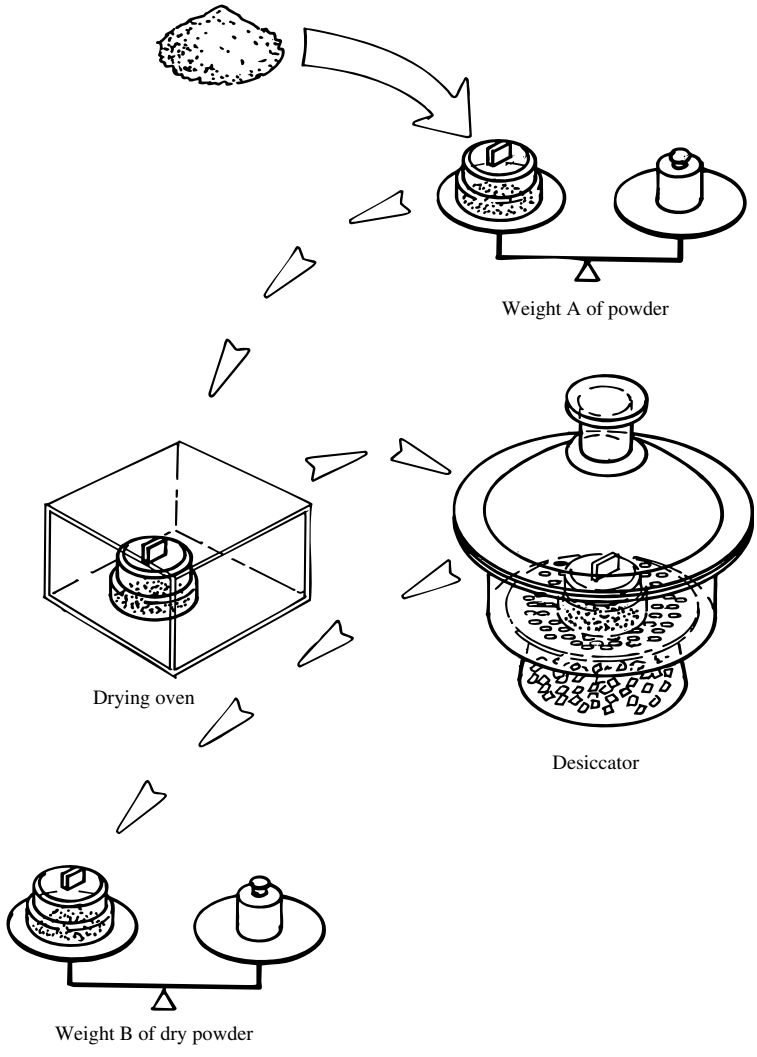


Fig. 5.23 Determination of moisture content of powders.

Various quick methods for determination of moisture have also been developed. They usually work with a powerful heating lamp, the voltage of which can be adjusted. This type of equipment will never be as accurate as the drying oven method, but is a great help during operation of a plant, as the operator can have quick response from the laboratory, enabling the determination of suitable drying parameters. Automatic control of the moisture content is measured with infrared light. The reflection from the sample is directly proportional to the moisture content, and the output is used to control the outlet temperature by regulation of either the feed pump or the heat applied to the heating section of the Vibro-Fluidizer.

WPNI

The undenatured WPNI (i.e. expressed as mg of undenatured whey protein nitrogen g^{-1} of skimmed milk powder containing 3.16 g moisture g^{-1}) is a measure of the heat treatment applied to the milk during the manufacture of powder, and it is the basis of the following heat classification, such as high-, medium- and low-heat powder. Typically, the heat classification of SMP by WPNI is as follows: high <1.5, medium $>1.5 - <6.0$ and low >6.0 mg WPNI g^{-1}). The method of analysis is detailed by GEA Niro No. A 21 a (http://www.niro.com; Sørensen *et al.*, 1978; see also IDF, 1982); the casein and denatured whey proteins are removed by filtration after precipitation of the rehydrated powder with NaCl. By adding acid (e.g. HCl), the proteins denature and develop a turbidity depending on the concentration of whey proteins. The turbidity is measured as a percentage transmittance in a spectrophotometre at a wave length of 420 nm and, by using a standard curve, the observed reading can be converted directly into mg of undenatured whey protein nitrogen g^{-1}. The WPNI is calculated using the following formula:

$$\text{WPNI at 3.16 g moisture 100 g}^{-1} = \frac{(100 + (\%\ \text{moisture in the sample} - 3.16\ \text{g 100 g}^{-1} \times (\text{WPN})))}{100}$$

Casein number

The method for CN analysis in milk powders is based on the premise that casein and denatured whey protein precipitate at pH 4.7. In contrast, un-denatured whey protein is completely soluble at this pH. Sweetsur (1976) describes a simple method for estimation of CN. Total nitrogen and 'non-casein' nitrogen (NCN) are estimated by Kjeldahl digestion. The NCN is estimated in a filtrate prepared by precipitation of casein and denatured whey protein using a mixture of acetic acid and sodium acetate to yield a final pH in solution of 4.7. The CN is calculated using the following formula:

$$\text{Casein number} = \frac{\text{Total nitrogen} - \text{NCN}}{\text{Total nitrogen} \times 100}.$$

Typically, the heat classification of SMP by CN is as follows: low <82, medium 82–90 and high >90.

Bulk density

The bulk density is an economically, commercially and functionally important property. When shipping powders over long distances, the producers are interested in a high bulk density in order to reduce the shipping volume. A high bulk density also saves packing material and storage capacity. For some powders the aims is a low bulk density, obtained by agglomeration, for optical reasons, or because of requirements for instant powder production.

The bulk density is defined as the weight of a given volume of powder and is expressed in g mL^{-1}, g 100 mL^{-1} or g L^{-1}. The reciprocal value is the bulk volume, which is expressed in mL 100 g^{-1} or mL g^{-1}. The bulk volume is usually used when a graduated cylinder glass is used for the determination. The volume of 100 g of powder is then measured in the cylinder. As to the other method giving the bulk density, the weight of the powder in a 100-mL cylinder is measured. Both results can naturally be converted to the other expression (Fig. 5.24). The value may either be expressed as tapped 0 times (loose), tapped 10 times (poured), 100 times or 1250 times. Various types of equipment can be used for the tapping. Also manual tapping is used. The intensity of the tapping naturally influences the value (see also IDF, 1995b).

The bulk density of milk powders is a very complex property, as it is a result of several other properties. However, the primary factors determining the bulk density are as follows:

- Particle density – given by (1) the solids density, a function of product composition, or (2) the content of occluded air in the particles.
- Amount of interstitial air – air between particles (agglomeration).

Particle density/occluded air

The bulk density is given by the density of the powder solids and the occluded air that is in the particles. The powder solids density expresses the density of solids without any air and is given by the composition of the powder. When the composition and the density of the single components are known, the density of the solids (D_{solids}) can be calculated using the following formula:

$$D_{Solids} = \frac{100}{\frac{\%A}{D_A} + \frac{\%B}{D_B} + \frac{\%C}{D_C} + \text{etc.} + \%W} \tag{5.1}$$

where %A, %B and %C are equivalent to the composition and D_A, D_B and D_C the corresponding solids density and %W is the percentage of moisture. The solids densities of various typical components in milk powders are as follows:

Solids (air and moisture free)	Density (g mL^{-1}) at 20°C
Milk fat	0.94
Solids-not-fat	1.52
Calcium caseinate phosphate complex	1.39
Amorphous lactose	1.52
β-lactose	1.59
α-lactose monohydrate	1.545

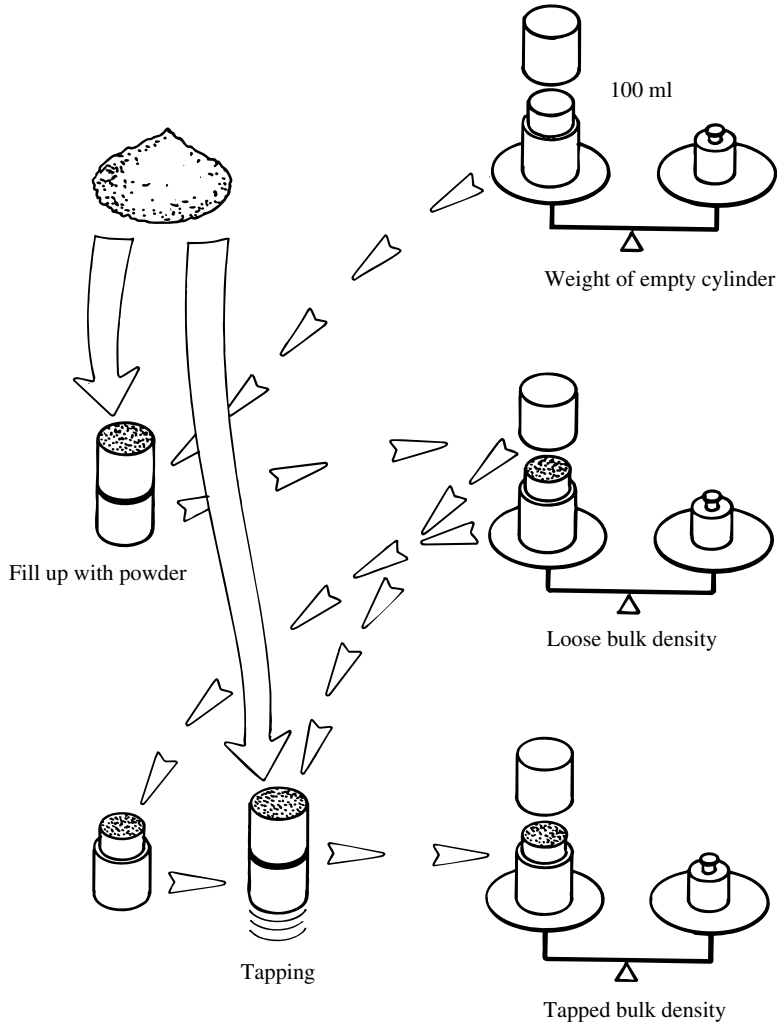


Fig. 5.24 Determination of powder bulk density.

Powder solids density cannot be changed without changing the composition and, thus, is for a given product constant.

The particle density may be measured in an air pycnometer. However, as this equipment is not available in all laboratories, the petroleum ether method will be discussed. A given amount of powder is mixed with a given volume of petroleum ether in a graduated measuring cylinder:

$$D = \frac{W}{V_1 - V_2} \tag{5.2}$$

where D is the particle density (g mL^{-1}), W is the weight of the powder (g), V_1 is the volume of powder + petroleum ether in mL and V_2 is the volume of petroleum ether in mL.

The occluded air content is calculated as follows:

$$V_{oa} = \frac{100}{D_{particle}} - \frac{100}{D_{solids}} \tag{5.3}$$

where V_{oa} is the volume of occluded air in mL 100 g^{-1} powder, $D_{particle}$ is the particle density [see formula (5.2)] and D_{solids} is the density of solids [see formula (5.1)].

The particle density for the reciprocal value of the occluded air content is influenced by many factors previously discussed and they are summarised as follows:

- *Heat treatment temperature of the milk prior to evaporation* – changes the denaturation degree of the whey proteins, and thereby their physical state and behaviour during drying. High temperature results in many denatured whey proteins being very compact and different from un-denatured whey proteins that are sponge like. Un-denatured whey proteins have a higher 'water binding power'. A bigger Δt or driving force is necessary to evaporate the last moisture with case hardening as a result. A high degree of denaturation will give a low occluded air content (high particle and bulk density) and *vice versa* (see Fig. 5.25).
- *Amount of air in the concentrate milk* – The amount of air in the feed naturally gives a high content of occluded air, especially if the surrounding air temperature during the critical stage of the drying is high causing case hardening.
- *Foaming ability of the concentrate* – The foaming ability of the feed is determining how much of the air whipped into the concentrate will remain there and in the created droplets.

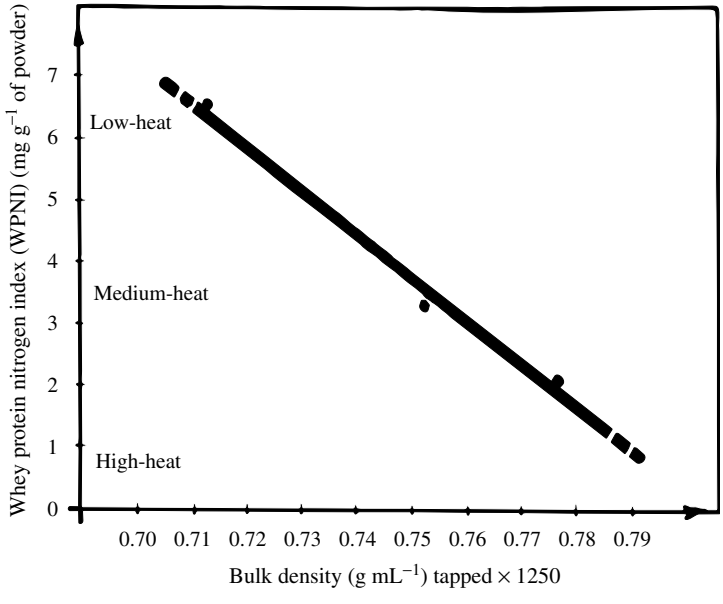


Fig. 5.25 Bulk density of skimmed milk powder (SMP) as influenced by the heat treatment temperature of the milk before concentration and drying.

- *Type of the wheel used or the size of the nozzle* – Besides the foaming ability of the concentrate, the type of a wheel and a nozzle is decisive in determining the amount of air that will be whipped into the concentrate.
- *Solids content in the concentrated milk* – The feed concentration plays an important role and high concentration gives less occluded air content.
- *Drying conditions (one-stage or two-stage)* – The drying conditions and temperature of the particle during the drying are one of the main factors. Gentle drying, that is, low surrounding temperatures as in two-stage drying, results in low occluded air.

Interstitial air

Interstitial air is a very complex property, the less the interstitial air (i.e. the air that occupies the space between particles/agglomerates) the higher the bulk density. The amount of interstitial air is determined by the particle size distribution and the degree of agglomeration. The content of interstitial air can be calculated as follows:

$$V_{ia} = \frac{100}{D_{powder}} - \frac{100}{D_{particle}} \tag{5.4}$$

where: V_{ia} is the volume of interstitial air in mL 100 g^{-1} powder, D_{powder} is the powder bulk density (tapped 100x) in g mL^{-1}, and $D_{particle}$ is the particle density in g mL^{-1} [see formula (5.2)].

A powder with particles of the same diameter would be ideal from a drying point of view, but undesirable from the bulk density point of view, as the air space (the interstitial air) between the particles will be very large thus resulting in low bulk density. The ideal is a wide particle size distribution with enough small particles to fill out the space between the medium and large particles thus resulting in a powder with high bulk density. There is, however, a limit as to how many small particles are wanted from a recovery point of view, plus the fact that a powder with many small particles will be dusty. Furthermore, they will negatively affect the flowability.

A wider particle size distribution, but in a bigger particle size spectrum is therefore wanted. This can be obtained by using high solids content and/or viscosity, reducing the velocity of the wheel or the pressure of the pressure nozzles, or using bigger nozzle sizes. The result will be very dubious in a single-stage dryer where the bigger particles call for higher outlet temperature thus increasing the occluded air content due to reasons already discussed (case hardening). Powders with extremely high bulk density can only be achieved in two-stage dryers.

As mentioned elsewhere, the powder leaving the chamber will be slightly agglomerated due to the primary agglomeration. In a one-stage dryer equipped with pneumatic conveying system, the problem does not occur due to the mechanical treatment it is exposed to. However, in a two-stage dryer, the primary agglomeration is significant. The agglomeration is developed due to the powder being more thermoplastic. As the mechanical treatment in the Vibro-Fluidizer is very gentle, the agglomerates are not broken up. A pressure conveying system is recommended, if a powder with very high density is wanted. It should be pointed out that the primary agglomeration has a positive influence on the flowability of the powder.

It has been observed that freshly made powder often exhibits a low bulk density that increases several days after the production. This is caused by the electrostatic charge of the powder making the particles stick together, forming 'agglomerates'. As the time passes, the powder will lose the charge and behave normally. An effective earth connection of all parts of the drying equipment can to some extent solve this problem.

Flowability

Flowability of a powder is not fully understood. Two different types of free-flowing powders mixed together will not necessarily be free flowing. A good flowability is obtained from large particles or agglomerates without small particles that will tend to decrease the bulk density. Also, the particle surface plays an important role, especially the content of free fat. Nozzles are generally believed to produce particles with better flow properties than the wheel, especially in WMP. A powder with a good flowability will especially increase the poured and loose bulk density.

Many attempts have been made to develop a suitable method for measuring the flowability. Some methods measure the angle of repose for a given amount of powder, whilst other methods measure the time it takes the powder to pass through a hole in a funnel with a given diameter. Common features for these methods are, however, that they are suitable for powders with a good flowability, whereas they cannot be used if the powder is not free flowing. Furthermore, the result is influenced by the ambient conditions, especially the humidity of the air. In addition, a method developed by NIRO is suitable for any kind of powder. In this method, the time is measured for a given volume of powder to flow through well-defined slits in a drum rotating with a given revolution min^{-1} (see Fig. 5.26).

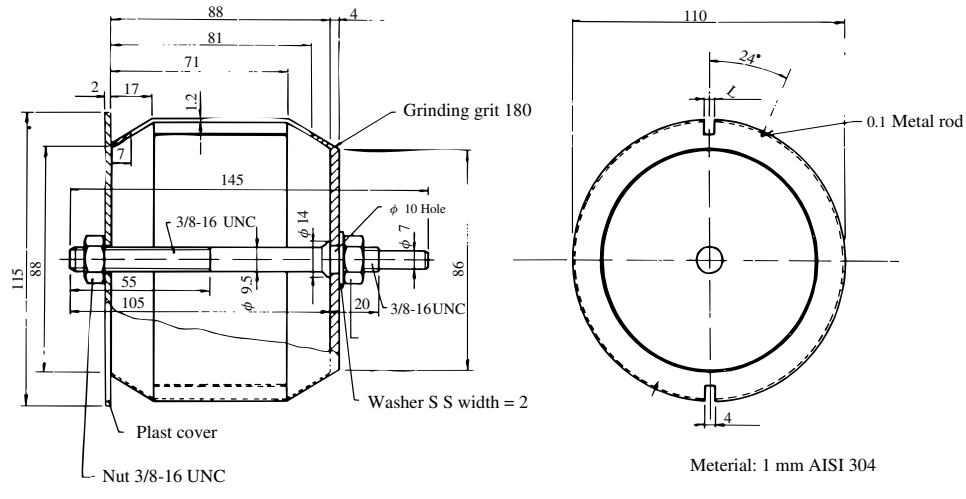
Solubility

It is obvious that milk powder has to be soluble in water. However, not all of the components in the powders are soluble when reconstituted in water. In powders produced in modern dryers, this amount is very small and approaching 100% solubility. Nevertheless, powders with a bad solubility are still produced and any dryer can in fact be incorrectly operated, resulting in a powder with bad solubility.

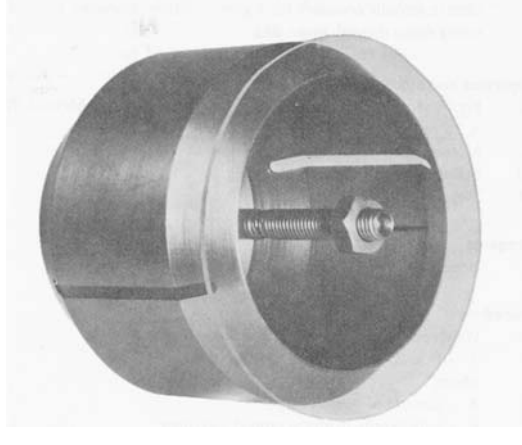
The method that is described below for measuring the solubility is very simple, well defined and easy to perform.

> Ten g of SMP, 13 g WMP or 6 g of whey powder (or equivalent amount of concentrated milk depending on solids content) is mixed with 100 mL of water at ~24°C in a mixer at high speed for 90 s. The milk is then left for 15 min, after which it is stirred with a spatula; 50 mL is filled into a graduated 50 mL centrifuge glass tube with conically graduated bottom. The glass tube is spun in a centrifuge for 5 min, the sediment-free liquid is siphoned off, the glass tube is filled up again with water (to make the reading easier), and the content is stirred up. The glass tube is then put in the centrifuge and spun for 5 min, after which the sediment is read (Fig. 5.27; IDF, 2002a, b).

The sediment is expressed in mL and is termed *insolubility index*. It is usually below 0.2 mL in powder from good quality milk dried in modern well-designed evaporators and



(a)



(b)

Fig. 5.26 An apparatus for measuring the flowability of milk powder.

dryers. The reasons for high insolubility index (i.e. bad solubility) in a powder may be many. It is usually denatured caseins or very complex combinations of casein–whey protein and lactose, the chemistry of which is not fully understood. The main contributing factors are as follows:

- Bad quality milk with a high development of lactic acid, meaning the bacterial activity, will result in a high insolubility index, as any extensive heat treatment will cause an irreversible protein denaturation, especially of the caseins.
- High temperatures of the concentrate during the evaporation will cause a pronounced age-thickening, resulting in viscosity increase and bad atomisation, that is, high temperatures during the drying.

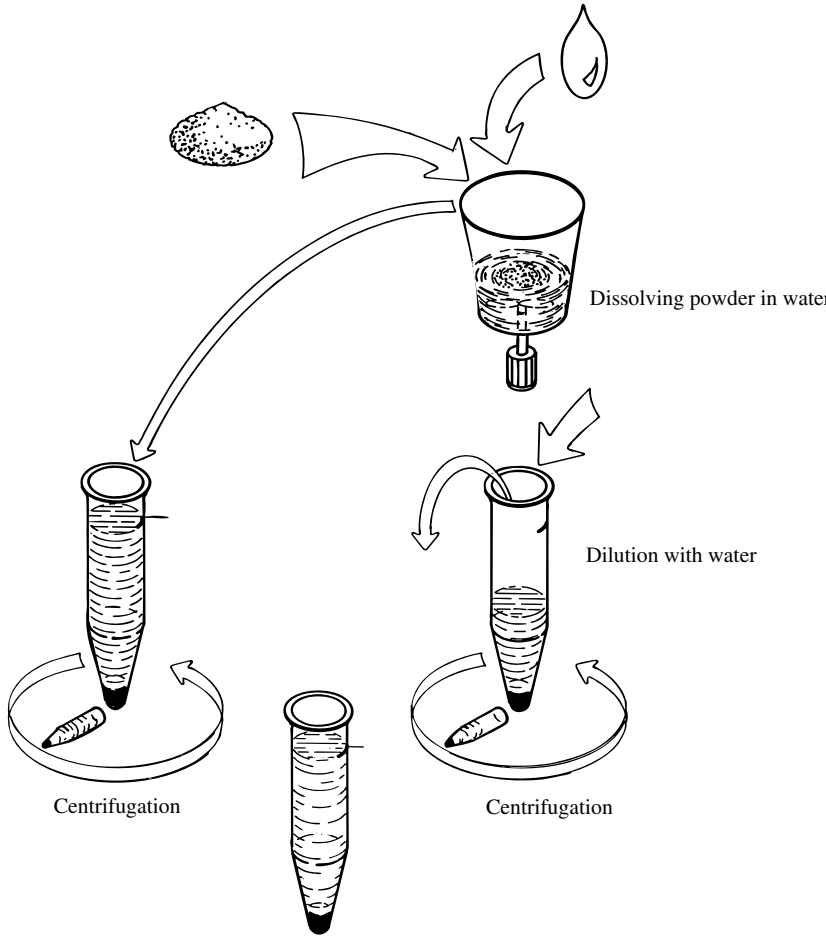


Fig. 5.27 Determination of insolubility index (II) of powders.

- Generally, it may be said that with higher temperatures and viscosities during the processing, a higher insolubility index may be expected. Powders with high lactose content, such as baby food, will practically never get a high insolubility index, as lactose protects the proteins from denaturation.

Powders dried according to the one-stage drying principle will more easily get a high insolubility index than from the two-stage drying principle.

It is not only the dryer which is to blame for high insolubility index. The evaporator may also harm the concentrate. It is, however, measured very rarely. If a factory has untraceable problems, it is recommended to investigate the concentrate. This is done by using the same method as described above, but with an amount of concentrate depending on the solids content and corresponding to the specified amount of powder. If milk powders with high insolubility index are used in 'compounded' products like baby food, a correspondingly higher insolubility index should be expected.

Scorched particles

Scorched particles are generally accepted to be a measure for any deposits in the drying chamber having been exposed to high temperatures, thus getting scorched, discoloured and at the same time insoluble. However, it is not only the dryer that contributes to the scorched particles, as even the raw milk may contain some dirt or sediment and, if not clarified in a separator, these will be found in the powder. Also from the evaporator, brown, insoluble, jelly lumps may contribute to the scorched particles, if deposits have been formed in the tubes due to insufficient coverage of the tubes or insufficient cleaning. If it has been concluded that the scorched particles originate from the dryer, the cause is very often deposits in the wheel or around the nozzles or in the air disperser. How to solve the problem may differ from case to case, but adjustment of the air disperser will usually help in most cases.

The test for determining scorched particles is simple and rapid:

> Twenty five g of SMP, 32.5 g WMP or 15 g whey powder (or equivalent amount of concentrate depending on total solids) is mixed with 250 mL of water of 18–28°C in 60 s in the same kind of mixer as used for the insolubility index. The milk solution is filtered and the filter pad is compared with a standard for classification. The scorched particles are expressed as A, B, C, or D depending on the intensity and colour of the particles left on the filter (see Figure 5.28).

The total fat content

The total fat content in WMP is a question of standardising the raw milk prior to the processing, and has got nothing to do with the drying process. The standardising is carried out either by adding skimmed milk or cream to the milk, or removing cream from the milk, depending on the content of fat in the raw milk and the fat content intended in the final powder. Standardising tanks equipped with agitators are used in most cases, but other methods are also recommendable. As the fat content in the raw milk in practically all cases is too high when producing WMP, SMP is sometimes used for standardising. The equipment needed is an in-line powder/liquid blender known from recombining plants. As the solids content will increase by adding SMP, the evaporator should be designed accordingly. For an accurate determination of the fat in WMP, the Rose-Gottlieb method is used and, for a quick determination of the fat content, the Gerber method can be used.

Surface free fat

In WMP, the fat is present as fine globules covered with a membrane substance and distributed evenly in the particles. However, not all the fat is protected by a membrane, especially on the surface of the particle, but it is also found inside the particles. This type of fat is termed *free fat*; it has a direct influence on the shelf-life of the powder and is directly responsible for the non-wettable surface when the powder is mixed with cold water.

Free fat in the WMP cannot be avoided, but may be reduced considerably by adopting the following approaches:

- Avoid excessive pumping and agitation of the raw milk, and recirculation in the evaporator should be avoided by all means.

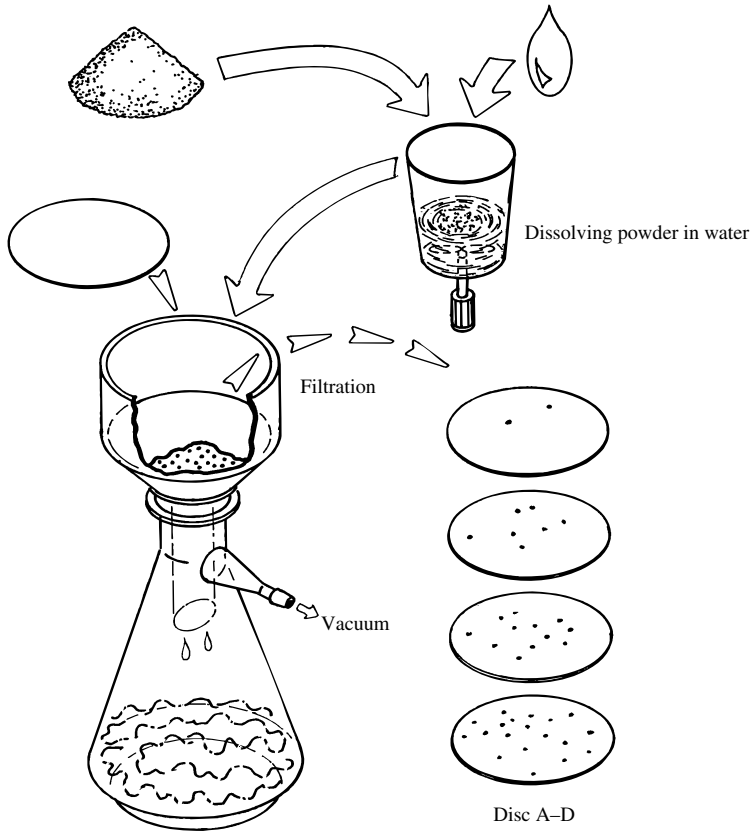


Fig. 5.28 Determination of scorched particles in powders.

- The heat treatment of the milk prior to the evaporator plays a role. For example, direct heating, especially at low temperature, results in low viscosity of the concentrate and a fine atomisation with a big surface to mass ratio leading to increased free-fat content. The free fat is most efficiently reduced by homogenisation of the concentrate, preferably in a two-stage homogeniser. In the first stage, a pressure drop of 6.9–9.8 MPa (70–100 kg cm^{-2}) is applied. The fat globules will disintegrate into small globules, which might – due to static electricity – agglomerate again, that is, they will consist of many small fat globules. In the second stage, a pressure drop of 25–50 kg cm^{-2} is applied breaking up the above-mentioned agglomerates.
- It is a general rule that nozzles produce a powder with a lower free-fat content than with the wheel, mainly due to the homogenisation effect of the nozzle.
- Any strong mechanical handling of the powder should be avoided, and then it is not surprising that the two-stage drying gives a powder with a lower free-fat content than the one-stage drying does.
- In plants with integrated fluid beds, the free fat will increase if the bed temperature is too low, signifying too high a moisture content in the powder, which results in lactose crystallisation (see Section 5.5.2).

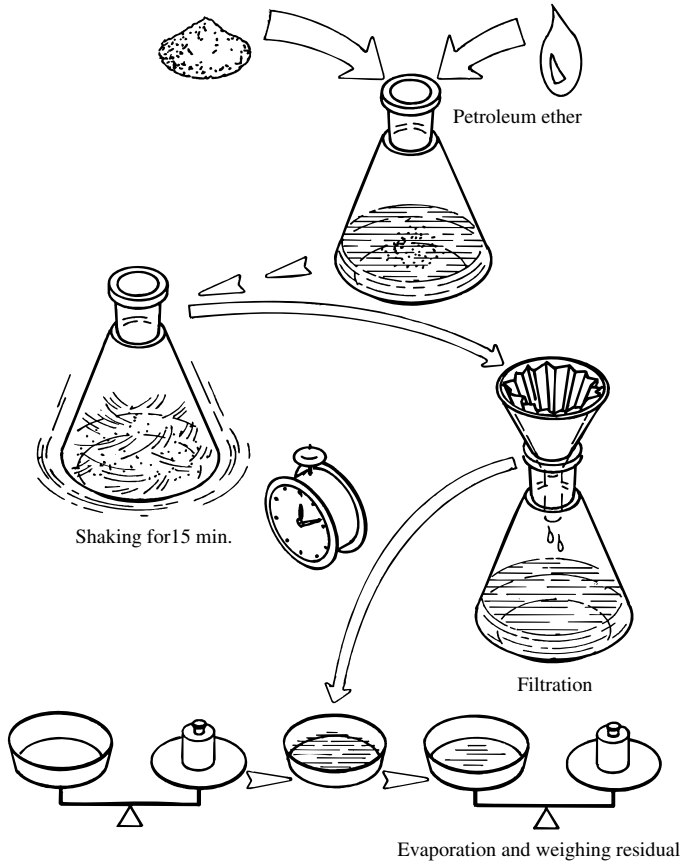


Fig. 5.29 Determination of surface free fat in powders.

To determine the free fat in the powder, 50 mL of petroleum ether and 10 g of powder are mixed slowly for exactly 15 min. The mixture is filtered and 25 mL of the filtrate is evaporated. The residue is weighed and the free-fat percentage is calculated either based on total fat or more commonly based on the powder (Fig. 5.29).

In another method for determination of the free fat, toluene is used, and the extraction time is sometimes as long as 24 h. The result will naturally be different from that obtained by petroleum ether extraction.

It is generally accepted that the petroleum ether extraction method gives out results representing the surface free fat, whereas the toluene extraction method gives out the total free fat, that is, also what is inside the pores and capillary network of the powder particle.

Wettability

Wettability is a measure of the ability of a powder to be wetted with water at a given temperature. This analytical method is used only when producing instant powders. It is obvious that the wettability depends on the surfaces of the agglomerates or single

particles – are they water repellent or will they absorb water too quickly, thus forming a film through which the water cannot penetrate?

Generally speaking, wetting is a process in which the gaseous phase at the surface of the solid phase is replaced by a liquid phase, all three phases coexisting for some time, so that a certain amount of intermixing and solutions (mainly of the solid and the liquid phase) are not only possible but usually unavoidable. Besides this, milk powder must be considered as a composite surface with the separately enclosed surfaces connected by more or less stable 'bridges' to form a complex capillary network. For simplification, let us first discuss the mechanism of wetting a single surface.

The factor deciding whether there will be any wetting at all is the interfacial tension between the particle surface and the water. SMP particles will usually be wetted easily (provided there is less than 0.03 g fat 100 g^{-1} on the surface), as the powder material is mainly lactose, being in an amorphous phase, and protein, both absorbing water readily. However, WMP particles are always covered by a layer of fat, making them water repellent. The amount of this surface free fat varies between 0.5 and 3 g 100 g^{-1} of the powder. This water repellency of the particles caused by their fat coating may be overcome, and an interfacial tension facilitating the wetting may be achieved, by adding a surface active agent to the surface free fat. It has been known for years that phospholipids such as lecithin are well suited for this purpose. Lecithin has the advantage of being a natural product and even a natural component of milk, and being both lipophilic and hydrophilic it is able to absorb water.

When the particles have been wetted, the individual components of the milk powder start dissolving and dispersing, thus forming a concentrated solution of milk around the particles. At the same time, the particles start sinking to the bottom, but it should be mentioned that, in order to make the particles sink, the density of the particles has to be greater than that of the water. The density of a particle depends on its composition and the amount of occluded air. During the first stages of reconstitution the density of the particles decreases, mainly because the lactose and the minerals, which are the heaviest milk components, start dissolving faster than the other components. At the same time, the density of the solution being formed is increased because of the dissolving lactose, so that the difference between the densities of the particles and of the surrounding liquid is reduced. The particle density may even become the same or lower than that of the liquid, so that, after the initial sinking, the particles start to rise again. To prevent this, the particle density should be high, that is, the content of occluded air should be low.

The reconstitution of a mass of powder is more complicated. As already mentioned, powder is a composite surface with a greatly ramified system of capillaries of various dimensions and a complicated geometrical pattern, thus having different capillary attraction effects. Under these conditions, there will be wetting not only on the surface of the water but also of particles lying above the surface, as the water is drawn towards them by capillary attraction. This replacement of interstitial air by water through capillary penetration is very often incomplete, as the amount of penetrating water is insufficient, thus leaving air bubbles between the wetted particles. In this way, all three phases are going on simultaneously, resulting in the coexistence of their products of varying concentrations. This coexistence is very dangerous, because after a short time, the space between the particles will be filled with milk of different, including high, concentrations. This results in a sticky jelly with islands

of unwetted powder and residual air. Furthermore, lumps that are wet and swollen outside and dry inside are created. As these are impervious to water, their complete reconstitution is extremely difficult even with strong agitation. However, to obtain fully reconstituted milk in a reasonably short time and with minimum effort, capillary penetration of water into the powder must be avoided. The capillary effect depends on the structure of the powder, such as the size of the agglomerates, the size and the amount of non-agglomerated particles, the amount of interstitial air and the specific surface area of the powder. Penetration of water into the powder is easily avoided/delayed – to allow dispersion before dissolution – when the powder consists of large agglomerates.

The analytical wettability method is simple and easy to perform:

> Ten g of SMP or 13 g of whole milk is poured into 100 mL water at a given temperature, usually 20°C ± 2°C. The time required for all the powder to be wetted is measured by means of a stop watch. The International Dairy Federation method (IDF, 1979) describes the use of 10 g SMP or WMP in 250 mL water at a temperature of 25°C (see Fig. 5.30).

SMP should be wetted within 15 s to be termed instant. For WMP, there is no requirement, but many producers of instant WMP manufacture the powder to the same standard as

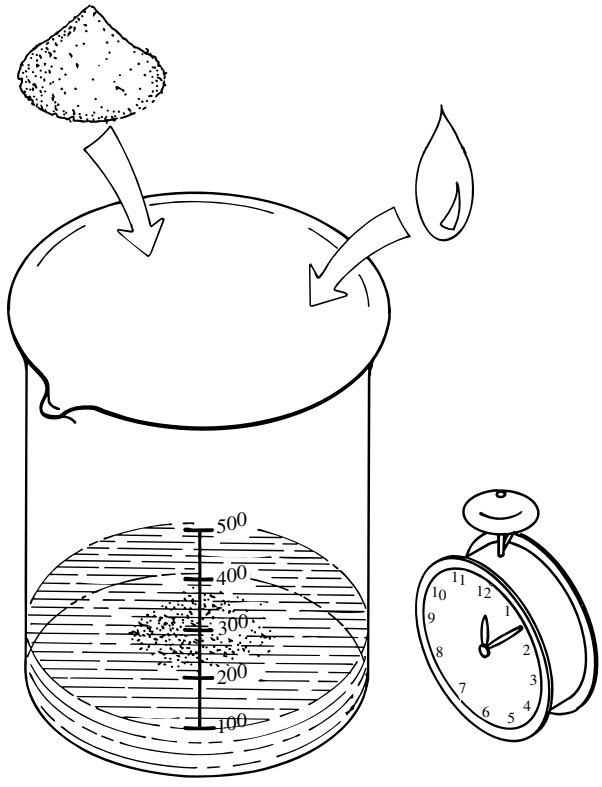


Fig. 5.30 Determination of wettability of powders.

valid for the SMP. However, for the subsequent dispersing process, especially for WMP, it is advantageous that the wettability is about 30–60 s, as it eases the subsequent dispersion of the powder into the water.

Dispersibility

Dispersability is another important property of instant powders. It is the ability to disperse in water by gentle stirring. This means that the powder should disintegrate into agglomerates, which again should disintegrate into single primary particles. To obtain good dispersibility of a powder, it is necessary that the powder is wettable and that the agglomeration is optimal, that is, no fine particles should be present.

The analytical dispersibility method is very difficult to define and perform, and the reproducibility is very poor. There are numerous methods, and the results cannot be compared. The (IDF, 1979) dispersibility test is based on determining the capability of a powder (25 g of skimmed milk or 34 g of whole milk) poured onto a surface of water (250 g at 25°C) to disintegrate into particles capable of passing through a 150-μm sieve when applying the prescribed manual stirring for 20 s. The amount of powder passing the sieve and being dissolved or dispersed is found by the determination of total solids of the filtrate and expressed in percentage as dispersibility (Fig. 5.31). The powder is considered instant by the International Dairy Federation (IDF, 1988, 1995a, 2005b), if the dispersibility is at least 85% (whole milk) or 90% (skimmed milk). However, plants with modern drying technology easily produce powders with a dispersibility of 95%.

There is no doubt that this test presents a more reliable basis for assessment of instant milk powders than the wettability test. On the other hand, it is a test requiring a relatively extensive amount of work, so it can hardly be used as a routine test. Besides, even when done by skilled workers, the reproducibility is rather poor. A more simple method is to pour 10 g of SMP or 13 g of WMP into 100 mL of water at room temperature and then manually stir with a teaspoon until the powder is dispersed, leaving no lumps on the bottom of the glass. The time used is measured by means of a stop watch. After some training, the reproducibility is fairly good and the method is quick. Furthermore, it has the supreme advantage that it is just what the consumer does when preparing a glass of milk.

Sludge

Sludge is similar to the International Dairy Federation dispersibility method (used only for instant cold water-soluble whole milk) (IDF, 1979), but only 12.5 g powder in 100 mL water at 25°C and 85°C is used. A 600-μm sieve is used. The residue on the sieve after filtration is weighed and recorded.

Slowly dispersible particles (SDP)

The same procedure, as for sludge, is used to determine the SDP of powders. After filtration through the 600-μm sieve the milk is poured into a test tube which is emptied again immediately. The remaining film with undissolved particles/agglomerates is compared with

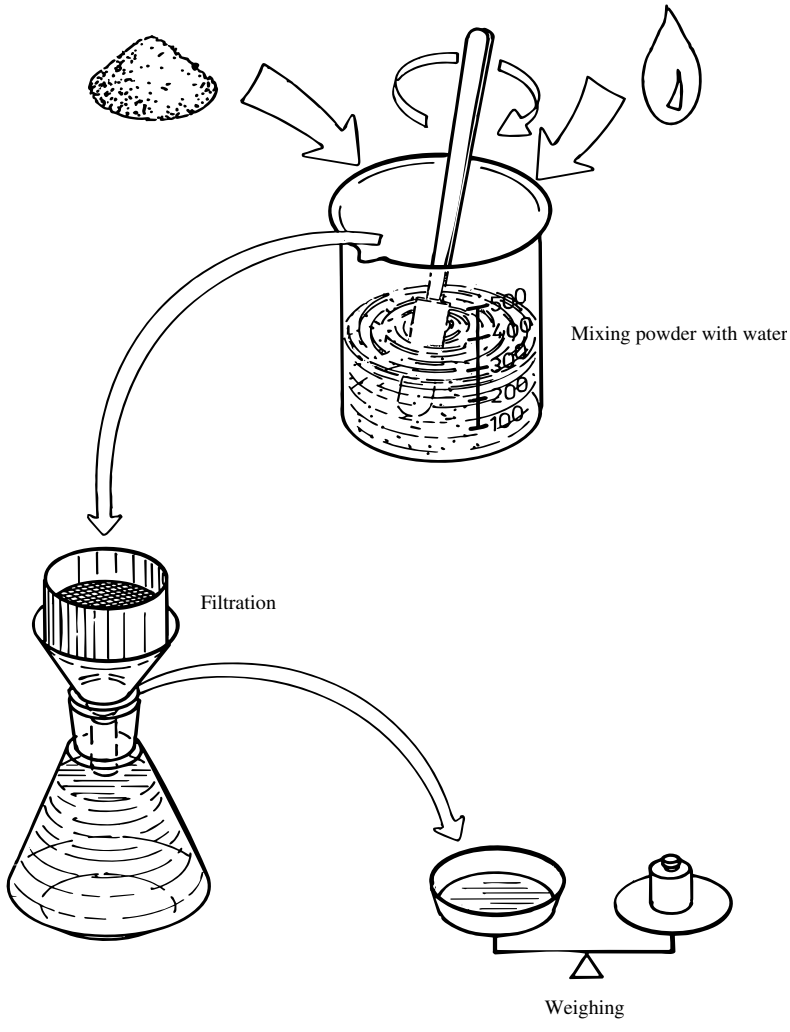


Fig. 5.31 Determination of dispersibility of powders.

a photograph 5-grade scale. The SDP is determined in both 25°C and 85°C warm water. The remedy to improve the SDP value is agglomeration.

Similar to the sludge test at 85°C, the reconstituted filtered milk is poured into two graduated conical centrifuge glasses and spun in a centrifuge for 5 min (i.e. similar to what is used for the solubility index). The result is expressed in mL sediment from the two glasses added together. The result should preferably be <0.2 mL.

Hot water test (HWT)

The HWT is similar to the sludge test using 85°C water. The reconstituted filtered milk is poured into two graduated conical centrifuge glasses, similar to those used for the

insolubility index, and spun in a centrifuge for 5 min. The result is expressed in mL sediment from the two glasses added together. The result should preferably be <0.2 mL.

Coffee test

The Coffee test is similar to the HWT, but using coffee at 85°C (see IDF, 2005c). Like SDP and the HWT, the result is determined by the degree of agglomeration but, for the coffee test, the heat treatment of the milk prior to the evaporation (80–85°C for 15 s, and the WPNI is ~3 mg g^{-1}), the content of Ca^{++} and total protein content are also important. However, with a high content of proteins in the milk (e.g. Jersey cows or milk from late lactation) it is difficult to produce powders with a good coffee test, which should be <0.4 mL. Standardising the milk with lactose or permeate is used in the industry to adjust the protein content. Concentrate preheating to 80°C and/or addition of phosphates and/or citrates to precipitate the ionic Ca^{++} can be used as well.

5.7 Conclusions

The microbiology of dried milk is determined by (a) the initial bacterial load in raw milk; (b) selective growth of psychrotrophic bacteria during transport from the farm to the processing plant; (c) pre-processing and bulk storage at the factory; (d) processing conditions during powder manufacture, especially heat treatment; and (e) the nature and extent of recontamination following processing. Techniques for hazard identification and control of critical operations are well understood and, as a result, it is usual for the microbiological quality of milk powder to be of a uniformly high standard.

The physical properties of milk powder are determined by (a) the heat treatment of the milk and the concentrate; (b) the spray dryer atomising equipment; (c) the dry matter content of the concentrate; (d) the handling of powder fines; and (e) the air flows and temperatures of the spray dryer. If the raw material is of good quality, the choice of an appropriate heat treatment and the right dryer concept for the product(s) to be dried will secure a high-quality product. A number of analytical methods exist to be used for production control as well as control of the final product quality.

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6 Casein and Related Products

H.S. Rollema and D.D. Muir

6.1 Introduction

Caseins have a long history as essential ingredients in the food and non-food industry. Caseins were already used in ancient times as a constituent of paints and glues. The first patent for the application of rennet casein in one of the first natural plastics dates back to the end of the nineteenth century. Up to now, various casein-derived preparations have been lending structure and/or functionality to a broad variety of products. Caseins are unique proteins in that they show an exceptionally wide spectrum of functionalities. The functionality of caseins is based on their peculiar molecular characteristics. All caseins are amphiphilic proteins. Due to their specific primary structure, they contain alternate hydrophobic and hydrophilic (charged) regions, which allow them to interact with both non-polar and polar matrices. In addition to this, caseins contain a number of serine phosphate residues, which can interact with divalent metal ions and calcium phosphate. All caseins show a tendency to association. In isolated state, they all self-associate in a specific manner; in a mixture of caseins, mixed associates are formed (Schmidt, 1982; Rollema, 1992; Horne, 2002).

The interaction between caseins is governed by a delicate balance between hydrophobic and electrostatic interactions. Electrostatic interactions can be tuned by pH (change in net charge) or metal ion binding to the phosphoserine residues (decrease in charge by screening). Caseins have an open and flexible structure (hardly any defined secondary structure), which can adapt to different conditions and matrices; this explains their wide applicability. Because of this particular property, caseins have been characterised as rheomorphic (from Greek: *rheos* = stream, *morphe* = form) proteins (Holt & Sawyer, 1993).

Caseins are able to form a wide range of structures. These can vary from an open flexible network as in sodium caseinate solutions to relatively inert dense particles, such as casein micelles. The difference between the two systems is the presence of calcium phosphate, which in the casein micelle crosslinks the caseins. In the presence of calcium, an intermediate system is obtained (calcium caseinate); induced by charge screening due to the binding of calcium ions to the phosphoserine residues, micelle-like particles are formed lacking the crosslinking effect of the calcium phosphate.

Their amphiphilic character combined with their flexible structure and their ability to adapt their structure in the presence of divalent metal ions and calcium phosphate, lends the caseins their functional properties with respect to heat stability, structure formation, thickening, foaming, emulsifying potential and water binding.

The methods used for the preparation of most casein fractions from milk all basically involve defatting of the milk followed by a destabilisation of the desired casein fraction

by insolubilisation. The destabilisation is either an isoelectric precipitation of the caseins (with a concomitant dissolution of the colloidal calcium phosphate of the casein micelle) or an enzymatic destabilisation of the casein micelle (renneting step).

Preparation of phosphocasein only involves the removal of serum components by membrane processes, whilst the preparation of enriched casein fractions requires a combination of different processing steps; often use is made of differences in dissociation behaviour of different casein components at low temperature.

The functionality of all protein fractions obtained from milk will critically depend on the quality of the raw material (milk) and on the processing conditions (heat load is one of the major factors) applied in the manufacturing procedure. From a nutritional point of view, caseins and caseinates are calcium-rich, carbohydrate-poor protein sources.

The manufacture, the functional properties and the application of casein fractions have been covered by a number of reviews (Southward, 1989; Mulvihill, 1989; Fox, 2001; Audic *et al*., 2003; de Kruif, 2003; Mulvihill & Ennis, 2003; Munro, 2003; Southward, 2003).

6.2 Products – definitions and structure

6.2.1 *Acid casein*

Acid casein is defined as the product obtained after isoelectric precipitation (pH 4.6) of the caseins from milk followed by a washing and a drying procedure. Distinction is made between lactic acid casein and mineral acid casein. The former type of casein is obtained from acidification by lactic acid produced by a starter culture growing on lactose. In general, lactic acid casein will show a low-lactose content. Mineral acid casein is obtained from acidification by mineral acids (e.g. HCl, HNO_3, H_2SO_4). The acidification dissolves the colloidal calcium phosphate in the casein micelles; part of the calcium remains bound to the casein. In the washing step, residual whey components are removed. Acid casein will contain a certain amount of calcium, which remains bound to the proteins. In addition, acid casein can contain some residual plasmin and plasminogen because this enzyme and its zymogen are known to associate with the casein fraction. The proteolytic activity of plasmin can affect the stability of products containing acid casein. Acid casein as such is, by definition, insoluble in aqueous media and it can be dissolved at an increased pH.

6.2.2 *Caseinates*

Caseinates are obtained by addition of alkali to acid casein. The increase in pH will dissolve the caseins. Depending on the cation of the alkalising agent, different types of caseinate, are obtained. Commonly used types are sodium-, potassium-, calcium- and ammonium caseinates. They essentially only differ in the type of counterion. Because of the particular interaction of calcium ions with the phosphoserine residues of the caseins, calcium caseinate is an exception; the functionality of calcium caseinate is quite different compared to that of sodium-, potassium- and ammoniumcaseinates. Dissolved calcium caseinate is a milky colloidal dispersion due to calcium-induced formation of micelle-like casein aggregates. Sodium-, potassium- and ammonium caseinates form nearly clear solutions; in most cases slight opalescence is observed due to the association of the caseins.

The composition of caseinates is, except for the mineral content, essentially similar to that of acid casein. Caseinates in principle show good solubility at neutral pH values. Sodium-, potassium- and ammonium caseinates associate in solution to form particles with sizes in the 20-nm range (Chu *et al.,* 1995; Dalgleish, 1997; Farrer & Lips, 1999; Lucey *et al.,* 2000; Horne, 2002). Addition of calcium induces the formation of considerably larger particles.

The functionality of caseinates can be affected by residual plasmin (for the same reasons as mentioned for acid casein), by the processing of the milk and by the processing in the caseinate-manufacturing process. In case the processing of the milk involves a more intense heat treatment, part of the whey proteins will denature and will form covalently linked aggregates with the caseins. Too high heat load during caseinate manufacture can induce chemical changes in the caseins, such as deamidation, dephosphorylation and hydrolysis (van Boekel, 1999; Hustinx *et al.,* 1997). Both whey protein denaturation and heat-induced changes will affect the functionality of the caseinate.

6.2.3 *Phosphocasein*

Phosphocasein is by definition a micellar casein suspension, that is, casein micelles to a certain extent free of whey proteins and other whey components. In this sense it is a model system for casein micelles. Phosphocasein is prepared by microfiltration (MF) of fresh skim milk often accompanied by diafiltration (Brule, 1979; Kelly *et al*., 2000). In this process, whey proteins, other whey constituents and possibly serum caseins are permeated; the casein micelles are retained. Diafiltration will enhance the purity of the preparation. In order to retain micellar integrity, the diafiltrant should match the milk serum composition [e.g. ultrafiltration (UF) permeate of the MF permeate]. Phosphocasein is a stable colloidal dispersion and, in this sense, is relatively inert. It is essentially low in whey protein and, therefore, will have a relatively high heat stability. Moreover, phosphocasein shows excellent rennet coagulability, which is to a much lesser extent affected by heat treatments as compared to milk.

6.2.4 *Rennet casein*

Rennet casein is obtained by proteolytic destabilisation of the casein micelles. In most cases, chymosin-like proteinases are used (bovine or microbial rennet), which cleave the $Phe_{105}-Met_{106}$ bond of κ-casein. The renneted casein micelles form a curd, which is cooked (heat treatment at $\sim 60^{\circ}C$ to increase the firmness and to inactivate the coagulant), washed and dried. Rennet casein consists of para-casein micelles, native micelles lacking their stabilising layer of caseinomacropeptide (CMP) (κ-casein-$_{(106-169)}$ peptide). In this respect, rennet casein curd is similar to cheese curd, the only differences is the absence of starter cultures and rennet activity (i.e. absence of proteolysis). Rennet casein is not soluble in water; it can only be solubilised at extreme pH values or by using calcium sequestering salts, such as phosphates, polyphosphates and citrates. Because rennet casein is precipitated at neutral pH, it has a high mineral (calcium phosphate content).

Most of the rennet casein is used in the production of processed cheese and cheese analogues because it can significantly contribute to the structure and the functional properties

of these products. In these processes, rennet casein is partially solubilised by the use of melting salts (calcium sequestering compounds) and its properties are tuned to the specific application (Southward & Walker, 1980; Mulvihill & Ennis, 2003).

6.2.5 *Co-precipitate*

A co-precipitate is a mixture of caseins and whey proteins in denatured form. The milk is heated to such an extent that the majority of the whey proteins are denaturated. Upon acidification, the denatured whey proteins co-precipitate with the casein fraction, and the precipitate is washed and dried. Denaturation of the whey proteins involves formation of aggregates of whey proteins, but also of whey protein–casein aggregates. The latter interaction will change the functional properties of the caseins considerably. Co-precipitate is a total milk protein preparation with properties different from those of the native protein fractions.

6.2.6 *Milk protein concentrates and isolates*

Milk protein concentrate (MPC) is usually obtained by UF of skimmed milk (removal of lactose and minerals) and spray drying. MPC has a protein content of 56–82 g 100 g^{-1} and it contains both casein (largely in the form of casein micelles) and whey protein in the same proportion as milk. The lactose content varies according to the protein concentration.

Milk protein isolate (MPI) is also prepared from skimmed milk by a combination of UF and a protein fractionation procedure. The preparations are characterised by a high protein content ($\sim$90 g 100 g^{-1}), and are low in lactose and fat; the casein–whey protein ratio corresponds to that of the initial milk.

Both MPC and MPI are protein sources low in carbohydrates. They are used both for their nutritive value and for their contribution to the texture of various food products (Huffman, 1999; Havea, 2006).

6.2.7 *Isolated and enriched casein fractions*

Interest in preparations enriched in one or more casein components is based on the particular functionalities of individual casein components (Maubois & Ollivier, 1997; Maubois, 1998; Mulvihill & Ennis, 2003). Fractionation methods for the preparation of enriched casein fractions have been developed to a stage where industrial application is feasible. This creates possibilities to exploit the specific properties of intact caseins (e.g. the emulsifying potential of β-casein) to develop novel products or to fine tune the functionality of milk proteins in product innovation.

Most preparations of milk protein fractions enriched in one or more casein components essentially use the temperature dependence of the dissociation of β-casein from casein micelles and β-casein polymers. At low temperatures (4°C), isolated β-casein occurs in a monomeric form (Payens & van Markwijk, 1963). In milk, a considerable part of micellar β-casein dissociates at low temperature into the serum phase (Dalgleish & Law, 1988). At low temperature, β-casein is even soluble at its isoelectric pH. Relatively pure β-casein

fractions and the complementary fractions enriched in α_S- and κ-casein have been prepared from sodium caseinate (Murphy & Fox, 1991), calcium caseinate (Ward & Bastian, 1996), phosphocasein (Pouliot *et al*., 1994) and renneted skimmed milk curd (Le Magnen & Maugas, 1992; Huppertz *et al*., 2006).

Recently, a method has been patented that allows preparation of relatively pure fractions of β- and α_S-casein, and a highly enriched κ-casein fraction from milk and sodium caseinate (Law & Leaver, 2003). In this method, the dissociation property of casein micelles or sodium caseinate particles at high pH, and the selective precipitation of α_S- and β-casein by calcium are utilised, which leaves an enriched κ-casein fraction. Subsequent precipitation at pH 4.6 of the α_S-/β-casein fraction at low temperature separates the α_S- and β-caseins. In principle, the processing applied is suited for large-scale applications, and the fraction enriched in κ-casein can be used to prepare a relatively pure CMP fraction.

6.2.8 *Casein fragments*

In the past few decades, protein fragments derived from milk proteins attracted a lot of interest because of their biological activity (Silva & Malcata, 2005; Fitzgerald & Murray, 2006; Korhonen & Pihlanto, 2006; Chen *et al*., 2007). The bioactivity of casein-derived peptides ranges from nutritional aspects (e.g. CMP, phosphopeptides) to effects on the cardiovascular, immune, nervous and digestive systems.

Caseinomacropeptide

CMP, κ-casein$_{(106-169)}$ peptide, a reaction product from the enzymatic cleavage of κ-casein by chymosin-like enzymes is a negatively charged partly glycosylated peptide with a molecular weight of ~8 kDa. CMP is also known as glycomacropeptide, caseinoglycomacropeptide and casein macropeptide.

A variety of biological activities, such as immunosuppression, inhibition of pathogen invasion and induction of satiety, are assigned to CMP (Brody, 2000; Thomä-Worringer *et al*., 2006). Moreover, the CMP is devoid of aromatic amino acids, which makes it a suitable protein source for patients suffering from phenylketonuria. CMP can be isolated from cheese whey (Doultani *et al*., 2003) or derived from rennet-induced proteolysis of κ-casein containing casein mixtures or κ-casein rich fractions (Thomä-Worringer *et al*., 2006).

Casein phosphopeptides

These are casein fragments harbouring the serine phosphate residues, which constitute the metal and the calcium phosphate binding sites of α_S-, β- and κ-casein. Because of the latter property, phosphopeptides can bind (divalent) metal ions and may function as carriers for a variety of minerals. It has been demonstrated that casein phosphopeptides possess an anticariogenic effect. Therefore, application of phosphopeptides has been proposed in the treatment of dental diseases (Silva & Malcata, 2005). Some of the phosphopeptides have an immunomodulating effect. In addition, phosphopeptides are released *in vivo* (e.g. in cheese) or they can be obtained by enzymatic hydrolysis using serine proteinases such as trypsin.

Casein peptides

In casein hydrolysates and in casein-containing fermented products, a wealth of bioactive peptides are encountered (Silva & Malcata, 2005; Korhonen & Pihlanto, 2006; Fitzgerald & Murray, 2006). The first bioactive peptides discovered related to milk are the β-casomorphins, fragments of β-casein-$_{(60-70)}$ segment. At present, a series of opioid peptides are known, originating also from κ- and α_{S1}-caseins.

A number of peptides, originating from all casein components, are known for their immunomodulating potential. They stimulate the immune system and, therefore, contribute to the human body's defence mechanism. In this respect, peptides possessing antimicrobial activity should be mentioned. These peptides are derived from β- and α_S-casein. A number of peptides have found an application in commercially available dairy products. Proline-rich fragments of κ- and β-casein are applied as antihypertensive agents in several products; they are known to inhibit the angiotensin-converting enzyme (ACE). The decapeptide α_{S1}-casein$_{(91-100)}$ is applied in a flavoured milk drink for its stress-relieving activity.

6.3 Methods of manufacture

6.3.1 *Introduction*

It is imperative that only milk of high quality is used in the preparation of casein and casein derivatives to conserve the full functionality of the products. The key objective is to prevent protein breakdown or reaction with other milk components. Protein breakdown is caused either by proteolytic enzymes or by exposure to alkaline conditions or excessive heat.

Two sources of proteolytic enzymes are common: first, enzymes may be derived from bacteria that grow in refrigerated raw milk. These micro-organisms are classed as psychrotrophic and comprise a mixture of Gram-negative rods, the most abundant being *Pseudomonas fluorescens*. This bacterial strain/species and most other psychrotrophs that contaminate refrigerated bulk milk produce extracellular protease that readily degrades milk protein. Although the bacteria responsible for secretion of these enzymes are readily killed by heat treatment at or ~65°C, the enzymes are remarkably heat stable – a considerable proportion of the enzyme activity survives ultra high temperature (UHT) heat treatment (typically, 140°C for 2–5 s).

The second source of proteolytic enzyme in raw milk is derived from the cow. When the mammary gland is under stress, tight junctions – normally closed – between the secretory cells lining the udder become 'leaky' and allow passage of plasmin from the bloodstream into the milk. Plasmin shares, with the extracellular protease from psychrotrophic bacteria, the ability of to survive severe heat treatment. The following three conditions predispose the mammary gland to leak plasmin into milk:

- In very early lactation when the gland is supplying components of the maternal immune system to the new-born calf, the plasmin levels are very high. This secretion – commonly called colostrum – must be excluded from the milk supply.

- In late lactation, the gland begins to involute in preparation for the dry period between lactations. As involution progresses, the extent of transfer of plasmin into the milk increases, leading to progressively higher levels of proteolytic degradation of the casein.
- When the mammary gland is injured or infected, components of the immune system flood the affected region. As a result, plasmin can pass freely into the milk potentiating protein breakdown.

Simple steps can be taken to avoid the ingress of degradative enzyme into milk. First, milk must be sourced from healthy cows in mid-lactation; this will ensure that plasmin levels are minimal. Second, bacterial contamination must be minimised, and the length and temperature of storage of the raw milk must be carefully controlled to ensure that the bacterial count does not exceed 10^6 colony-forming units (cfu) mL^{-1}. Several detailed studies have shown that, below this threshold value, there is little chance of premature product failure that can be linked to proteolysis.

Protein functionality can also be adversely affected by uncontrolled chemical reaction. The most likely losses of functionality are associated with exposure to high pH values during the conversion of acid casein to neutral caseinate or complex formation between whey protein and casein during heat treatment of raw milk. However, if appropriate care is taken during manufacture, conditions that can result in poor functionality can be avoided.

6.3.2 *Acid casein – conventional treatment*

Formation of acid casein relies on the fact that the solubility of the casein complex in skimmed milk reaches a minimum at the isoelectric point, corresponding to a pH value around 4.7. In contrast, the whey proteins in milk remain soluble at this pH value. Separation of the casein curd from the whey is achieved by sedimentation or sieving. Sedimentation can be enhanced by the application of centrifugal separators. The key to successful manufacture lies in effecting a clean separation of the acid casein from the whey followed by a washing process that completes removal of low-molecular-weight components.

The process falls into several distinct steps (Fig. 6.1):

- As much fat as possible is removed from whole milk by separation in a high-speed cream separator. The process is optimised to enhance the efficiency of fat separation. The optimum temperature of separation is around 45°C at which fat particles greater than 0.7 μm in diameter are successfully removed. The residual fat content is usually less than 0.04 g 100 g^{-1}. If a lower fat content is required, the residual small fat globules may be removed by using MF (Cheryan, 1998).
- The skimmed milk is heat treated to kill pathogen and spoilage bacteria and to inactivate enzymes. Ideally, pasteurisation is carried out close to the minimum severity allowed by law, that is, at 72°C for 15 s. More severe heating is avoided to minimise denaturation of whey protein. When conditions allow protein denaturation to occur, denatured whey protein forms a complex with the casein – with κ-casein in particular – and this complex co-precipitates with casein during the following processing steps.
- Acidification may be carried out over a range of conditions. Typically, pasteurised skimmed milk (4–6°C) is acidified using food grade acid. Hydrochloric (0.25 M),

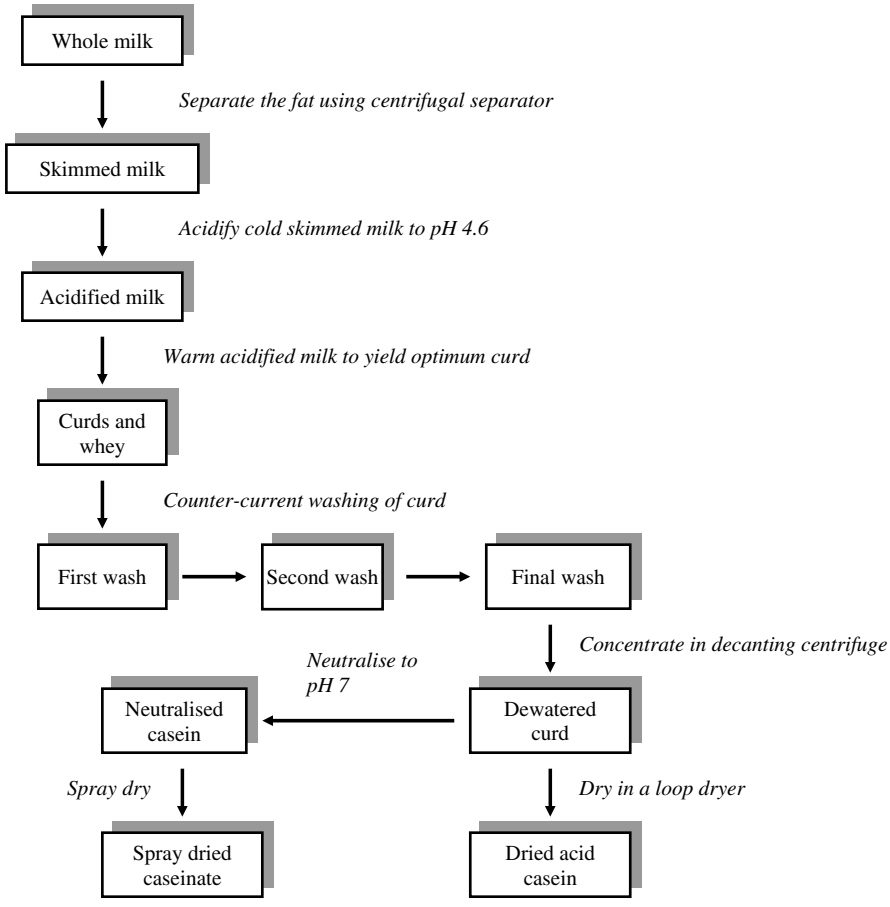

Fig. 6.1 Flow chart for the manufacture of acid casein and sodium caseinate.

sulphuric or lactic acid may be used to lower the pH of the skimmed milk from around 6.6 to 4.6 – the isoelectric point. At the isoelectric point, the casein attains its lowest solubility; however, the nature of the precipitate is highly temperature dependent. At low temperature ($<15^{\circ}$C) a very fine precipitate is formed, and this material is difficult to separate. However, as the temperature is raised, the size of the flocculated casein increases and the casein particles ultimately reach a size where precipitation is rapid. Moreover, at a higher temperature ($>50^{\circ}$C), coarse and dense particles are formed that precipitate freely, but contain added materials, such as lactose and whey protein. Ideally, the acid casein particles should sediment quickly to allow ready separation, but should be sufficiently porous for included material to be washed free from the particle. This can be achieved by injecting steam into the cold, acidified skimmed milk to raise the temperature to $40-42^{\circ}$C. The process is carried out in a series of tubes that ensure turbulent flow and encourage formation of discrete particles.

- Once particles with the appropriate physical properties are obtained, they are washed in counter-current towers with warm/hot water. On leaving the tower, particles are

recovered either on inclined screens or by centrifugal separation using a decanting centrifuge. The decanted curd is re-suspended in water and subject to further washing to reduce the content of lactose, whey protein and mineral, especially calcium salts. The casein may be heated by a final wash at 70–75°C with a holding time of 10–15 min.

- Next, the acid casein curd is dewatered in a decanting separator, then dried to <12 g moisture 100 g^{-1} in a dryer designed for drying curd, e.g. fluid bed driers, loop driers and attrition driers. Loop or ring driers comprise a large ring-shaped duct through which high-speed air and moist curd are continuously circulated. Residence time is short, avoiding heat damage, and the dried curd is tempered and blended before being milled to the desired particle size. In contrast, attrition driers carry out the processes of particle size reduction and drying simultaneously.

6.3.3 *Rennet casein*

Rennet casein is distinctly different from acid casein. Its manufacture depends on the action of an enzyme on κ-casein. Although κ-casein comprises only 11–12 g 100 g^{-1} of whole casein, it plays a key role in stabilisation of micellar casein. κ-casein is located largely at the surface of casein micelles, and confers stability by virtue of charged and glycosylated moieties on the molecule. Chymosin – the major enzyme found in rennet – cleaves κ-casein into glycomacropeptide (containing the charged and glycosylated groups responsible for colloidal stability) and para-κ-casein. After treatment with chymosin, casein micelles lose their stability and, in the presence of calcium salts, will precipitate.

During manufacture of rennet casein no acid is used and the pH remains around 6.6 throughout the process. Fat is removed and the skimmed milk is pasteurised as described for acid casein. However, the skimmed milk is then heated to around 30°C and rennet (ca. 0.014%) is added. After treatment at 30°C for around 40 min, renneting is complete and curd particles are formed. The mixture of curds and whey is then heated gently to encourage syneresis, and the curd particles are removed on an inclined screen. The optimum temperature for cooking depends on the concentration of casein in the milk, and is a compromise between formation at higher temperatures of large curds that are difficult to wash and fine precipitates that are easily washed, but difficult to sediment and separate by screening. The curd particles are washed in a series of counter-current steps then dewatered using a decanting separator and finally dried in a fluid bed, belt or loop drier.

6.3.4 *Caseinate*

Acid casein (and rennet casein) is not soluble and this limits the number of applications in which it is used. However, if the protonated acid casein is neutralised with an alkali metal base, the casein is converted to caseinate. Caseinate is highly soluble producing viscous solutions that are excellent for forming foams and emulsions. Conversion of acid casein to caseinate is simple, but must be carried out with care to avoid irreversible damage to the protein. The acid casein is suspended in water with vigorous stirring and alkaline solution (NaOH, NH_4OH, KOH, $CaCO_3$ or Ca $(OH)_2$) is added to raise the pH to 7.0. Concentration during the re-constitution phase is limited by the ultimate viscosity of the titrated protein, and it is usually below 20 g 100 g^{-1} solids. However, viscosity can be

reduced by increasing the temperature at which neutralisation occurs. Titration of the protein proceeds slowly and, as a result, care must be taken to avoid alkaline excursions of the pH of the slurry during the neutralisation process. If care is not taken and the pH of the slurry is allowed to rise significantly above pH 7.0, alkaline degradation in the form of deamidation and dephosphorylation may occur. Such damage has a marked effect on the functionality of the casein, damaging both the nutritive value and the ability of the protein to stabilise emulsions and foams. Once the acid casein is completely converted to caseinate, the solution is spray dried. The viscosity of the caseinate solution limits the efficiency of the drying process. To some extent, this problem can be ameliorated by heating the feed solution to the drier. Superheated feed, that is the feed heated at temperatures in excess of 100°C, has been reported to have been dried effectively. The use of such high temperatures depends on the fact that caseins are not denatured by heat, and are, thus, undamaged by heat treatment provided the pH is maintained at or below neutrality. However, if the pH exceeds 7.0, there is a serious danger of alkali-mediated damage to the protein.

6.3.5 *Co-precipitate*

It has been noted that in the manufacture of pure acid casein, denaturation of whey protein must be avoided. However, this 'defect' can be exploited to isolate almost all the true protein in milk. If skimmed milk is severely heated – typically at 90–95°C for 30 min – whey proteins are completely denatured. When the pH of the heated milk is subsequently reduced to pH 4.6 with mineral acid, not only is casein precipitated but the denatured whey protein is also rendered insoluble. The curd or coagulum formed in this way can be removed from the whey using inclined screens or separators, washed in a series of steps analogous to those used for purification of acid casein ($CaCl_2$ is often added to ensure complete recovery of the total milk protein) and dried in much the same way as acid casein. Various methods have been described to yield highly soluble co-precipitates. For example, Lankveld (1985) adjusted the pH of milk to 7.5 and then heated the milk at temperatures ranging from 80–145°C at residence times from around 20 min to a few seconds. After cooling, the pH was adjusted to 4.4–4.7 and the precipitated curd was separated, washed, dispersed in water, neutralised to pH 6.7 using alkali and the soluble product was spray dried. Grufferty and Mulvihill (1987) used a somewhat similar process to yield a totally soluble co-precipitate.

6.3.6 *Acid casein – supercritical fluid processing*

High pressure or supercritical precipitation with carbon dioxide (CO_2) provides an alternate means of isoelectric precipitation of casein from skimmed milk (Tomasula, 1993; Tomasula *et al*., 1998). It relies on the fact that CO_2 hydrolyses in aqueous solution to form carbonic acid:

$$CO_2 + H_2O \rightarrow H^+ + HCO_3^-.$$

Once sufficient hydrolysis has taken place, the pH of the solution falls into the range associated with isoelectric precipitation of casein (<4.8). To achieve successful hydrolysis

of CO_2 and casein precipitation, it is necessary to increase the pressure in the system to 14 MPa at 38°C.

A major advantage of CO_2, as a coagulant, is that there is no difficulty in removing the precipitant after the reaction occurs, and the casein has been separated from the whey. The use of CO_2 as a precipitant remained an experimental curiosity until the advent of continuous reactors. The skimmed milk is pressurised using a high-pressure piston pump (of the type used for homogenisation), CO_2 is mixed with the milk under pressure and reaction takes place in a series of holding tubes (Tomasula *et al*., 1998). Depressurisation must be carried out in a controlled and step-wise manner to maintain curd particle size. Although differences in functionality have been reported between traditional acid casein and carbon dioxide precipitate, these are limited in scope, and there is no evidence of fractionation of the casein taking place.

6.3.7 *Fractionation of casein*

Whole casein is highly functional and, as a result, has found widespread application. Nevertheless, this functionality is compounded from individual components with contrasting character. One key feature of variation between caseins is the number of phosphoserine residues, for example, α_{s2}-casein may have 12 serine phosphate groups, whilst β-casein has between 7 and 9 phosphoserine residues. In addition, the amphiphilic properties of the individual caseins vary markedly. In particular, β-casein has distinct hydrophilic and hydrophobic domains. The hydrophobic domain is rich in proline and confers unusual solubility characteristics upon β-casein. Such differences are reflected in the ability of the protein to bind calcium and in important physical properties. Although large differences exist between the individual caseins, these differences have been largely ignored because of practical difficulties in separating the caseins on a commercial scale. Fractionation of casein is readily carried out in the laboratory using powerful dispersants followed by chromatographic treatment. However, such methods are not practical for large-scale preparation of casein fractions. Nevertheless, two methods of preparing casein fractions have received attention with a view to commercialisation.

The first method yields an almost pure β-casein fraction together with a corresponding β-casein depleted material. The essence of the technique relies on the fact that at temperatures at or around 0°C (freezing point), β-casein is soluble – that is, it is the non-micellar form – over a wide range of pH values including its isoelectric point (pH 4.6). Thus, if a solution of sodium caseinate is cooled to 2–4°C and the pH is subsequently reduced from ca. 7.0 to 4.6, the α_{s1}-, α_{s2}- and κ-casein precipitate, whereas the β-casein remains in solution. The β-casein-depleted material may be removed by centrifugal separator and, after re-suspension and washing, is dried. Maubois (1991) described a number of applications of membrane technology that allow β-casein to be selectively removed from skimmed milk. Recovery of the β-casein from the supernatant is straightforward. The cold acidified solution is warmed to 35°C, and a flocculate of pure β-casein is formed. The pure β-casein is recovered by centrifugal separation, and may be spray dried after conversion to caseinate. To achieve complete separation it is necessary to repeat these operations several times – the rate limiting step is the solubility that can be achieved by the β-casein at its isoelectric point (see also Hoffmann *et al*., 2006, for an alternative method for enrichment of milk protein fraction by MF).

A second newer method of fractionating casein involves dispersion of casein in alkaline solution (above pH 10.5), followed by selective precipitation using $CaCl_2$ solution (Law & Leaver, 2003). By choice of appropriate reaction conditions (casein concentration, temperature and calcium concentration), fractions rich in κ-casein and depleted of κ-casein can be formed. For example, κ-casein concentration in the enriched material can be increased to twice that of the starting material, and in the depleted fraction it can be reduced to 25 g 100 g^{-1} of that of whole casein. The processes used are very simple involving mixing, centrifugal separation and spray drying (Fig. 6.2).

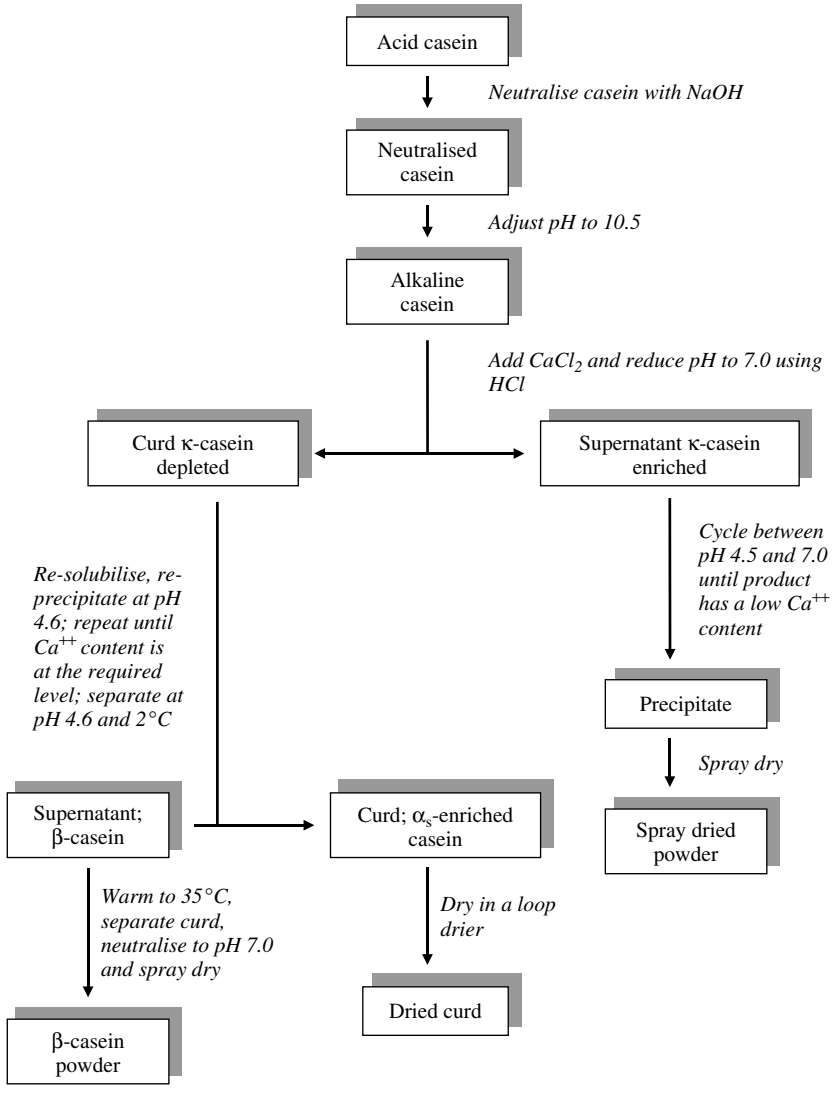

Fig. 6.2 Fractionation of whole casein. Data compiled from Law & Leaver (2003).

6.3.8 *Total milk protein*

Although co-precipitate can be accurately described as total milk protein, heat treatment, essential to ensure recovery of whey protein, results in a product with poor solubility. However, if skimmed milk is concentrated by membrane processing, concentrates containing 38–42 g 100 g^{-1} protein may be obtained. In practice, almost all protein is retained using membranes with a nominal cut-off corresponding to 30 kDa. Such membranes reject little lactose, and maintain high flux rates for substantial periods. If the process of diafiltration is also applied, the lactose and mineral contents can also be reduced (typically to <0.1 g 100 g^{-1} in the case of lactose). In practice, diafiltration may be applied in different ways. However, it is often convenient to apply the following sequence of operations:

- First, concentrate skimmed milk by UF to 20 mL 100 mL^{-1} of the original volume.
- Second, add two volumes clean, potable water to concentrate/retentate and mix.
- Third, re-concentrate by UF to the original final volume.
- Repeat steps two and three.
- Spray dry the concentrate.

Total milk concentrate manufactured in this way retains the protein in its natural, that is, undenatured state and conserves functionality.

Kelly *et al*. (2001) reported on the methods of manufacture of products that they classified as milk proteinate and phosphocasein. Milk proteinate was produced by first reducing the pH of skimmed milk to well below the isoelectric point of both casein and whey protein (in the pH range 2.3–3.5) (Kelly & O'Kennedy, 1998). In this range, the protein is soluble, but has a net positive charge. The acidified milk was then heated at 90°C for 10 min and the proteinate precipitated at pH 4.6. The acid proteinate curd was treated in essentially the same way as normally produced acid casein and converted to proteinate.

6.3.9 *Casein-derived peptides*

There is a growing interest in protein fragments (peptides) isolated from casein that exhibit biological activity. Some examples include the following:

- *Phosphopeptides from* β*-casein* – possess activity related to calcium availability; they are used for calcium supplemented drinks and also for natural repair of teeth.
- *CMP from* κ*-casein* – alleged to promote satiety by stimulating secretion of cholecystokinin.
- *Proline-rich peptides from* β*-casein* – shown to have antihypertensive activity.
- *Decapeptide from* α_{s1}*-casein* – promoted as a natural relaxant providing relief from stress.

The principles involved in the manufacture of enriched sources of these peptides involve a simple sequence of steps.

- First, if possible, the substrate should be rich in the target peptide. For example, for peptides derived from β-casein, such as phosphopeptide, treatment of purified β-casein will ensure a high yield (potentially three times that of whole casein).
- Second, enzymes should be chosen to selectively cut the protein backbone to yield the maximum quantity of the target peptide.
- Third, effective separation of the target peptide from the mixture of hydrolysed protein is required. This can be achieved by complex formation, differential solubility or on the basis of molecular size.

Phosphopeptides from β-casein – Preferably, the target peptides should be isolated from pure substrate. In the case of β-casein, this material, though currently expensive, is available in commercial quantities from the application of the Law & Leaver (2003) process. Typically, such material contains a minimum of 80 g 100 g^{-1} β-casein. By subjecting the β-casein to degradation by trypsin, a mixture of degradation products including the desired phosphopeptide is readily obtained. Aoki *et al*. (1998) describe ethanol precipitation as a means of large-scale preparation of casein phosphopeptide complexed with micellar calcium phosphate. Naito *et al*. (1994) provide an alternate method of separation of phosphopeptide from a trypsin digest. They treated the digest at pH 4.0 to about 5.0 and discarded the precipitate. The pH of the supernatant was adjusted to about 3.0 and the precipitate was once more discarded. The pH of the remaining supernatant was raised to pH 6.0, and ethanol and $CaCl_2$ were added to precipitate an acid-soluble phosphopeptide.

CMP can be isolated from cheese whey or from rennet casein whey. The latter substrate provides a purer product because there is less chance of contamination by starter cultures or salt – the necessary ingredients of cheese. Kawasaki *et al*. (1993) provide the basis for separation. Although the molecular weight of monomeric CMP is 7000 Da, at pH 7.0, CMP exists in an aggregated form. The pH of clarified whey is reduced to pH 3.5 and the whey is treated using membranes with a cut-off value of ~50 kDa. A volume concentration of 5 was applied, followed by diafiltration (volume dilution = 5) to yield retentate containing whey protein and permeate containing CMP (and other low-molecular-weight material). The CMP was recovered from this permeate after adjustment of the pH to 7.0. This promotes aggregation of the CMP allowing selective recovery using a membrane with a cut-off of 20 kDa (a volume concentration factor of 90 was applied).

Proline-rich peptides – in particular, valine-proline-proline and isoleucine-proline-proline – have well-documented antihypertensive properties. They may be isolated from trypsin digests of casein (Tolton *et al*., 1999) or from fermentates of *Lactobacillus helveticus* (Tossavainen *et al*., 2005). The fermentation route produces a substrate mixture rich in the target peptides, and subsequent separation is made on the basis of size using UF membranes with cut-off values between 1.4 and 3.0 kDa.

Stress-relieving peptide has been isolated from α_{s1}-casein and its efficacy has been confirmed in a series of clinical trials. The peptide is a decapeptide (residues 91–100) from α_{s1}-casein, and is prepared by selective membrane separation after enzymic hydrolysis of α_{s1}-casein; the product is marketed under the trade name of Lactium.

6.4 Functionality

6.4.1 *Solubility*

Acid and rennet caseins and some co-precipitates are not soluble in water and, as a result, their applications in food are restricted to fat and water binding or to nutritional fortification. In contrast, sodium, potassium and ammonium caseinates are highly soluble in water at neutral pH. Calcium caseinate is stable to some degree above pH 5.5, but stability is calcium dependent and the protein takes the form of aggregates or 'micelles'. Within the isoelectric well (pH 3.9–5.2), whole casein is insoluble – β-casein is an important exception due to its being weakly soluble at its isoelectric point, provided the temperature is below 5°C. It is interesting to note that between 5 and 0°C the solubility of β-casein increases significantly with decreasing temperatures. Below pH 3.5, the solubility of caseinate increases progressively as pH is lowered.

6.4.2 *Heat and alcohol stability*

Sodium, potassium and ammonium caseinate, milk proteinate and total milk protein are remarkably heat stable – they endure treatment at 140°C for at least 1 h. In contrast, calcium caseinate is susceptible to heating – a dilute (1 g 100 mL^{-1}) solution gels at 50–60°C.

The alcohol stability of caseinates follows heat stability. At low calcium concentrations, caseinates are remarkably stable in ethanol and this property makes them ideal for use in compound alcoholic beverages like cream liqueurs. In this context, Kelly *et al*. (2001) report that milk proteinate prepared by their process (Kelly and O'Kennedy, 1998) conferred a further improvement in the stability of model cream liqueurs when compared to standard sodium caseinate.

6.4.3 *Viscosity*

The viscosity of caseins has been extensively studied as a tool for exploration of the hydrodynamic behaviour and conformation of the protein. However, in practical applications the data are less certain. Nevertheless, viscosity limits the preparation of concentrated solutions of caseinate. Most caseinates can be used to prepare solutions in the concentration range 10–15 g 100 g^{-1} but, even at high temperatures, it is difficult to handle solutions with greater than 20 g 100 g^{-1} solids. Viscosity is logarithmically related to protein concentration, but the solutions are non-Newtonian and shear dependence increases with increasing concentration. Temperature dependence is inversely related to temperature–log viscosity $\propto 1/T$ (where T is in kelvin). In addition, calcium concentration and pH also influence viscosity. Viscosity of sodium caseinate solutions is highly dependent on pH with a minimum value ca. pH 7.0.

6.4.4 *Formation of protein-stabilised emulsions*

From a technical perspective, a valuable aspect of the functionality of casein is its ability to form extremely stable emulsions. β-casein is more surface active than α_s-casein; the

lowering of surface tension is β-casein > α_{s1}-casein > κ-casein > whey protein. Formation of protein films at lipid interfaces takes place in three phases:

- First, the protein molecules diffuse to the interface.
- Second, the protein is adsorbed at the interface – hydrophobic protein is favoured over hydrophilic protein.
- Finally, following adsorption conformational change may follow. At this stage β-casein may displace α_s-casein. However, equilibrium is reached comparatively slowly and the properties of the emulsion may be adjusted before equilibrium is reached and the properties of the interfacial layer become fixed.

It is not necessary for the protein to be intact for its functionality to be expressed. For example, CMP – a fragment of κ-casein – has been reported to possess good emulsifying properties. Casein foams easily, though the foam is not particularly stable. Given the wide range of functionality described above, it is not surprising that casein and related milk protein products find widespread use in food applications. It is beyond the scope of this article to describe these in detail – for a comprehensive review the reader is referred to Southward (1989).

6.4.5 *Functionality of peptides derived from casein*

Although a substantial number of bioactive peptides have been described, only a limited number have been commercially exploited. The four peptides dealt with in this article are credited with the following biological functionality:

- Casein phosphopeptide is believed to enhance absorption of dietary calcium by aiding transport of the calcium across the gut wall. In addition, preparations of the peptide containing calcium are credited with the ability to repair damage to tooth enamel.
- CMP is distinguished by its lack of the amino acid phenylalanine (and tyrosine). As a result, it can play a key role in supplying protein nutrients to those who suffer from Phenylketonuria. In addition, CMP has been reported to induce secretion of cholecystokinin – a gastric hormone that influences satiety. As a result, CMP may provide a useful aid in appetite control.
- Proline tripeptides are known to have activity like ACE, a well-known agent for relief of hypertension.
- The decapeptide from α_{s1}-casein $f_{(91-100)}$ has been found to possess stress relieving properties.

6.5 Quality control

It is commonly known that different commercially available preparations of apparently identical milk protein products can show a significant variation in functionality when used as ingredient in certain applications (Ennis & Mulvihill, 1999, 2001; Lucey *et al*., 2000; O'Sullivan & Mulvihill, 2001). Therefore, an efficient quality control of all ingredients

required for an industrial application is essential to ensure a constant product quality.

The quality of a casein preparation will be determined by a number of factors:

- *Quality of the raw material (milk)* – Raw milk with a low bacterial count from a well-defined (constant) source should be used. The heat load applied to inactivate micro-organisms should be minimal: sufficient to ensure good microbial quality and mild enough to prevent any denaturation of whey proteins.
- *The processing applied* – The processing conditions should enable the elimination of any indigenous proteinases (e.g. plasmin and cathepsin D) and the inactivation of any enzyme used during processing (e.g. chymosin in the case of rennet casein). The overall heat load in the processing should be minimised to avoid heat-induced changes in the proteins or denaturation of residual whey proteins. Finally, the processing should ensure an efficient removal of non-protein components.

Some general guidelines concerning the standard composition and specifications of casein preparations are given by Southward (2003).

In general, relevant parameters for the characterisation of casein preparations are as follows:

- General quality: protein, fat, lactose and moisture- and ash contents.
- Minerals: Ca, Mg, Cl, K, Na.
- Nitrogen distribution: total nitrogen (total N), non-casein nitrogen (NCN) and non-protein nitrogen (NPN).
- Protein pattern by reversed-phase high-performance liquid chromatography (RP-HPLC) and/or capillary zone electrophoresis (CE).
- Proteolytic activity: for example, plasmin and residual chymosin.

The lactose and the mineral contents are indicative of the efficiency of washing. The nitrogen distribution reflects the integrity of the proteins. The protein pattern is indicative of the protein integrity, but also reflects the heat load applied in processing (heat-induced changes in proteins often cause increased heterogeneity and lead to peak broadening in chromatograms). The (residual) proteolytic activity reflects processing conditions, and can be decisive with respect to stability of products derived from the specific casein preparation. In case of hydrolysates and preparations of casein fragments, the content of the active component (purity of the preparation) will be of importance.

Apart from the general quality aspects mentioned above, a test related to the specific functionality required in a given application, will be decisive for the suitability of the casein ingredient. An example of such a test has been given by Ennis *et al.* (1998) and Ennis & Mulvihill (1999) for rennet casein used in the manufacture of processed cheese.

The qualifications of a casein preparation will strongly depend on the specific applications. In some cases, specifications involving general compositional and functional parameters will suffice. In other cases, most probably with more sophisticated applications, the development of specific tests aimed at the desired functionality will be required in order to characterise and evaluate casein preparations.

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7 Dried Whey, Whey Proteins, Lactose and Lactose Derivative Products

P. Jelen

7.1 Introduction

Whey, the by-product of the cheesemaking and casein manufacturing processes, consists primarily of water (about 93 g 100 g^{-1}), with the lactose and the whey proteins being the most abundant and economically important components of the total solids (TS) fraction (Table 7.1).

The whey constitutes 90% of the original milk volume with 50% of the original milk components remaining when the casein is removed. Some of these components are present in the fraction referred to globally as 'whey protein', presently attracting much research attention regarding the potential physiological functionality of this fraction and/or of its individual constituents (Anonymous, 1996; Regester *et al*., 1997; Gill & Rutherfurd, 1998; Nelson *et al*., 2002; Middleton *et al*., 2003, 2004). Similarly, the most abundant component of whey, the milk sugar lactose, can be considered as having positive nutraceutical roles (Schaafsma & Steijns, 2000; Jelen & Tosavainen, 2004) besides being the main source of energy for the newborn. The problem of total whey utilisation essentially means finding new opportunities for total lactose utilisation, and several promising lactose derivative products are now being manufactured industrially. Finally, depending on the origin of the whey, some or most of the milk minerals are also found in the various types of whey produced by the dairy industry today (Sienkiewicz & Riedel, 1990).

Although the various types of whey and the main whey-based products contain some of the most valuable milk nutrients, processing of whey into marketable attractive final products faces a major economical roadblock, the high moisture content. This is compounded by the sheer volumes of the diluted material that must be handled (~9 kg of whey is generated for every kilogram of cheese produced). The use of the various whey, whey protein, lactose or mineral products as food ingredients or as raw materials for further conversion into novel nutraceutical products requires, in almost all cases, that the products are available in a dry (or at least in a highly concentrated) form. Thus, the removal of water is a major technological step in the handling of this important dairy material. This treatise is intended to provide a brief overview of some of the challenges encountered in converting a major ecological burden into an array of economically attractive ingredients and retail powdered products.

7.2 Types and composition of raw whey and main whey-based powders

There are two basic types of whey: (a) sweet, originating from manufacture of cheese and casein produced by the rennet coagulation of casein and (b) acid, resulting from processes

Table 7.1 Typical chemical composition (g L^{-1}) of sweet and acid whey.

Component	Sweet whey	Acid whey
Total solids	63.0–70.0	63.0–70.0
Lactose	46.0–52.0	44.0–46.0
Protein	6.0–10.0	6.0–8.0
Calcium	0.4–0.6	1.2–1.6
Phosphate	1.0–3.0	2.0–4.5
Lactate	2.0	6.4
Chloride	1.1	1.1

Data compiled from various sources.
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based on destabilisation of the milk casein colloid by acidification to below pH 5.0. While both whey types contain approximately the same amounts of whey proteins and lactose, the main difference is found in the calcium and lactic acid contents (Table 7.1). For more details on the various types of whey and their composition, numerous reviews, book chapters and books can be consulted (see Jelen, 2003).

7.2.1 *Standard and modified whey powders*

Worldwide, the bulk of the dried whey-based products is represented by regular dried whey, produced with little or no major pre-treatments except for the often-used removal of residual milk fat and/or of casein fines. Demineralisation of the whey by electrodialysis or ion exchange (Burling, 2003) is practised with increasing frequency, and results in modified whey powders suitable for use in infant formulas and other applications where the high mineral content of the regular whey powder would constitute a problem. Similarly, a portion of the lactose can be removed by crystallisation for production of partially delactosed whey powder that has a proximate composition similar to that of skimmed milk powder (SMP) and can be used in some cases as the SMP substitute. Removing the whey proteins, usually by ultrafiltration (UF) or other membrane processes, for production of various whey protein products (see Sections 7.4 and 7.6) results in the production of a whey-like fluid, the UF permeate, which can also be considered as an example of a 'modified whey product' for the purposes of this writing. Typical compositions of the standard and modified whey powders are shown in the Table 7.2.

7.2.2 *Whey protein*

Approximately $^1/_5$ of the protein fraction found in milk, referred to summarily as whey proteins, are neither rennet nor acid coagulable, and thus remain in the whey generated by practically all the commonly used cheese- or casein-making processes. It is well known that most of the protein species included in this heterogeneous whey protein fraction are sensitive to heat; heat treatments at temperatures higher than 72°C lead to denaturation

Table 7.2 Typical chemical composition (g 100 g^{-1}) of major types of dried whey products.

Product	Protein	Lactose	Minerals
Regular dried whey powder	12.5	73.5	8.5
Demineralised (70%) whey powder	13.7	75.7	3.5
Demineralised (90%) whey powder	15.0	83.0	1.0
UF permeate powder	1.0	90.0	9.0
Special whey protein concentrate[a]	35.0	50.0	7.2
Whey protein concentrate	65.0–80.0	4.0–21.0	3.0–5.0
Whey protein isolate	88.0–92.0	<1	2.0–3.0
Traditional lactalbumin	86.0–90.0	3.5–5.0	1.5–3.0

UF = ultrafiltration.
[a]Skimmed milk replacer.
Data compiled from various sources.
Reprinted from Whey processing –utilization and products, Jelen, P., from Encyclopedia of Dairy Sciences, (eds. Roginski, H., Fuquay, J.W. & Fox, P.F.), Academic Press, pp. 2739-2745, Copyright 2004, with permission from Elsevier.

and, especially in the absence of casein, to coagulation, insolubilisation and precipitation. In most cases, the precipitate is prone to sedimentation; however, under conditions of low ionic strength and pH 6.5–7.0, heating of whey protein systems produces non-sedimenting aggregates termed as *soluble polymers* (Britten *et al*., 1994). The heated whey protein coagulum is generally very fragile, and can be easily broken down into very fine particles which, however, are still insoluble and thus produce a sediment in heated whey systems, especially after acidification of these systems to approximately pH 4.5 aqueous systems. The sediment can be decanted, washed and dried easily; such whey protein powder is often referred to as *traditional lactalbumin* or simply (but incorrectly) lactalbumin.

The whey protein fraction includes some of the most nutritionally and nutraceutically valuable milk components, such as lactoferrin, lactoperoxidase, immunoglobulins and other minor protein species. Although these unique powders are being manufactured by some specialised processors, the bulk of the whey protein powders being manufactured industrially today consists of whey protein concentrates (WPCs) and whey protein isolates (WPIs). Typical composition data for of some of these products, included in Table 7.2, were generalised from numerous references and information from industrial manufacturers. More information on the various whey protein products, including their characteristics and technological processes used in their production, are provided elsewhere in this volume.

7.2.3 *Lactose and modified lactose products*

Another traditional whey-based dry powder (after regular dried whey) is isolated lactose, in its various industrial forms. The predominant crystalline lactose powder is α-lactose, produced nowadays predominantly by crystallisation from UF permeate below 93.5°C, followed by separation and drying of the more or less (depending on the grade of lactose being produced) pure lactose crystals. The much less frequently produced β-lactose requires crystallisation above 93.5°C where this lactose form becomes less soluble than its

Table 7.3 Pharmaceutical lactose products and their trade names.

Product	Specifications/processing conditions	Trade names
α-Lactose	100 mesh (>125 μm)	Tablettose
	Agglomerated/granulated	Cellactose
	Spray dried (4/5 crystals, 1/5 amorphous)	Ludipress, Pharmatose
Anhydrous lactose		
α-Lactose	Heated to >130°C	NA
β-Lactose	Crystallised at >93.5°C	NA

NA = not applicable.

mutarotatory isomer α-lactose. In general, lactose is neither very soluble nor very sweet. The process of lactose hydrolysis can be used to increase both sweetness and solubility; however, this also increases the hygroscopicity of the final mix. For this reason, hydrolysed lactose products are typically available as concentrates rather than as dried products.

One of the main uses of dried lactose is in pharmaceutical applications as a binder in production of pills. Some manufacturers offer modified lactose powders with additional ingredients including cellulose and other 'lubricants'. Examples of typical pharma-lactose powders are given in Table 7.3.

Since the market for lactose has been generally static, many attempts to use lactose as a raw material for production of a variety of commercially attractive products have been recorded in the literature and several such products are now available industrially. An overview of commercially produced derivatives of lactose is shown in Table 7.4. The market value of the lactose derivatives is generally higher than that of lactose, and it can be anticipated that ongoing research efforts will likely lead to more products being developed in the future, providing significant opportunities for application of lactose derivatives as functional food ingredients. One of the first commercially successful products was lactulose, used frequently as a laxative. In recent years, attention is being focused on prebiotic lactose derivatives, such as lactosucrose or other galacto-oligosaccharides. Numerous reviews focusing on the main lactose derivatives can be found in the literature (e.g., Harju, 1993;

Table 7.4 Main types of lactose derivative products and their forms.

Product	Form	References
Hydrolysed lactose	Syrup	Geilman (1993)
Lactulose	Syrup, possibly powder	Strohmaier (1998)
Lactitol	Crystalline powder	Harju (1993), Timmermans (1998)
Lactobionic acid	Spray dried powder	Harju (1993), Gerling (1998)
Galacto-oligosaccharides	Powder	Timmermans (1998)
Lactosucrose	Powder	Kawase *et al.* (2001)
Tagatose	Powder	Bertelsen (2002)

Table 7.5 Composition of a dairy mineral mixture powder (data from a single manufacturer).

Component	Content (g 100 g^{-1})
Total minerals	77.5
Calcium	24.0
Phosphorus	13.5
Lactose	10.0
Protein	5.0

Geilman, 1993; Timmermans, 1998; Strohmaier, 1998) and these provide some general information on the principles of the manufacturing processes, including the conversion to dried powders where applicable. Technical details may be available in the patent literature, as most of these specialised processes were developed by the manufacturers as proprietary.

7.2.4 *Other whey-based powdered products*

With the increased interest in whey protein products (WPC, WPI), the fractionation of whey into its individual components has become a popular option. After the separation of the whey proteins and the recovery of lactose, the remaining fraction contains mainly the minerals. Commercial 'whey mineral powders' based on this residual whey fraction are now marketed by several companies as a physiologically optimal mixture, their composition being close to the mineral composition of human blood. Another possible use of these powders is as a substitute for NaCl in the production of butter or cheese, avoiding the need to purchase the salt for companies producing both types of products. Spray drying of whey mineral mixtures is easy since their lactose content is low. Table 7.5 shows a general composition of one such powder.

7.3 Unit operations in the production of concentrated and dried whey and whey-based products

As is typical of production of concentrated and dried foods, the classical multi-stage evaporation, followed by a suitable drying process, are by far the two most common unit operations relied on in production of whey-based concentrates and powders. Although in general, the overall process design is similar to the production of SMP, there are several important differences in the equipment design and the conduct of the drying operation. Most whey-based products are difficult to dry, needing specialised equipment. Even the finisher stage of the evaporator used for production of concentrated whey is of a different design from that used for the production of milk concentrates.

Other concentration and fractionation processes, used often especially in the production of modified whey powders and whey protein products, include the whole family of membrane processes, as well as the ion exchange, electrodialysis, chromatography and

other advanced processing techniques. In addition, virtually all processing schemes include the classical dairy technology steps of centrifugation, heat treatment or other forms of heating, and often various lactose crystallisation steps. The immediate heat treatment of all cheese whey materials containing starter micro-organisms is one of the most important pre-treatment steps to avoid excessive development of lactic acid by the continuing fermentative activities.

There are different reasons for the inclusion of the lactose crystallisation step, from the obvious, in the production of crystalline lactose, to the technologically necessary, in drying of regular and modified whey to avoid the resulting hygroscopicity of the dried product. Consequently, the construction of the crystallisers and the conduct of the crystallisation processes should be different. In the first case, the objective is to facilitate production of large lactose crystals for ease of their separation from the mother liquor and subsequent washing. In contrast, the pre-crystallisation of lactose before drying of the various whey powders should result in very small crystals (not exceeding 50–100 μm) so as not to impede the subsequent spray drying process. A typical pre-crystallisation step may consist of controlled cooling (about $2^{\circ}C\ h^{-1}$) for 12–16 h with seeding by finely ground lactose powder. Flash cooling should not be used to avoid spontaneous nucleation and production of false grain, resulting in excessively large particle size distribution.

Another step used sometimes in the drying of regular whey powders is the deliberate denaturation of the heat-sensitive whey proteins by a severe heat treatment in the 'hot well', or by high-temperature heating (80–90°C) combined with long holding in an extended tube assembly. Of course this treatment will produce whey powder with diminished protein solubility which, however, may not be detrimental in many applications of such whey powders. This heat treatment may diminish possible difficulties in the evaporation step where, if the temperature exceeds about 70°C, the heat sensitivity of the whey proteins could cause excessive burning-on on the heating surfaces and/or deposit formation in the calandria of the evaporator, leading occasionally to the complete stoppage of flow.

Most whey and whey-based powders are produced by the spray drying technique. Extensive description of the technological aspects of spray drying in general – and as applicable to whey and whey products in particular – is available (Sienkiewicz & Riedel, 1990; Pisecky, 1997) and will not be repeated here. Using inlet and exit air temperatures typical for the production of regular whey powders (160–180°C and 80–90°C, respectively) together with a properly conducted evaporation process, this technique is well established and does not result in excessive whey protein damage. For products where the native state of the whey protein is required, such as in the case of WPC and WPI powders, a two-stage drying should be used with rigorous control of the inlet and especially exit air temperatures. These and similar modified whey products require special care and, often, specialised drying equipment is employed not to impair the final product quality. The technologically simpler and much less expensive roller drying technique can sometimes be used economically for the production of lower-quality whey powders (Peters, 2005), but its harsh heat treatment effects diminish the utility of this traditional technique for producing high quality whey-based powders.

The UF milk or whey permeates are essentially lactose solutions with a relatively high level of impurity caused by the minerals and other minor whey components. Spray drying of permeates is not only difficult due to the high lactose content but also uneconomical

due to the present low market value of these powders. The lactose pre-crystallisation step is a necessity, further increasing the costs. A recently developed and patented Tixotherm process is claimed to avoid these expensive steps by combining evaporation in a specially constructed paddle processor with the lactose crystallisation step, followed by drying in a much less expensive fluid bed dryer (Pisecky, 2005). An on-line literature search revealed at least five companies presently offering similar processing lines, based on evaporation to very high solids content ($\sim$76 g 100 g^{-1} concentrate) with subsequent further water removal by highly modified spray dryers (in one case using an upstream dryer) or by alternative drying means. At the present time, experience in these novel technologies is scarce; it appears that the critical control points are the homogeneity of whey supply, control of excessive viscosity of the high TS concentrates to minimise difficulties in handling and avoidance of any proteolysis in the whey supply.

In the production of isolated lactose, an additional challenge arises regarding the need to dry or otherwise dispose of the residual mother liquor, an especially difficult problem in the case of lactose production from UF permeates. Since the typical yield of isolated lactose is only about 60–65% currently, additional lactose recovery from the mother liquor is of great commercial interest. Demineralisation of the UF permeates increases the purity of the mother liquor dramatically, from approximately 50% to up to 90%. The use of fluid bed dryers, belt dryers or the even simpler tumble dryer tunnels is common in the lactose manufacturing plants for drying the washed lactose crystals, but equipment needed for handling the mother liquor is complex.

Dried whey and modified whey products may require special packaging techniques, especially in the cases where the lactose crystallisation techniques have not been included or are not being used properly. The spray drying of poorly pre-crystallised product will result in highly hygroscopic powder that would cause caking in storage if not properly protected by moisture-impermeable packaging. The high-value WPCs and WPIs offered in 'high-end' retail markets, such as for body builders and active sport enthusiasts, are often packaged in portion control aluminium packages, sometimes using inert or modified atmosphere gases.

7.4 Technological complexities in the production and storage of whey-based products

Drying whey and whey-based products is not easy and requires specialised equipment and various technological adjustments. The main causes of these difficulties are the high content of relatively insoluble and hygroscopic lactose, the heat sensitivity of the whey protein and, especially in the case of acid whey products, the high content of relatively non-volatile lactic acid. The continued development of lactic acid in the improperly stored and handled sweet whey before its processing may make the situation even more difficult.

7.4.1 *Heat sensitivity of whey protein*

The heat sensitivity of the whey proteins, which, in the absence of casein, self-associate and form insoluble complexes at temperatures above 70°C, is one of the main reasons

for some special aspects of drying technology as applied to whey and whey products. As indicated above, the whey drying technology sometimes includes a deliberate whey protein denaturation step to minimise the production difficulties caused by the formation of milk stone, protein deposits and films on the equipment surfaces. This treatment usually diminishes the processing difficulties but results in the limited solubility of the final dried product, which may or may not be a problem depending on the intended application. The heat sensitivity of the various whey proteins becomes an important consideration in the production of various whey protein-enriched products.

7.4.2 *Low solubility and hygroscopicity of lactose*

The limited solubility of lactose (~19 g 100 g^{-1} water at room temperature, but increasing rapidly with increased temperature, see Table 7.6) determines the operating conditions for the evaporation step, especially for ordinary whey powders produced without the pre-crystallisation of lactose. In this case the whey is evaporated into approximately 42–44% TS to minimise difficulties with the formation of non-uniform lactose crystals. The concentrate is then fed directly into the spray dryer. The resulting regular whey powder will be hygroscopic because, in the drying step, the non-crystalline lactose will not have time to crystallise, forming the hygroscopic lactose glass. Such products must be packaged properly in water vapour-impermeable packaging to avoid caking. To produce a non-hygroscopic, non-caking powder, the lactose is pre-crystallised before the drying step by cooling the concentrate rapidly to about 25–30°C, adding a finely ground lactose powder as seeding material and stirring vigorously. In this case, the concentrate should have a much higher TS content (~60%) to facilitate the production of very small lactose crystals that do not jeopardise the spray drying step. This also results in higher process economy as removing the water by evaporation is much cheaper than by drying. The quality of the non-hygroscopic pre-crystallised whey powder is also generally higher than that of the ordinary whey powder.

7.4.3 *Content of lactic acid*

With the exception of rennet casein whey, most of other types of whey originate from processes involving micro-organisms capable of converting lactose to lactic acid. In some

Table 7.6 Effect of temperature on solubility of lactose.

Temperature (°C)	Concentration of saturated solution (g 100 g^{-1} H_2O)
20	19.2
30	24.0
50	44.0
70	77.8
80	98.9

Data compiled from various sources.

cases, this conversion is an integral part of the technological sequence (as in production of cottage or quark cheeses or sometimes acid casein), while in the cases of virtually all cheeses, the presence of starter micro-organisms in the whey can lead to rapid production of lactic acid if the whey is not cooled down and/or heat treated immediately after draining from the cheese vat. The presence of lactic acid is detrimental to the whole whey drying process and affects negatively the final product quality. Drying acid whey, containing high amounts of lactic acid, is exceedingly difficult as lactic acid is relatively non-volatile and so cannot be removed easily from the concentrate by drying, thus making the product very sticky. The evaporation concentrates containing high amounts of lactic acid are very viscous, and their handling can become unmanageable especially in the lactose pre-crystallisation step. However, high acidity *per se* is not necessarily detrimental for drying, as evidenced by the ease with which hydrochloric acid whey resulting from the production of acid casein can be dried. Neutralisation of the lactic acid wheys by calcium hydroxide is sometimes used, resulting in a high-calcium lactate content; this is preferable to using sodium hydroxide (Pisecky, 1997).

7.4.4 *Propensity for non-enzymatic Maillard browning reaction*

Since lactose is a reducing sugar, exposing concentrated whey with their high protein and lactose contents to high temperatures during evaporation, hot-well treatments or drying may result in unavoidable but generally undesirable non-enzymatic browning. This problem may become even more acute during improper storage of the dried whey powders as the reaction rate is known to be accelerated at low a_w, especially in more acidic environments (Dattatreya *et al*., 2006, 2007). Thus, the initial acidity of the whey being dried (and consequently, the final acidity of the dried whey powder) may play a significant role in the development of the undesirable brownish discolouration. To minimise the browning defect, the temperature control during manufacturing as well as storage of whey powders is essential.

7.4.5 *Foam formation and its potential detrimental effects during drying*

The tendency of the whey proteins to foam, especially in heated whey (Jelen, 1973), can lead to undesirable effects during spray drying. Foamed whey concentrate can cause severe equipment damage, especially during production of pre-crystallised whey powders. In at least one known case (Jelen, personal experience), excessive foaming has been the reason for frequent breakages of the atomiser wheel shaft in spray drying of cheese whey in which significant proteolysis occurred as a result of microbial activity. The tendency of the foam to climb upwards on the atomiser shaft, carrying with it the sharp abrasive lactose crystals, resulted in the crystals being lodged in the atomiser assembly causing its malfunction.

7.4.6 *Free moisture in lactose powders*

Dried whey powders typically contain $\sim$2–2.5 g 100 g^{-1} residual-free moisture; due to the strong water binding properties of the whey protein, the residual water does not affect the product quality. However, in the case of crystalline lactose powders, a rigorous control

of the free moisture content in the finished product is extremely important for its keeping quality. The free moisture content in the α-lactose monohydrate powders should not exceed 0.3 g 100 g^{-1} maximum, (0.1–0.2 g 100 g^{-1} for large volume packages) as this water is not bound by the crystalline material. By increasing the a_w of the final product, free moisture will cause excessive mould development. Of course, this free moisture is in excess of the water of crystallisation which the α-lactose monohydrate contains in its crystalline structure. This approximately 4.5 g 100 g^{-1} of tightly bound water is not exerting any vapour pressure and does not affect the a_w of the final product, but with the growth of mould, some of this water will be freed up, thus exacerbating the problem.

7.5 Modified whey-based products and their uses

The principal component of whey is lactose; thus user of dried whey powders must keep in mind the difficulties that lactose may cause for the consumers, especially for those who are lactose intolerant. The lactose intolerance aspects, as well as the technological difficulties of low solubility, crystal formation in concentrated systems and limited sweetness, can be counteracted by hydrolysing the lactose with the β-galactosidase enzyme (Gänzle *et al.*, 2007). However, drying of whey products with hydrolysed lactose is extremely difficult as the two monosaccharides resulting from the hydrolysis – glucose and galactose – cannot be crystallised before the drying. Specialised drying equipment is available to dry whey powder with partially hydrolysed lactose, but because of the drying difficulties, products with hydrolysed lactose are usually marketed as concentrates and syrups.

An alternative approach is to remove a portion of the lactose by crystallisation, resulting in production of partially delactosed products that can be dried relatively easily, with no lactose pre-crystallisation being necessary. These powders can be used in some instances as replacement for skim milk powders as their proximate composition is similar (Table 7.2).

Dried whey and whey products can be used in a variety of dried mixes, including cookie dough, powdered drinks, fat-filled whey powder and ice cream mixes. In many such applications, the insolubility of the whey protein caused by the excessive heat treatment may not be detrimental, or may even be advantageous. Because heat-coagulated whey proteins have a much higher water binding capacity than undenatured, soluble whey proteins, the use of the traditional lactalbumin in pasta dough was shown to produce much better results than using a soluble whey protein powder (Schoppet *et al.*, 1976). In cases where good solubility is required, such as in some powdered drinks containing dried whey, it may be possible to minimise the protein insolubilisation problem by applying a microparticulation treatment (Iordache & Jelen, 2003). When applied on a suspension of heated whey protein, this treatment resulted in resolubilisation of the heat-denatured aggregates and the drying of the treated suspension did not counteract the beneficial effects of the process.

7.6 Future trends

At the present time, dried whey is a valuable commodity and so is the isolated lactose powder, with market prices reaching hitherto-unseen levels. In addition to the high industrial

demand, there are examples of dried whey being marketed in retail, both in pharmacies and in common food supermarkets, to consumers seeking healthful dietary aids or alternatives to the empty-calorie powdered drinks (Jelen, personal observations in the Czech Republic). With its high content of whey proteins that are being shown with increasing regularity to have physiological functionality, whey may indeed become a popular item for the health-conscious consumer. Dried whey is being commonly used in many powdered foods as inexpensive filler or even as a functional ingredient. It is foreseeable that incorporation of suitable nutraceutical products or, possibly, prebiotics into these dried whey products will be forthcoming. The whey components (especially the heated whey protein aggregates) appeared to be suitable for providing protection to probiotic bacteria during the spray drying (Picot & Lacroix, 2004).

With an increased interest in the production of lactose, much additional research is still needed to elucidate many of the factors that hinder lactose production and to maximise the lactose yields. In contrast to the traditional, evaporative, crystallisation from highly purified sugar beet or sugar cane juices used in the production of table sugar, lactose is still crystallized in a slow cooling process. The impurity levels in the concentrated 'juice' (mostly UF milk or whey permeate) are very high in comparison to those used in the table sugar industry. Effects of impurities, especially the minerals, on the formation, crystal growth velocity and shape of the lactose crystals (Jelen & Coulter, 1973; Bhargava & Jelen, 1996) still remain to be explained. While drying of the α-lactose is routine, the crystallisation step is still far from being completely understood and improving its efficiency can be considered one of the most pressing research tasks, especially in the current situation of high demand for lactose. Increasing the yields of the lactose manufacturing operations and developing new technologies for the processing of the mother liquor concentrates is presently of great commercial interest. Industrial production of the various alternative lactose forms (especially the β-lactose) still awaits perfection.

One of the costliest aspects of drying whey and whey products is the need to remove very large amounts of water. At the present time water is usually discarded, especially from the evaporation and drying operations. With water becoming a scarce commodity in many parts of the world, it is not inconceivable that its recovery from the whey may become a principal target of future processing approaches, perhaps necessitating even modifications of the currently used evaporation and drying technologies. It appears that the time is approaching when dried whey and whey-based powders will finally become a fully equal partner in the family of dried dairy products.

7.7 Sources of further information

In addition to some of the chapters elsewhere in this volume and the references cited in the text above, numerous books and other literature sources are available for additional information concerning many aspects of the products and processes discussed here. In particular, the following books and monographs are recommended.

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8 Specialised and Novel Powders

P. Havea, A.J. Baldwin and A.J. Carr

8.1 Introduction

The development of specialised and novel dairy powders has been driven not only by specific market demands but also by a technology push based on better understanding of the background science that underpins various characteristics of dairy products. A significant advance in the development of novel dairy powders has been to meet the needs of global trends in healthy eating and the production of nutraceuticals. The consumer is becoming more health conscious, creating a large market for new products that meet the requirements of the healthy-eating category. To meet the demands of the nutraceutical end of the market, products that are based on isolating intact protein fractions with specific bioactive properties have been developed. Another approach has been to hydrolyse proteins in order to modify the digestion of dairy proteins. Other developments fall into the category of convenience products.

Application of sound science has resulted in the invention of various technologies that make the manufacture of new products possible. This chapter discusses the most recent developments in various dairy powders. These include coffee whitener powders, cream powders, whey protein powders and more specialised powders such as hydrolysates and milk minerals.

8.2 Principles

The powders that are dealt with in this chapter are often quite different in composition and functional properties from standard milk powders. This poses challenges in the formulation, manufacturing process and the storage of these powders. The particular issues that need to be considered are discussed in this section.

8.2.1 *Moisture content*

Stability in storage is a primary requirement of a dried product and is influenced largely by water activity as a key parameter. This is routinely monitored during mass production by measuring the moisture content of the product. Thus, an appropriate moisture specification for each product must be established.

Compared with the moisture content of skimmed milk powder (SMP), the moisture content of low-protein products needs to be lowered, whereas that of high-protein concentration

powders can be raised. When a new product is formulated, the moisture content should be set by establishing the moisture/water activity relationship. The water activity needs to be set in relation to the conditions experienced in transport and in storage in the market. For markets in countries where high ambient temperatures are experienced, the water activity may need to be set lower to cope with temperature variations (Pisecky, 1997).

It should be noted that many quality parameters, such as browning and crystallisation of lactose, are minimised by lower water activities. However, with products containing fat, there is an optimum water activity for minimising the rate of fat oxidation (Labuza, 1968).

8.2.2 *Carbohydrate content*

High-carbohydrate products can be sticky. Key factors are the proportion of carbohydrate in the product and the type of carbohydrate. For carbohydrates, there is a transition between a glassy state and a rubbery state, and in the latter state the carbohydrates are difficult to handle. The sticky point curve defines the parameters for spray drying conditions (Roos, 2002).

If a product has a very high proportion of carbohydrate, it may be desirable to reduce the stickiness by crystallising some of it, as is done in the manufacture of whey powder (refer to Chapter 7).

8.2.3 *High-fat content*

Up to a fat content in the powder of approximately 35 g 100 g^{-1}, the fat is well encapsulated by the other materials in the formulation; above this concentration, the particle surfaces become stickier and the powder more cohesive. Consequently, the powder is more likely to build up deposits in ducts, block cyclones and form powder arches that block hoppers.

High-fat powders may be dried successfully in short runs, but achieving long runs may require considerable experience. Thus, these products may be better suited to smaller plants rather than to dryers aimed at dealing with large quantities of milk without interruption.

Studies of powders with different fat contents and manufactured by spray drying indicate that there is a high concentration of fat on the surface that is more or less independent of the fat content. Hence, laboratory measurements do not demonstrate differences in flowability between whole milk powder (WMP) and high-fat powder (Fitzpatrick *et al*., 2007). In practice, however, more difficulty is experienced in handling high-fat powders in warm or hot air in conveying systems. It seems that fat exudes from the fat-laden particles and, when the particles slide along the surface fatty layers are built up on the surface of equipment.

Some steps that can be taken to mitigate these effects are to ensure that the fat is well homogenised, to keep the walls cool and to ensure that the velocities are kept as low as possible. Secondary drying and cooling should be undertaken with vibrating fluid beds (Pisecky, 1997), and conveying is best done with vibrating conveyors. However, dense phase conveyors may be used.

8.2.4 *Oxidation*

Fat is prone to oxidation and, thus, storage of fat-containing powders requires protection of the product from air. Standard practice is to use gas packing with nitrogen or a mixture

of nitrogen and carbon dioxide. For high-fat powders, special care needs to be taken and the inclusion of antioxidants could be considered to extend the shelflife once the package is opened.

Oxidation increases as the temperature increases. In terms of the shelf life of powders, consideration needs to be given to the ambient conditions in the countries in which the product is marketed.

An additional control method to gas packing is heat treatment in the manufacturing process. The heat treatment is generally applied to the formulated product prior to evaporation (preheating). With WMP, it has been demonstrated that the more severe the heat treatment, the more effective is the antioxidant activity (Baldwin *et al.*, 1991).

8.2.5 *Processing control*

Control of the process should be based on the analysis of 'critical control points' (CCPs). This applies not only to microbiological hazards, but should also encompass other aspects of the product. Critical functional attributes of value to the consumer or the food processor should be identified and controlled with key processing parameters and specific functional tests.

8.2.6 *Particle solubility*

Particle solubility is a complex topic because it depends on a number of factors. With dairy products, the temperature can have a marked effect on the ease of reconstitution. The optimum temperature is often about 50–60°C. Many milk products are slow to hydrate in water at ambient or refrigerator temperatures, and the proteins are liable to coagulate at high temperatures, that is >80°C. Adjustments to the mineral composition or the use of additives can increase stability in reconstitution. The major process step that affects particle solubility is the drying process. The casein proteins in SMPs are particularly vulnerable during drying in the moisture range (wet basis) 40–15 g moisture 100 g^{-1} SMP (Baldwin & Truong, 2007). A higher protein content, with a consequent lower lactose content, exacerbates the problem. The complete dissolution of the powder is important to the functional performance of a product (Damodaran, 1996).

8.3 Coffee whitener powders

Coffee whiteners were developed to satisfy consumers' need for the convenience of a powder while giving a satisfying taste and texture to a hot drink. Important characteristics are the aroma and flavour profile, the creaminess in the mouth and an appealing colour to the eye. Dry products have the advantage over liquids of long shelf life and reduced bulk.

8.3.1 *Chemical composition*

Because of the specific nature of these performance requirements, and the lack of nutritional demands, the composition of a coffee whitener is usually quite different from that of a

Table 8.1 Typical composition (g 100 g^{-1} of full-cream and low-fat coffee whitener powders.

Component	Full cream	Low fat
Fat	34.5	6.9
Protein	2.4	2.5
Carbohydrate	57.6	83.9
Moisture	2.5	2.5

traditional milk powder. The original coffee whiteners were formulated from vegetable oil, dairy protein and carbohydrate. Typical compositions of a full-cream coffee whitener and a low-fat coffee whitener are given in Table 8.1.

Coffee whiteners were originally labelled as 'non-dairy' because of the use of vegetable oil, despite the use of dairy protein. Vegetable oil was used to reduce cost; however, successful products can be formulated from all-dairy ingredients (Innes & Baldwin, 1980). The carbohydrate component can be formulated from glucose syrups, maltodextrins and sucrose, with proportions adjusted to target a specific sweetness level. Protein functions as an emulsifier; sodium caseinate and/or calcium caseinate function extremely well and milk protein concentrate (MPC) may also be effective. The production of sodium caseinate is expensive compared with the costs of manufacture of milk powder. For applications in coffee whitener or imitation cream, low-calcium milk made with a weak cation resin has been proposed as an alternative to sodium caseinate (Manner *et al*., 2007).

Flavourings and colourings are routinely added to coffee creamers to simulate the flavour and colour of liquid milk products.

8.3.2 *Manufacturing process*

The whitener products are generally manufactured by a spray drying process. The ingredients are mixed to give a concentrate and the mixture is homogenised. The colour may be manipulated by the processing conditions. In particular, higher homogenisation pressures can increase the whiteness, as measured by a colorimeter (Kneifel *et al*., 1992). The concentrate is then spray dried. The particle size of the powder can be controlled by taking fines from the dryer chamber or the ancillary fluid bed and recycling them to the atomisation zone to be agglomerated with the atomised concentrate. Because of their low protein content, relatively high-concentrate total solids (TS) (up to 65 g TS 100 g^{-1}) and high inlet air temperatures (up to 240°C) can be used in the manufacture of these products (Pisecky, 1997).

8.3.3 *Functional properties*

To perform satisfactorily, the powder must reconstitute rapidly in hot beverages without leaving powder residues or an unstable emulsion. One property of coffee whiteners that is of importance is stability during reconstitution in hot acidic beverages. Reconstitution to a stable dispersion is the first step in the consumer's perception of the product. Defects in

the reconstitution of products that can be evident are poor dispersion of the powder mass, leading to lumps, and particles that are slow to dissolve. The solubility of the particles is a very important property of the product; if the particles do not dissolve, the full whitening effect will not be achieved. In addition, the undissolved particles either will be evident to the consumer when they float to the surface or will form an unsightly residue if they sink to the bottom of the cup, easily visible when the drink has been consumed. The proteins need to be stable in the acidic environment while still partially dissolved and able to interact with each other.

Stability of the fat emulsion is also important. Any fat released on reconstitution will float to the surface and will form fat lenses that are readily apparent to the consumer.

The whitening power of the reconstituted product is another important attribute. The principles of appearance and colour are given in a number of texts (Francis & Clydesdale, 1975; Hutchings, 1994). The scattering of light depends on the size of the colloidal particles. Maximum reflectance is given at around 100–200 nm, a quarter of the wavelength of light (Francis & Clydesdale, 1975). Some of the natural casein micelles, if present, will be of this size. Homogenisation of the formulated product is a key step as it determines the fat globule size distribution and the whitening ability of the fat emulsion. An investigation of the whiteness of a number of dairy and non-dairy products reconstituted in coffee was carried out by Kneifel *et al*. (1992); these workers found that SMP and dairy-based whiteners gave somewhat higher whiteness figures than non-dairy whiteners.

The enzymatic reactions occurring in the manufacture of tea have given rise to a large number of phenolic compounds; over 500 have been identified (Graham, 1992), which contribute to the flavour and colour. Two principal types of pigments, the orange theaflavins and the brownish red thearubigins, contribute to the black colour of tea (Roberts, 1962). When a whitener is added to tea, the proteins react with the pigment compounds, leading to the creamy colour of milky tea.

Many organic acids and flavour compounds, and pigments, are formed in the coffee roasting process. These compounds, along with caffeine, lead to an acidic and bitter taste. As with tea, the components of the coffee whitener, particularly the protein, will interact with these compounds, leading to a colour in the range from brown to off-white. The change in conformation resulting from thermal denaturation exposes previously unavailable amino acid sequences. These newly exposed amino acid sequences are then susceptible to enzymatic cleavage in the second hydrolysis step.

The product will have a reasonable shelf life of 2 years or so when packed in a moisture-barrier container. However, when in use by the consumer, after multiple exposures to the atmosphere, the product will pick up moisture and will deteriorate slowly; in circumstances of intermittent use, storage in a refrigerator may be desirable.

One important application of coffee whiteners is in vending machines. In most jurisdictions, free-flow agents are permitted for vending machine applications; low (about 0.5 g 100 g^{-1}) concentrations of agents, such as silicon dioxide, will improve the flow in the narrow dispensing apertures and will improve storage stability in a cafeteria environment.

8.3.4 *Recent developments*

The market for coffee and tea whiteners is now very large. The predominant market is North America; Asia/Pacific is another major segment. Originally, from the 1960s to the 1980s,

powdered products were popular because of their convenience. A number of enhancements have been introduced to the coffee creamer market, reflecting the growing sophistication of the coffee drinking culture.

Whiteners may be formulated from carbohydrates to achieve low-fat contents. Flavour and colour components are added to mimic those of the conventional product (Okonogi *et al*., 1986a, b). Whiteners are now also being formulated with microparticulated proteins, which are being developed as fat replacers (Sargent *et al*., 2004a, b; Villagran & Baughman, 2004, 2006).

To improve the flavour stability, flavours and aromas may be incorporated by encapsulation, for example by 'locking' a small amount of flavour in a mixture of saccharide hydrolysate/saccharide, e.g. starch hydrolysate, lactose hydrolysate, cyclodextrin and their mixtures (Okonogi *et al*., 1986a, b).

Commercial companies have undertaken development work to provide products that produce a cappuccino or foaming effect (Stuglik, 1997; Bisperink *et al*., 2001; Zeller *et al*., 2001; Maier & Bachtler, 2002, 2005). As these products are made with foamable gas, the powder has a low bulk density. Products based on carbonate or bicarbonate salts that foam when the carbon-dioxide-producing ingredient dissolves have also been developed (Villagran *et al*., 2000). Another approach is to use a foam-stabilising protein such as egg albumin (Westerbeek *et al*., 1995).

The production of a white coffee powder, a combination of coffee and creamer, is particularly challenging because of the hot and acidic conditions when the instant coffee is reconstituted. These conditions are very detrimental to the stability of the milk proteins. A process to stabilise the whitener involves the incorporation of stabilising salts in the creamer and the application of an ultra high temperature (UHT) treatment to flocculate the whey proteins (Chaveron *et al*., 1997).

8.4 Novel whey products

Whey products, such as whey protein concentrate (WPC) and whey protein isolate (WPI), have come a long way from being a waste, in the 1970s, to being a significant worldwide business today. The world cheese and casein processing produced over 110 billion litres of whey in 2002. Of this volume, 74% was used in manufacturing WPC35 (330 000 tonnes), 17% in manufacturing WPC80 (75 000 tonnes) and 8% in manufacturing WPI (37 000 tonnes) (UBIC Consulting, 2004). The manufacture of WPC35 is a more convenient way of dealing with the large volume of available whey than treating it as a waste, especially in the United States and Europe. Manufacture of WPC80 and WPI is more desirable when the cost of transport is a major consideration, especially for large exporters such as Fonterra in New Zealand. Industrial processes and the manufacture of these products are covered elsewhere in this volume. This section deals only with the latest novel developments in the industrial manufacture of whey protein and other whey products.

8.4.1 *Whey protein in nutraceutical applications*

Much of the standard WPC products have been used as functional ingredients in many food applications (Kinsella & Whitehead, 1989). The most recent developments in whey protein

manufacture have been driven by the market demand for cheaper, more nutritionally valuable proteins. Since the early 1980s, the world consumer market has shifted more towards health consciousness, demanding low calories, low sugar, low fat and/or low cholesterol in diets, and giving rise to a higher demand for 'health' proteins, especially whey protein (UBIC Consulting, 2004). Whey protein is regarded as having high nutritional value, next to that of egg protein. The amino acid profile of whey protein contains higher levels of essential amino acids than that of most proteins (Schaafsma, 2006). It is also known to be easily digested and has excellent metabolic value (i.e. high biological value) (Barth & Behnke, 1997). It has high absorbability by the digestive system and is an attractive protein source among the sports world (Sinha *et al*., 2007). Whey protein is also believed to have a positive effect on cancer prevention (Kent *et al*., 2003), and has been linked to the enhancement of the nervous and body defence systems (Boehm *et al*., 1998; Tseng *et al*., 2006).

These benefits have led to food manufacturers looking at ways of adding more whey proteins to their food products. Research findings of the past two decades have led to the diversification of the functional properties of whey protein, and the invention of various manufacturing processes of novel products that are suitable for a wider range of applications than the traditional uses of standard whey products such as WPI and WPCs. The most significant development in whey protein manufacture is the production of various heat-denatured whey protein products.

8.4.2 *Heat-denatured whey protein*

When heated under appropriate conditions ($>75^{\circ}$C, $\sim$pH 6–8, >6 g TS 100 g^{-1}), whey protein will form gels (Havea *et al*., 1998). The manufacture of denatured whey protein products requires means of heat denaturing the protein in a way that retards or eliminates this gel formation. Heated protein is said to have lost some of its nutritional qualities because of the sensitivity of some of the essential amino acids, and this effect is dependent on the heating conditions (Li-Chan, 1983; Desrosiers & Savoie, 1991). The final product is a WPC paste or powder that can be used as a food ingredient that lacks the typical thickening or gelling characteristics of standard WPC powders. The most common method for making this range of products is the heat treatment of a whey protein solution to denature the protein under shear conditions prior to concentrating (e.g. evaporation) and drying.

Manufacture of lactalbumin (traditional denatured whey protein)

The most common denatured whey protein is known by the name lactalbumin. This is a denatured whey protein with >90 g protein 100 g^{-1}. It has been in the market for more than 50 years (Robinson *et al*., 1976). Lactalbumin is made by heating fresh whey under acidic conditions at $>90^{\circ}$C for a time to allow the protein to denature and form aggregates. The aggregated protein is then recovered and washed via a series of clarifiers. The final slurry is passed through a filter press to form a cake, which is then dried and milled using a ring or attrition dryer. The heated protein is fully denatured and lacks the ability to form gels. The process of making lactalbumin is relatively cumbersome. The heat treatment

conditions are such that most of the α-lactalbumin (α-la) and some other components are not incorporated in the aggregated protein, resulting in low yields. For these reasons, only the New Zealand dairy industry has supplied lactalbumin during the last two decades. Lactalbumin is used as a substrate for making whey protein hydrolysates (WPH). It is used in the protein fortification of processed cheese and other dessert formulations. It is also used as a fat mimetic in products such as yoghurt and ice cream.

Microparticulated whey protein (MWP)

The most recent novel whey protein powders are manufactured using a process referred to as microparticulation, in which a protein concentrate is heated under high shear conditions. The heat induces protein denaturation and aggregation and the shear breaks up the aggregated protein to obtain fine particles. The microparticulation conditions are well controlled so that the denatured whey protein is restricted to a certain degree of aggregation, resulting in the formation of specific particle size ranges. Steventon *et al*. (1994) have reported that the properties of the aggregate result from a dynamic balance between shear-controlled aggregate growth and shear-controlled aggregate break-up.

Several microparticulation processes have been used for the manufacture of denatured whey proteins. Singer *et al*. (1988) have disclosed a process (Simplesse) in which the protein is heated under high shear in a special shear apparatus. This system has been used to manufacture a number of MWPs such as Simplesse 100 and Simplesse 300. Other processes that have been employed to make MWPs include the use of a scraped surface heat exchanger (SSHE) (Spiegel & Kessler, 1998; Spiegel, 1999), homogenisation of a heated (for example, by direct steam injection) whey protein solution (Paquin *et al*., 1993; Holst *et al*., 1996) and extrusion (Queguiner *et al*., 1992).

Microparticulated whey protein products consist of very small, smooth, round particles that have the same effect on the sense of touch as an oil-in-water emulsion. These products are used to replace fat in low-fat ice cream, yoghurt (see Tamime & Robinson, 2007) and desserts. As they impart a mouthfeel that is similar to that of fat, ice creams or desserts will taste as if they contain similar levels of fat to those in the full-cream versions. Lucca and Tepper (1994) have suggested that whey protein aggregates in the size range from 0.1 to 3 μm impart a creamy mouthfeel. Aggregates >3 μm impart a powdery to gritty mouthfeel. It has also been reported that whey protein particles >10 μm can still impart a smooth creamy mouthfeel if they are compressible (Cheftel & Dumay, 1993; Huyghebaert *et al*., 1996). It appears that, in addition to the particle size, the nature of the protein aggregates is also important in delivering the desired mouthfeel.

It is a major challenge to make a denatured whey protein that delivers the specific functional properties. The heating and shear conditions need to be tightly controlled because there is a fine line between producing a denatured whey protein powder that delivers the required smooth mouthfeel and producing a powder that imparts a sandy or gritty mouthfeel when used as a food ingredient. Many studies (Dannenberg & Kessler, 1988; Spiegel, 1999; Spiegel & Huss, 2002) have demonstrated that the properties of the aggregate are closely correlated with the denaturation rate that is predominantly affected by the heating temperature. There are two distinct temperature ranges, corresponding to two different activation energies, i.e. straight lines with different slopes in an Arrhenius plot (Fig. 8.1, Spiegel,

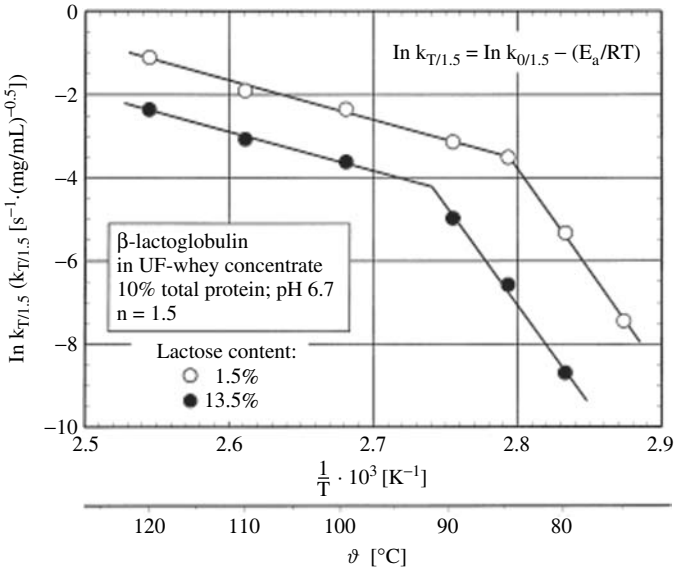

Fig. 8.1 Arrhenius plot for the rate constant $k_{T/1.5}$ of the denaturation of β-lactoglobulin in whey protein concentrates (WPCs). Reprinted with permission from Spiegel (1999).

1999). The temperature ranges may be different for different proteins (Dannenberg & Kessler, 1988). The temperature at which the straight lines of both ranges intersect is described as the transition temperature. Although the activation energies in these ranges do not change, the temperature ranges may be affected by other factors, such as the presence of lactose (Spiegel, 1999). It was reported that the unfolding of the protein molecules was the rate-determining reaction (400 kJ mol^{-1}) in the lower temperature range and that the aggregation of the unfolded protein was the rate-determining reaction (80 kJ mol^{-1}) in the higher temperature range. Depending on the heating temperatures, protein aggregates with different properties could be produced. These relationships are depicted in Figures 8.2 and 8.3 (Spiegel, 1999). With the knowledge of the composition of a protein solution, it should be possible to determine the temperature range in which heat treatment should be carried out in order to obtain a denatured whey protein product with the desired functional properties.

8.4.3 *Cold gelling WPCs*

A number of processes have been established for the manufacture of whey protein powders with the ability to form gel networks at ambient temperature on acidification or the addition of salt. There are a number of food applications in which such a product would offer an advantage, especially in those where no heat treatment is required. In these processes, the whey proteins need to withstand heating without forming gel networks. The key conditions for making these products include a whey protein solution with low ionic strength and a low-protein concentration (<10 g 100 g^{-1}), a relatively low heating temperature (~70–90°C) and a high pH (~7–9) (Gao *et al.*, 2006). Fresh whey is ultrafiltered (UF)/diafiltered (DF)

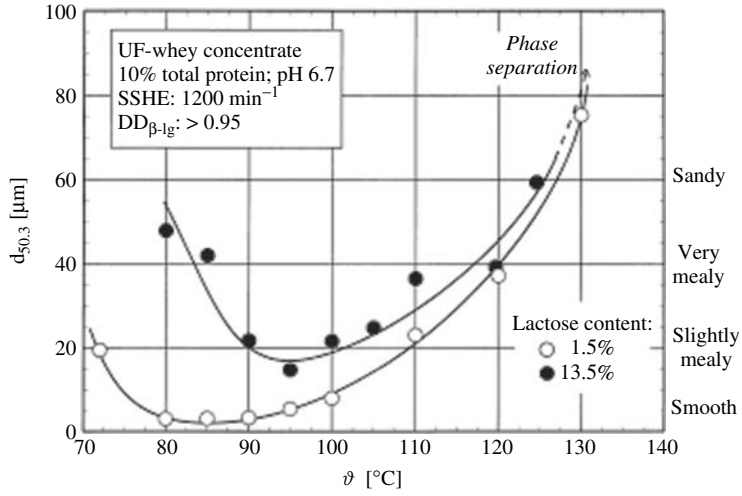

Fig. 8.2 Effect of the heating temperature on the aggregate size of heat- and shear-treated whey protein concentrates (WPCs). Reprinted with permission from Spiegel (1999).

to remove minerals and lactose and to obtain a protein concentrate. The concentrate can be further purified by passing it through an ion exchange column. The purified protein is then diluted (<10 g TS 100 g^{-1}), pH adjusted and spray dried. Most of the patented processes are slight modifications of these key steps. Detailed descriptions of these processes can be found in a number of patents, including Phillips and Evans (1981), Hudson *et al*. (2001) and Gao *et al*. (2006).

The WPCs produced from these processes have been reported to improve the consistency of Surimi and cold-set desserts, and have potential for use in acidified dairy products and calcium-fortified milk-based beverages (Keogh, 1998; Alting *et al*., 2003, 2004). There is no report of the commercialisation of any of these processes, which may be due to economics. Processing a cold-set WPC powder has a number of economic challenges. The heat treatment and spray drying are carried out at relatively low-protein concentrations. The benefits for these products do not justify the costs of production.

8.4.4 *Co-precipitation of whey protein with casein*

Incorporating whey protein into cheese is an economic way of using whey if the cost of whey protein is lower than that of casein. Many casein-rich products, such as milk powders and MPCs, are used as ingredients in cheesemaking. When these powders are used in cheese, the whey proteins are usually washed away with the cheese whey and are not incorporated into the cheese. Most of the recent developments have focused on promoting interactions between the casein and the whey protein, mainly by heat treatment under specified conditions, thereby incorporating the whey proteins in the cheese.

Blazey *et al*. (2000) have disclosed a process in which a milk preparation that may have added whey protein is heat treated at an elevated pH to allow co-precipitation of whey protein and casein. The milk preparation is then spray dried and is used as an ingredient

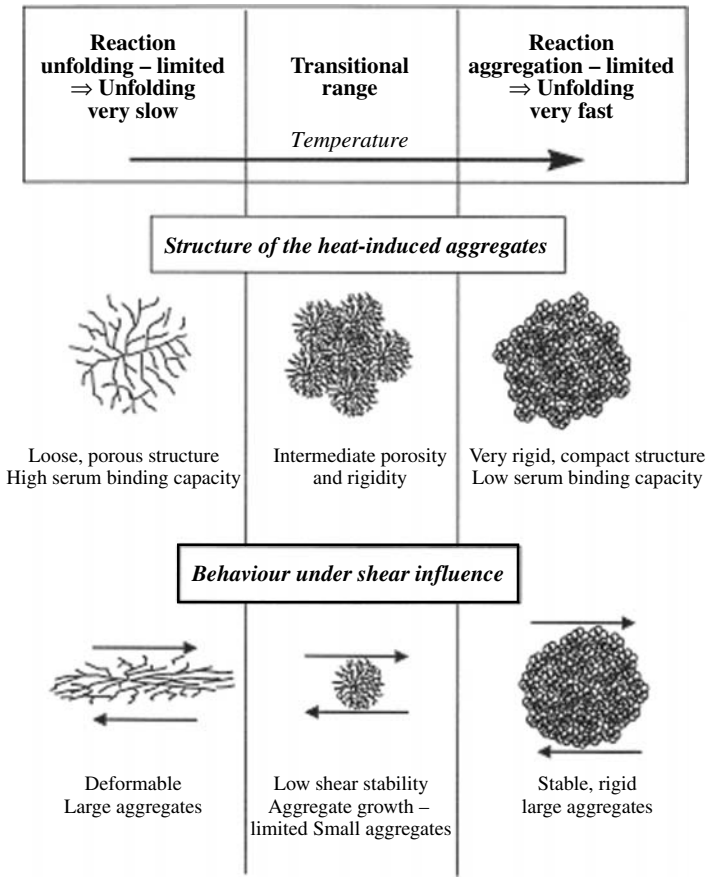

Fig. 8.3 Model of whey protein aggregation under shear influence. Reprinted with permission from Spiegel (1999).

in cheesemaking. This product is reported to increase the yield of the cheese. Bhaskar *et al*. (2004) have disclosed a process for making an MPC powder in which the whey protein is heat denatured. In this process, skimmed milk is ultrafiltered to obtain a milk retentate. This retentate is treated in an ion exchange column, which modifies the levels of calcium in the system. The product is then heat treated to denature the whey protein, which then aggregates with the casein. The heated solution is then evaporated and spray dried. This final product is reported to have an excellent cold solubility and to enhance yields in cheesemaking. Other similar or modified processes include those of Sueyasu *et al*. (1994), Yee *et al*. (1994), Onwulata (2005) and Anema and Lee (2006).

8.5 Milk mineral

The demand for calcium-rich ingredients/supplements is growing worldwide because of the many health issues that are associated with the calcium-deficient diets that accompany the

Table 8.2 Compositional data (g 100 g^{-1}) for a commercial milk mineral product (LactoCalcium).

Component	Manufacturer specifications
Total mineral	80.2
Calcium	24.0
Phosphorus	13.5
Magnesium	1.5
Iron	0.1
Lactose	10.0
Protein	5.0
Moisture	4.0
Fat	0.5

Adapted from Allen & Cornforth (2007).

fast pace of modern lifestyles. Calcium is the principal mineral in the human body, with 99 g 100 g^{-1} found in the bone, making it the most important mineral in maintaining bone health (Recker, 1993). Various calcium supplements are commercially available, including calcium carbonate, calcium citrate, coral calcium and milk mineral. Milk mineral powders, a by-product of the production of WPC, are becoming more common in the market. They are fine, gritty, white powders with varied particle sizes, depending on the manufacturing process, and contain around 80 g 100 g^{-1} mineral (Table 8.2, Allen & Cornforth, 2007).

Calcium exists in milk in the form of micellar calcium phosphate and, in this form, it is reported to be an excellent source of calcium with high absorption and bioavailability (Wong & LaCroix, 1980; Kansal & Chaudhary, 1982). Micellar calcium phosphate binds to the phosphate groups of caseins and cross-links the casein molecules through their phosphate groups (Aoki *et al*., 1987). The calcium and the inorganic phosphate in micellar calcium phosphate are in quasi-equilibrium with those in the soluble phase (Kato *et al*., 2002). In WPC manufacture, the permeate contains mainly lactose and milk mineral that consists predominantly of soluble calcium phosphate. Under appropriate conditions of pH and temperature, the milk mineral can be precipitated and then separated from the lactose, concentrated and dried. Various methods for the industrial manufacture of milk mineral have been disclosed (Sasahara *et al*., 1988; Nakagawa & Tanaka, 1990, 1993; Shigematsu *et al*., 2000). LactoCalcium is an example of commercially available milk mineral. It has been reported to readily release calcium by enzymatic digestion and is bioactive in stimulating bone formation in human osteoblasts (Rao *et al*., 2007).

In the industrial manufacture of milk mineral, the precipitation method changes the form of the calcium phosphate from amorphous calcium phosphate to a crystal such as hydroxyapatite (Aoki *et al*., 1987). In a separate study, Aoki *et al*. (1998) reported a method for the preparation of milk calcium phosphate from rennet casein as a complex with phosphopeptide. The phosphopeptide and the phosphate groups of casein were believed to stabilise the amorphous calcium and to prevent the formation of hydroxyapatite. Using X-ray diffraction and high-performance liquid chromatography (HPLC) methods, it was confirmed that

calcium phosphate in the micellar calcium phosphate–phosphopeptide complex was similar to that in casein micelles. It was also reported that the bioavailability of calcium in this form was higher than that of the hydroxyapatite form (Toba *et al*., 1999).

Milk mineral is used as a calcium source in many foods designed for bone health and children. It is used in bakery products such as biscuits, yoghurt, ice cream and other desserts. It is a preferred source of minerals, largely because of its origin and its 'natural' image. It has been suggested to have positive effects on traits of the metabolic syndrome, mostly on weight reduction and fat loss (Scholz-Ahrens & Schrezenmeir, 2006).

8.6 Cheese powder

The starting material is blocks of cheese; often, aged cheese is used to impart flavour to the product. The ingredients are assembled in a jacketed vat with strong agitation and liquid recycle. Diced cheese is added to hot water (50–60°C) to produce a slurry of total solids ~40 g 100 g^{-1}. Stabilising salts, such as sodium citrate and phosphates (e.g. disodium phosphate), are added at a concentration of about 2 g 100 g^{-1}. Minor ingredients, such as salt, flavours and colour, are added. The mixture is homogenised (~15 MPa), heated to at least 75°C for microbiological control and then spray dried. The inlet air temperature needs to be kept low and the product is discharged through a cooling bed.

The main difficulties with processing cheese powder are the materials handling in the feed formulation step and the formation of deposits in the spray dryer chamber and collection system. Wall sweeps may be an advantage, or a small dryer that can be cleaned easily may be used.

The composition (in g 100 g^{-1}) of cheese powder is typically 50 fat, 40 protein, 4 mineral, 3 carbohydrate and 3 moisture (Pisecky, 1997). Key attributes are flavour and colour. As some volatile flavour compounds may be lost in the spray drying process, ingredients are often added to boost the flavour; one such useful material is yeast extract. There are also often specifications for colour. Colour matching can be done by comparing the coloured concentrate with specification powder reconstituted to the same TS.

Cheese powder is used primarily to add flavour to baked goods, biscuits or snacks such as potato crisps. It can also be used in dip mixtures and similar liquid products (see also LiShui *et al*., 2007).

8.7 Hydrolysates

This section focuses primarily on novel products manufactured using enzymatic hydrolysis. Details on fermentation-based hydrolysate processing and products can be found in Farnworth (2003). Hydrolysates are often broadly classified in terms of the percentage of peptide bonds within the protein that have been hydrolysed, referred to as the degree of hydrolysis (DH). Products may be described as having a low, moderate or high DH. In addition, hydrolysates with a high DH are sometimes termed 'extensively hydrolysed', and are more challenging to manufacture. The essential steps involved in hydrolysing dairy proteins (Fig. 8.4) are the same regardless of the DH.

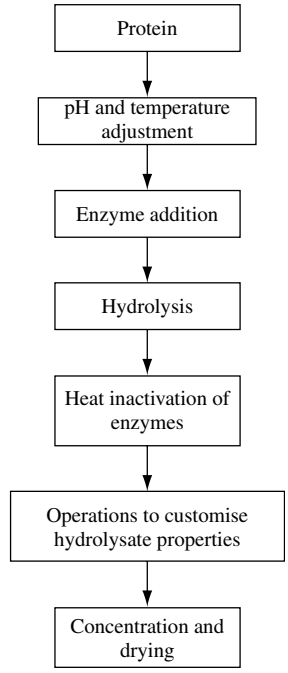

Fig. 8.4 General process for the manufacture of hydrolysates.

Although extensive hydrolysates of casein have been in use for oral, parenteral, infant and intravenous preparations since the 1940s (Shohl & Blackfan, 1940), extensively hydrolysed whey protein ingredients are a relatively recent development. The development of whey protein hydrolysates (WHPs) was hindered by the lack of availability of commercial supplies of purified whey before the 1970s. Unlike the development of casein-based hydrolysates, the development of extensively hydrolysed whey products has required overcoming problems associated with the globular structure of whey proteins, which sterically limits the access of enzymes to the entire amino acid sequence.

Most extensively hydrolysed protein products are used in hypoallergenic formulations. For a protein product to be labelled hypoallergenic, it must not possess any peptides that are recognised by the body as allergens. A general rule of thumb is that peptides must be no greater than about 5–10 kDa in size. Peptides that are larger than this range are referred to as *macropeptides*. To produce an acceptable hypoallergenic product, it is common to customise the molecular weight profile by using UF to remove macropeptides from the final hydrolysate (Olofsson *et al*., 1981). The introduction of a UF step limits process throughput and yield. These researchers demonstrated that the process throughput during UF could be improved by thermally denaturing the heat-labile macropeptide fragments prior to UF. By increasing the temperature–time profile of the heat treatment from 90°C/5 s to 98°C/30 min, they observed an increase in flux from 15 to 60 L m^{-2} h^{-1} during UF (Olofsson *et al*., 1981).

The key to minimising yield losses resulting from the removal of the macropeptides during UF is to manipulate the whey protein to allow a more complete hydrolysis. Improving

the overall efficiency of the hydrolysis is complex and depends on the desired characteristics of the hydrolysate, enzyme selection, protein substrate, substrate concentration, hydrolysis temperature, pH process control throughout the hydrolysis, ionic strength, mineral composition and pre-hydrolysis protein treatments that modify the protein conformation (Klostermeyer *et al*., 1975; Jost & Monti, 1977; Adler-Nissen, 1986, 1993; Camacho *et al*., 1998).

There are a few processes that claim not to require UF to produce a hypoallergenic WPH. Jost *et al*. (1991) disclosed a method that produces a WPH with a molecular weight cut-off of 10 kDa, using pancreatic enzymes, without the need for UF, through the use of a two-step hydrolysis coupled with an intermediate thermal denaturation step. The initial hydrolysis allows the partially hydrolysed whey protein substrate to be thermally denatured without gel formation. This enables further hydrolysis of the denatured substrate.

Sado *et al*. (1992) adopted a similar approach by having a two-step hydrolysis process. However, after an initial hydrolysis, they increased the temperature of the partially hydrolysed substrate beyond the initial denaturation point of the whey proteins and then completed the hydrolysis through the selection of thermo-stable enzymes. This method is reported to have the advantage of eliminating the need for the energy-intensive intermediate heating and cooling step required by Jost *et al*. (1991).

As a protein is hydrolysed, there is a concomitant release of one free amino group and one free carboxyl group, which, depending on the system used, will result in a net shift in pH. To maintain the pH of the system in the range that is optimum for the enzymes, it is usual to add either a base or an acid. As the DH increases, the concentration of acid or base increases and the resulting salt may need to be removed from the product before drying. Thus, in practice, it is likely that, for an extensively hydrolysed protein product, a UF/nanofiltration (NF) step may still be desirable to remove excess salt.

UF, in addition to its use in the manufacture of extensively hydrolysed protein products to ensure hypoallergenicity, has also been used to customise the molecular weight profile of moderate- and low-DH hydrolysates (Samuelsson & Poulsen, 1991) and to purify bioactive peptides (Davis *et al*., 2003). Optimisation of hydrolysis for the purpose of manufacturing bioactive peptides generally requires a lower DH than that required for hypoallergenic applications. Excessive hydrolysis can result in yield losses through hydrolysis of the peptides of interest (Schlothauer *et al*., 2006).

Historically, the main driver in enzyme selection in extensive hydrolysates has been to achieve a hypoallergenic product. Although hypoallergenicity may be an important characteristic for at-risk infants, new research indicates that the role of infant formula is more than merely a vehicle for the supply of protein (Moya, 1993). In particular, a German Infant Nutritional Intervention (GINI) study (von Berg *et al*., 2003) showed that the hydrolysed infant formula consumed in the first 4 months of life can act as a preventative for the manifestation of some allergies at the age of 1 year. It was also noted that extensively hydrolysed whey-protein-based and casein-based formulae had different effects on tolerogenicity. Fritsche *et al*. (2004) disclosed a method for preparing a hypoallergenic extensively hydrolysed composition containing an efficacious concentration of tolerogenic peptides.

In recent years, there has been an increased recognition, initially stemming from research into probiotic bacteria, that, upon hydrolysis, physiologically active peptides that can

have a range of beneficial effects, including opiate, antithrombotic, anti-hypertensive, immunomodulating, antibacterial, antidiarrhoeal, antigastric, satiety inducing, skincare, anti-cariogenic and mineral carrying properties, are released from milk proteins. A review of the biologically active peptides that are released from milk can be found in Matar *et al.* (2003).

In general, the manufacture of ingredients containing bioactive peptides involves the following steps:

- Pre-treatment of the substrate
- Hydrolysis with a selection of specific enzymes or probiotics
- Purification of the bioactive
- Drying

The ingredient can then be added to an appropriately formulated product for consumption. In some cases, the isolated peptide must be reacted with another component in order to realise the bioactive state. The commercial anti-cariogenic product Recaldent™ requires the hydrolysis of casein to obtain a solution of a phosphopeptide containing the amino acid sequence Ser(P)-Ser(P)-Ser(P)-Glu-Glu. The phosphopeptide must then be mixed with calcium ions and inorganic phosphate, under alkaline conditions, to obtain a mixture that is isolated and then dried (Reynolds, 2004). It is only when the phosphopeptide exists as an amorphous calcium phosphopeptide complex that the product has anti-cariogenic properties.

An additional consideration when hydrolysing proteins is the impact of the hydrolysis on the organoleptic properties of the end application. Bitterness of protein hydrolysates is complex and readers are referred to Adler-Nissen (1993) for a detailed review. Roy (1992, 1997) discussed general methods of flavour improvement in hydrolysates. Schlothauer *et al.* (2004) provided a solution to the flavour problems, commonly associated with the production of bioactive peptides from whey protein, by selecting heat-labile enzymes that release the desired bioactive peptide, in this case angiotensin-converting enzyme inhibitors, at low DHs. This approach allows the hydrolysis reaction to be terminated before excessive concentrations of bitter and brothy peptides are produced. Additionally, through choosing heat-labile enzymes, Schlothauer *et al.* (2004) were able to inactivate the enzymes under mild temperature–time conditions, and thus avoid the aggregation and gelation of the intact whey proteins and macropeptides that occur under standard enzyme inactivation conditions.

Silcock *et al.* (2004) reported an interesting advance in the flavour management of bioactive peptides and found that the bitterness of an anti-cariogenic casein hydrolysate could be significantly improved by cross-linking with transglutaminase without any loss in efficacy. Other workers in the field have found that desirable flavours can be obtained by manipulating the casein and whey protein hydrolysis conditions (Schlothauer *et al.*, 2004). With the increasing drive for better nutrition, hydrolysis is also proving to be a valuable tool in allowing formulators to increase protein levels in foods without loss in organoleptic acceptability. A recent example of this is the use of hydrolysates in high-protein bars by Paulsen *et al.* (2004), who reported that the hardening of the bars on storage can be significantly reduced by replacing intact protein with hydrolysed protein.

8.8 Cream powders

Cream powders refer to a group of milk powder products containing fat at higher concentrations than those of WMPs. The focus of this section is restricted to the technological aspects of milk fat powders and their manufacture. There is a vast knowledge in this area, and readers are referred to excellent reviews by Dickinson (1997), Rousseau (2000), Robins *et al*. (2002), van Aken *et al*. (2003) and Vega and Roos (2006).

8.8.1 *Why dried cream powders?*

Although the concept of cream powders existed even before the 1960s (Hall & Hedrick, 1966), research and development into their industrial manufacture became more significant during the 1980s when there was surplus milk fat and depressed milk fat prices worldwide [International Dairy Federation (IDF, 1990)]. The industrial production of milk fat products (including butter) has been experiencing a long-term decline on the world scale, but especially in Europe (IDF, 2006). This is largely due to the increasing use of liquid milks for the production of liquid dairy products, cheese and WMP. There is also a worldwide shift in consumer patterns, with people moving away from fat consumption for health reasons. Because of these trends, much of the development has been more of a technology push rather than a market pull. The development of processes to manufacture cream powders had to deal with key challenges. These included the following:

- The need to be able to dry the fat in such a way that it would have a longer shelf life. This required some means of preventing oxidation and the development of off-flavours during storage.
- The technological challenges, such as how to successfully dry the fat because fat is known to stick to the dryer and powder handling equipment.
- The need to retain the functional attributes of the fat after going through the process. When food manufacturers purchase the dried fat product, the cream/fat should be able to deliver the required functional properties.
- There was also cost pressure to manufacture the cream powders in a way that offered consumers benefits in a surplus market. This had to be a major factor when drying high-fat powders was considered.

Milk fat products are easily perishable. They are readily susceptible to oxidation and rancidity. Upon refrigeration, they last for only weeks or a few months at the most. As preservation by freezing is expensive, drying milk fat presents a more viable option (Onwulata *et al*., 1996).

8.8.2 *Emulsion stability*

The drying of fats and oils starts with a stable emulsion (Dickinson & Stainsby, 1982), such as the dispersion of fat droplets in water in the case of dairy creams. Stable emulsions are emulsions in which there are no observable changes in their droplet size distribution or their state of aggregation or their spatial arrangement within a sample over a

time scale of observation (Dickinson, 1994). They are formed by encapsulating them with functional encapsulants, such as milk proteins. When a mixture of fat and protein in water is homogenised to a sufficient degree, the fat is broken down to very fine droplets. The stability and the state of aggregation of these fat droplets depend on the ability of the protein to adsorb to the surface of the fat droplets. This depends on many factors, such as the surface covered by the protein, the surface layer thickness and other aqueous conditions, for example pH, ionic strength and mineral profile (Vega & Roos, 2006). Instability of an emulsion is manifested by creaming, that is when the emulsion undergoes phase separation (Robins *et al*., 2002).

8.8.3 *Processing of cream powders*

Processes vary considerably for each powder depending on the target use. However, these processes are modifications of the general typical process given in Figure 8.5, which was obtained from Hansen (1980), Kieseker *et al*. (1979a, b) and Patel *et al*. (1987). The cream is separated from whole milk, pasteurised and then standardised. Standardisation is a process of altering the composition (e.g. by adding skimmed milk) so that the final composition suits the target uses. The cream is concentrated using, for example, centrifugation or evaporation.

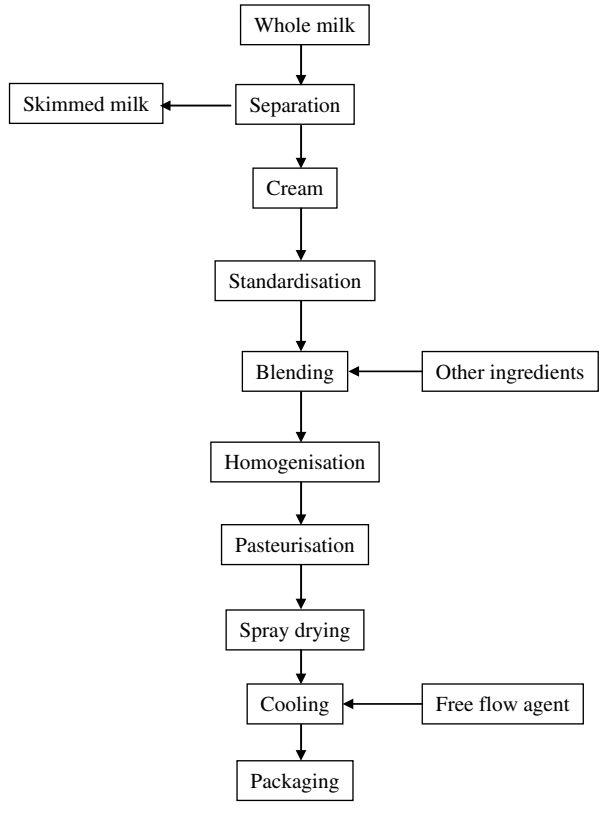

Fig. 8.5 Typical process for making cream powders.

The standardised cream is then blended with other ingredients. These ingredients serve not only to provide the right levels of fat in the final product but also, more importantly, to emulsify and stabilise the fat. The most commonly used emulsifiers include caseinates, whey proteins, MPCs and SMPs. The choice of which emulsifier to use depends on considerations including the ability to stabilise the fat and flavour. Other ingredients that might be added include antioxidants and salts. When cream powders are for specific target uses, other ingredients can be added so that the cream powders provide not only the fat but also other ingredients for that application, for example cream powder for ice cream having all the ingredients, such as sugar or corn syrup, corn starch and egg yolk, included. This blending process is usually done at around 40–60°C. The mixture can be homogenised at 20–50 MPa and 45–65°C, and then pasteurised, by heating at 72°C for 60 s. The mixture is then spray dried, cooled, packed and stored. Packaging is an important consideration because of the need to enhance the shelf life of the product. Oxygen-impermeable plastics and nitrogen flushing of bags are used to exclude oxygen from the product, thus limiting the oxidation of fat.

Holsinger *et al*. (2000) estimated that the manufacture of a milk powder containing 50 g 100 g^{-1} fat using a plant that could process 57 tonnes per day would cost ~US$0.23 kg^{-1} plus the cost of the fat and ingredients. This study was based on encapsulating milk fat with all-purpose flour, modified corn starch or sucrose. They also suggested that the cost of processing was about 25% of the value of the product and that encapsulation offers market opportunities for the processing of fat.

The industrial production of cream powders has been significantly advanced in recent years, resulting in large quantities of milk fat being traded internationally in the form of cream powders. The current research and development focus is on more advanced and efficient processing (Lin & Chen, 2007).

8.8.4 *Physicochemical properties of dairy cream powders*

Cream powders need to have good physicochemical properties, including flowability, wettability and resistance to caking and rancidity. The flowability of powders is affected by several factors including particle size, density, shape and composition (Prasad & Gupta, 1983). Recent work showed that flowability is affected mainly by the surface composition of the powder particles. Fäldt *et al*. (1993) developed a method known as *electron spectroscopy for chemical analysis* (*ESCA*) that enables scientists to measure the fat content of the powder particle surface. Using this technique, it was reported that the ability of whey protein to encapsulate soyabean oil was rather low compared with that of sodium caseinate, with a large part of the powder surface covered by fat after spray drying (Fäldt & Bergenståhl, 1996a). After storage for 4 days under 75% relative humidity (RH), this powder released fat on to the surface, resulting in changes in structure and particle agglomeration (caking). When the emulsion was dried in the presence of up to 25 g 100 g^{-1} lactose, the powder did not show any oil release or changes in structure. In another study, Fäldt and Bergenståhl (1996b) reported that, in the presence of lactose, the whey-protein-stabilised emulsions remained intact in the powder matrix during drying and that the emulsion droplet size distribution of the redispersed spray dried emulsion was unchanged. In the absence of lactose, there was an increase in the droplet size in the redispersed, spray

Table 8.3 Bulk and surface composition (g 100 g^{-1}) of industrial spray dried powders estimated by electron spectroscopy for chemical analysis (ESCA).

	Bulk composition			Surface composition		
Powder product	Lactose	Protein	Fat	Lactose	Protein	Fat
Skimmed milk	58	41	1	36	46	18
Whole milk	40	31	29	2	–	98
Cream	13	12	75	1	–	99
Whey protein concentrate	8	86	6	6	41	53

Adapted from Kim *et al*. (2005a), reproduced with permission.
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dried, whey-protein-stabilised emulsion powders, showing that there was coalescence of emulsion droplets during drying or the redispersion process.

Kim *et al*. (2005a) recently reported that, although there are several factors that affect the flowability of dairy powders, it is influenced strongly by the surface composition of the powders. The presence of free fat on their surfaces is critical in determining the flowability of dairy powders. In the same study, they also showed (Table 8.3) that the surface fat content of the powder particles was much higher than the total fat content of the bulk powders.

The process of the reconstitution of powder in water has been reported to involve four steps: (a) wetting, (b) submerging, (c) dispersing and (d) dissolving (Freudig *et al*., 1999; Kim *et al*., 2002); wetting is the rate-determining step. The surface composition of powders plays an important role in the wetting process. It appears that some components can migrate preferentially to the surface of the powder during or after drying, altering the wettability of the powders dramatically. The presence of fat renders the surface hydrophobic and affects wettability adversely. In another study, (Kim *et al*., 2005b) reported that high melting triglyceride species present in the free-fat fraction accumulated more on the surface of the powder, again adversely impacting on the wetting properties of the powder. Industrial spray dried cream and WMPs could not be completely wetted in cold water ($\sim$10–37°C). The wetting time decreased sharply at 42°C for cream powder and at 38°C for WMP.

It is clear that the surface composition of the powders affects their physicochemical properties. The difficulties with reconstitution can be overcome by reconstitution in warm water or by using coating technologies (Baldwin & Sanderson, 1973). Although there is already a wealth of information and knowledge on how the surface composition of powders dictates the reconstitution properties, the challenge that remains for the food industry is how to apply powder manufacturing coating and packaging technologies to achieve not only a long shelf life, but also good reconstitution properties.

8.9 Concluding remarks

A number of technology innovations have resulted in the introduction of new specialised dried dairy products to the market in recent years. These powders have been driven by

the need to meet specific market demands. In the development of innovative products, the manufacturer needs to consider key manufacturing principles, to ensure that the new products deliver their functional and nutritional requirements at the time of manufacture and during subsequent distribution in the supply chain to the consumer. But doing so also poses key technological challenges; continuing scientific research provides sound knowledge that helps manufacturers overcome the challenges, and in turn moves the whole dairy industry forward. This cycle will continue to expand the dairy category in the future of the world's food markets.

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9 Infant Formulae–Powders and Liquids

D.-H. Montagne, P. Van Dael, M. Skanderby
and W. Hugelshofer

9.1 Introduction

Infant formulae are designed as a substitute for breast milk for infants, who cannot be breastfed or whose mothers decide not to breastfeed (WHO, 1986). The essential composition of infant formulae has to meet the particular nutritional requirements to enable normal growth and development of the infants for whom they are intended. Breastfeeding (Fig. 9.1) is nevertheless the ideal form of infant feeding during early life as multiple benefits have been associated with breastfeeding for child health (Koletzko *et al*., 2000).

As a consequence, breastfeeding is recommended and actively promoted at international level. The World Health Organisation (WHO) has recommended that pregnant women and mothers with newly born babies be informed of the benefits and superiority of breastfeeding. Within this context, it has issued the International Code of Marketing of Breast-Milk Substitutes, which has been adopted by the World Health Assembly in a resolution, and was published nearly three decades ago (WHO, 1981).

If the decision to formula feed is taken by the health-care professional and the mother of the newborn, it is important to give instructions on correct method(s) of preparation, emphasising that unboiled water, unsterilised bottles, incorrect dilution and inappropriate storage after reconstitution can lead to malnutrition and illness to the baby.

Data on the essential composition of human milk provide guidance for the composition of infant formulae, but compositional similarity should not be the only indicator for the safety and nutritional adequacy of infant formulae. Indeed, the nutritional adequacy of an infant formula should be confirmed by comparing, during the first months of life, the physiological (e.g. growth patterns), biochemical (e.g. plasma markers) and functional (e.g. immune responses) outcomes of formula-fed infants with those of exclusively breastfed infants (Koletzko *et al*., 2005). The present chapter gives a general overview of infant formulae from the aspects of history, regulations, classification, composition and production.

9.2 Historical background

Substitutes or complements to breast milk are as old as mankind, and have been used when no or insufficient human milk is available. Ideally, the substitute is the milk of another mother, but most often milk from ruminants, for example cow (Fig. 9.2), has served as substitute. In the historical Roman legend, wolf's milk was used as a breast milk substitute (Fig. 9.3). The development of nutritionally elaborated infant formulae is rather recent and dates back to the late nineteenth century. It has been stimulated by the discoveries in

Fig. 9.1 Internationally accepted logo for breast feeding.

Fig. 9.2 A view of a dairy cow.

Fig. 9.3 Historical Roman legend where a wolf breast feeds Romulus and Remus.

Fig. 9.4 Soya bean plant.

biology and medicine providing a scientific basis for the development of substitutes for human milk, and by the technological progress enabling the access to new food processing methods and ingredients (Packard, 1982; Jost, 2007; Anonymous, 2003; Boehm *et al*., 2007). Some of the important developments are briefly summarised as follows:

- Heat treatment of the milk and aseptic filling in order to make it shelf stable in liquid form (i.e. Ready-To-Feed or concentrated).
- Dehydration of the milk or liquid baby formula by spray drying extends the shelf life and stability of the product.
- Adaptation of cow's milk in order to improve or adjust its composition to meet the nutritional needs of infants (e.g. modification of the whey-to-casein ratio, replacement of the milk fat by vegetable oil mixes, and supplementation in vitamins and minerals).
- Utilisation of alternative protein sources, such as isolated soya protein (Fig. 9.4).

9.3 Definition and classification of infant formula

Infant formula is often used as a generic name to cover a broader range of formulae consumed during infancy and childhood (Table 9.1). However, infant formulae are defined as the sole source of nutrition intended for particular nutritional use by infants during

Table 9.1 Legal definition and classification of the various forms of infant nutrition.

Classification	Definition/age of the baby
Infant formula	Breast-milk substitute manufactured to satisfy, by itself, the nutritional requirements of infants during the first months of life up to the introduction of appropriate complementary feeding. From 0 up to 12 months of age
Follow-on formula	A food intended for the particular nutritional use by infants when appropriate complementary feeding is introduced and constituting the principal liquid element in a progressively diversified diet as well as by young children as a liquid part of the weaning diet. From 6 till 12/36 months of age[a]
Infant cereals	Processed cereal-based foods intended for feeding infants as a complementary food generally from the age of 6 months onwards. From 6 months till 36 months of age[a]
Meals and drinks[b]	Foods intended primarily for use during the normal infant's weaning period and also for the progressive adaptation of infants and children to ordinary food. From 6 months till 36 months of age[a]
Growing-up formula[c]	A food intended for use as a liquid part of the weaning diet, when breast feeding has stopped. From 10/12 months of age onwards

[a]Generally it is recommended to breastfeed exclusively for the first 6 months and introduce solid food from 6 months onwards, but there are presently some debates within the scientific community that the earlier introduction of complementary food may be beneficial (Agostoni *et al*., 2008).
[b]The term 'meals and drinks' is often used, but there is no globally recognised legal definition of this term; baby foods is often used as an alternative term for this category of infant product.
[c]Although the term 'growing-up formula or growing-up milk' is widely used, there is no globally recognised legal definition of this term.

the first months of life, and are considered as a breast-milk substitute. In order to assure safe infant nutrition products, regulations have laid down legal specifications for infant nutrition products according to the age-specific requirements of infants and young children. In addition to the legal classification, infant formulae are often classified according to the origin of the ingredients (Table 9.2). An alternative classification of infant formulae is shown in Table 9.3, and it is based on how the product is utilised.

9.4 An overview of the world market of infant formulae

9.4.1 *Annual production figures*

The overall global market value of infant formula, infant cereals, meals, drinks and growing-up formulae has been $\sim 16 \times 10^9$ euros in 2006. About 46% of this value can be attributed to infant formulae (Fig. 9.5). In view of high and specific scientific knowledge required for the development of infant formulae, and the very high level of quality control and safety procedures throughout the entire process of manufacturing and marketing of these products. In addition, these products are manufactured by only a small number of companies

Table 9.2 Classification of infant formula based on the origin of ingredients used.

Raw material	Component(s)
Cow's milk	Utilisation of non-modified milk resulting in <22 g whey protein 100 g^{-1} total protein
Whey (adapted)	Utilisation of whey protein enriched milk resulting in >22 g whey protein 100 g^{-1} total protein
Soya	Utilisation of soya protein as the sole protein source (always enriched with L-methionine)
Elemental	Utilisation of free amino acids as the sole protein source
AR	Utilisation of a thickener like gelatinised starch or locust bean gum
Lactose free	Replacement of lactose (i.e. <0.1 g 100 g^{-1}) by maltodextrin

AR = Anti-regurgitation.

Table 9.3 Classification of infant formula based on the age of its consumers.

Type	Recommendation
LWBF	<2.5 kg body weight at birth
IF	0–6/12 months
FUF or FOF	6–12/36 months
GUM	10/12 months and above
FSMP	For infants with specific physiological and/or metabolic needs

LBWF = low birth weight formulae; IF = infant formulae; FUF = follow-up formulae; FOF = follow-on formulae; GUM = growing-up milk; FSMP = formulae for special medical purpose.

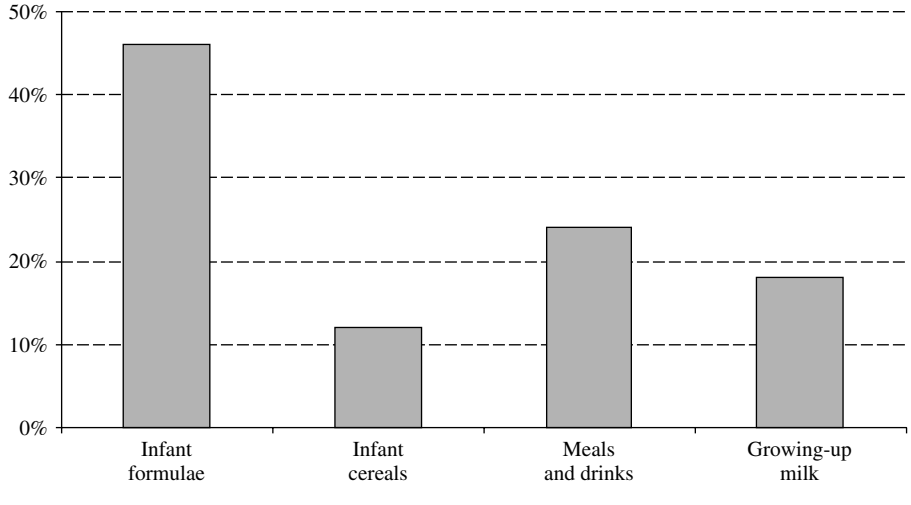

Fig. 9.5 An overview of different categories of infant foods in 2006. Source: Euromonitor International. Reproduced with permission from Euromonitor International 2007.

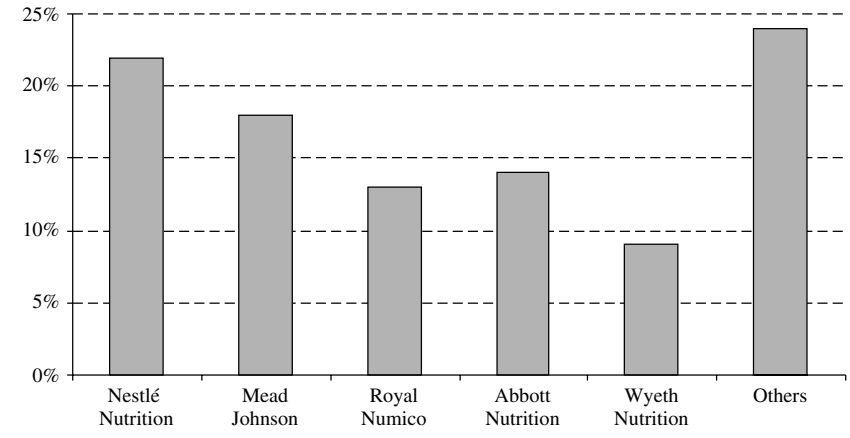

Fig. 9.6 Market share of infant formula in 2006. Source: Euromonitor International. Reproduced with permission from Euromonitor International 2007.

worldwide, five of which accounts for 70% of the global market value (Fig. 9.6). North and South America covered about 46% of the worldwide sales of infant formulae, followed by 31% for Africa and Asia, and finally 24% for Europe (Fig. 9.7).

9.4.2 *Worldwide manufacturers of infant formulae*

Although numerous small or local companies produce infant formulae worldwide, only a few are the major players both at the global and regional level.

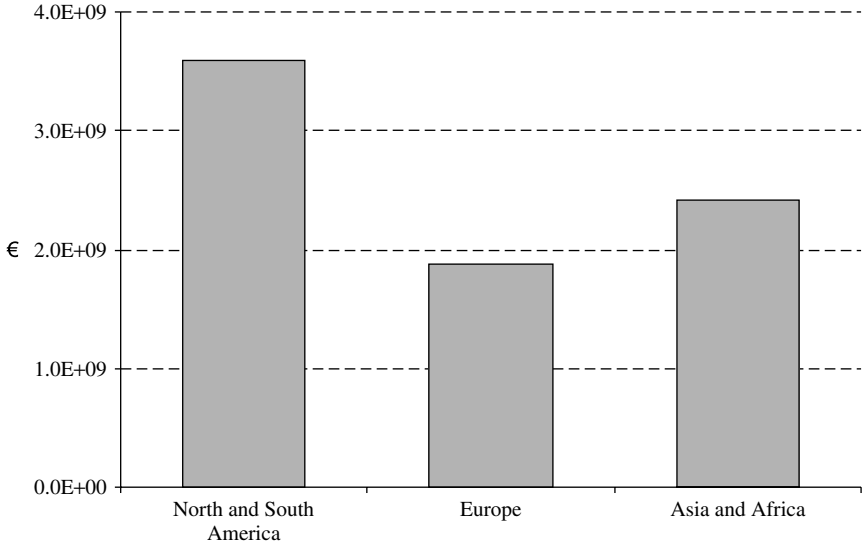

Fig. 9.7 Global consumption of infant formulae in 2006. Source: Euromonitor International. Reproduced with permission from Euromonitor International 2007.

Nestlé nutrition

The nutrition and wellness division of Nestlé SA, a Swiss based multinational food company (Vevey, Switzerland), addresses the needs of customers, such as infants and children, hospitalised patients, elderly and athletes. Its main infant formula brands are Nestlé®, NAN, Lactogen, Beba, Nestogen, Guigoz and Good Start. Recent innovations have resulted in hypo-allergenic (HA) formulae and infant nutrition products containing probiotic bacteria. Following the acquisition of Gerber and of the Medical Nutrition division of Novartis in 2007, Nestlé Nutrition can be considered the global leader in infant nutrition.

Mead Johnson Nurtritional

The nutritional division of the US pharmaceutical group Bristol Myers Squibb (Evansville, Indiana, USA) addresses the needs of infants and children. Its leading infant formula brand is Enfamil. A recent innovation resulted in the development and implementation of LIPIL that is a mixture of docosahexaenoic acid (DHA) and arachidonic acid (ARA), the two long-chain poly-unsaturated fatty acids (LC-PUFA) found in breast milk.

Abbott Nutrition

The nutritional division of the US pharmaceutical company Abbott (Columbus, Ohio, USA) addresses the needs of customers requiring specific nutritional support, such as infants and children, hospitalised patients and elderly. Its leading infant formula brands are Similac, Isomil and Alimentum. Their latest innovations have addressed the addition of nucleotides to infant nutrition products.

Royal Numico

A Dutch-based food company (Amsterdam, the Netherlands) that specialises in nutrition for particular health-care purposes targeting infants, children, hospitalised patients and the elderly. Their leading infant nutrition brands are marketed under Nutricia, Cow & Gate, Milupa, Mellin and Dumex. The latest innovation targeted the addition of a prebiotic mixture composed of galactooligosaccharides (GOS) and fructooligosaccharides (FOS) to infant nutrition products. In 2007, Royal Numico was taken over by the French-based multinational food company Danone (Paris, France) and integrated in 2008.

Wyeth Nutrition

The nutritional division of the US pharmaceutical group Wyeth (Madison, New Jersey, USA) addresses the needs of infants and children. Its leading infant formula brands are S-26, Promise, Promil and Progress. The latest innovation targets the addition of lutein, an antioxidant, to infant nutrition products.

Several other companies are either small global players or strong local players in infant nutrition, and some examples follow.

- Snowbrand, Morinaga and Meiji are Japanese companies and important players in Japan and Asia.
- Heinz, the US-based multinational food company, markets infant formula under the brands Farleys and Plasmon where the main markets are UK, Italy and some Asian countries.
- Friesland Foods, a Dutch-based company, markets infant formulae under the Friso brand, and is mainly active in the Middle East and Asia.
- Danone (France) products are Bledina and Gallia and are marketed mainly in France. Danone has acquired Royal Numico.
- Hipp and Humana companies (Germany) are important players in the Germany, Austria and Switzerland.

9.5 Regulations governing infant formulae

9.5.1 *General background*

The goal of regulatory bodies is to protect consumers and to set a normative framework for free trade. At international level, Codex Alimentarius Commission, under the auspices of the Food and Agricultural Organisation (FAO) of the United Nation and the WHO, elaborates global food standards, which aim to provide a regulatory guidance to national authorities when developing local legislation (FAO/WHO, 2006). In addition, Codex Alimentarius standards serve as the legal basis for any trade dispute when raised at the World Trade Organisation (WTO). At national or supranational level, several regulatory bodies have issued regulations laying down the legal requirements for infant nutrition products. A review of regulations currently applicable in the field of infant nutrition products is given in Table 9.4.

Within the scope of infant nutrition regulations, the definition of infants, young children and infant nutrition products is extremely important. Infants are commonly defined in regulations as children under the age of 12 months. The definitions of infant nutrition products covering the infant's nutritional needs (during early life) as currently laid down by regulatory bodies are summarised in Table 9.5. As far as the regulatory criteria are concerned, most regulatory bodies lay down criteria for the essential chemical composition, the use of food additives, food hygiene, contaminants and labelling recommendations.

9.5.2 *Cultural and religious aspects*

Religious or cultural aspects can require specific manufacturing conditions. Foods for consumption by Moslems and Jews have to comply with 'halal' and 'kosher' requirements, respectively. In order to comply with the vegetarian status, foods have to fulfil well-defined criteria in terms of origin of ingredients. Both cultural and religious aspects are highly specific, and require expert assistance in order to be compliant with the recommendations.

Table 9.4 Infant nutrition regulations in some countries.

Legislation	Infant formulae	Follow-on formulae	Formulae for special medical purpose	Pre-term or low-weight birth formulae
Codex Alimentarius Commission (international)	FAO/WHO (2007) Codex STAN 72-1981 – Revised Section A	FAO/WHO (1987) Codex STAN 156	FAO/WHO (2007) Codex STAN 72-1981 – Revised Section B	NA
United States of America	FDA (1985, 2007a, b) Infant Formula Act, 21 CFR 107	NA	FDA (1985, 2007a, b) Infant Formula Act, CFR 107	
European Union (EU)	EU (2006b) Commission Directive 2006/141/EC	EU (1999) Commission Directive 1999/21/EC	EU (1989) Council Directive 89/398//EEC	
Australia & New Zealand	Food Standards Australia and New Zealand Standard 2.9.1[a]			

NA = not applicable; FAO = Food and Agricultural Organisation; WHO = World Health Organisation; FDA = Food and Drug Administration.

[a]http://www.foodstandards.gov.au/_srcfiles/Standard_2_9_1_Infant_Formula_Products_v95.pdf
http://www.foodstandards.gov.au/thecode/foodstandardscode.cfm,
http://www.foodstandards.gov.au/_srcfiles/Standard_2_9_2_Infant_Foods_v69.pdf, and
http://www.hc-sc.gc.ca/fn-an/alt_formats/hpfb-dgpsa/pdf/res-rech/mfhpb01_e.pdf

9.5.3 *Labelling*

Labelling refers to any written, printed or graphic matter that is present on the label or is displayed near the food, including that for the purpose of promoting its sale or disposal. In general, the specific provisions for labelling include (a) the name of the food, (b) list of ingredients, (c) the declaration of the nutritive value, (d) date marking and storage instructions, (e) information for use and (f) additional labelling requirements.

The Codex states in detail the requirements of the general standard for the labelling of pre-packaged foods (FAO/WHO, 1991), the guidelines on nutrition labelling (FAO/WHO, 1993), the recommendations for labelling and claims of foods for special medical purpose (FAO/WHO, 1991) and the guidelines for use of nutrition and health claims (FAO/WHO, 2004), which all have a bearing on infant formulae and formulae for special medical purposes for infants. These requirements include a prohibition on the use of nutrition and health claims for foods for infants and young children except where specifically provided for in relevant Codex standards or national legislation. In view of the importance of labelling, details are provided on each of the specific item(s) as shown in Table 9.6. Similar provisions apply in the European Union (EU), except that the labels may bear under specifically described conditions of nutrition and health claims in accordance with a new Regulation 1924/2006 (EU, 2006a).

Table 9.5 Infant nutrition definitions in some countries.

Legislation	Reference	Definition
Codex Alimentarius Commission (international)	FAO/WHO (2007) Codex STAN 72-1981 – Revised Section 1	Infant formula is defined as a substitute for human milk in meeting the normal nutritional requirements of infants. The term infant means a person not more than 12 months of age.
United States of america (USA)[a]	FDA (1985, 2007a, b) Infant Formula Act, 21 USC 321 (7)	A food that purports to be or is represented for special dietary use solely as a food for infants by reasons of its simulation of human milk or its suitability as a complete or partial substitute for human milk.
European Union (EU)[a]	EU (2006b) Commission Directive 2006/141/EC	Infant formulae means foodstuffs intended for particular nutritional use by infants during the first 4–6 months of life (or the first months of life) and satisfying by themselves the nutritional requirements of this category of persons (or of such infants[b] until the introduction of appropriate complementary feeding).
Australia & New Zealand	Food Standards Australia and New Zealand Standard 2.9.1	A product represented as breast-milk substitute for infants[b] and which satisfies the nutritional requirements of infants aged up to 4–6 months.

Australia and New Zealand: FAO = Food and Agricultural Organisation; WHO = World Health Organisation; FDA = Food and Drug Administration.
[a]For further information refer to Table 9.4.
[b]Infants mean children under the age of 12 months.

9.5.4 *Procedures for placing infant food product on the market*

Authorities usually apply one of the two distinct procedures for placing the infant formulae on the market, namely *notification* and *registration*. The former aspect generally implies that the manufacturer forwards a label of the infant formula to the competent authority when a new product is placed on the market. Registration, on the other hand, requires that the manufacturer obtains clearance from the competent authority prior to placing the product on the market.

In general, notification puts more emphasis on the responsibility of the manufacturer as compared to registration. Indeed, although notification requires a self-regulatory approach to be taken by the manufacturer, in order to avoid any regulatory issues to come up after the product launch, registration clearly implies a pre-launch approval by the regulatory authorities.

9.6 Essential composition

9.6.1 *Introduction*

With breastfeeding as the reference, the essential composition of human milk is most often used as guidance for the essential composition of infant formulae. However, more than

Table 9.6 Labelling information on packaged infant formulae as specified by Codex Alimentarius.

Name of the product	• The text of the label and all other information accompanying the product shall be written in the appropriate language(s). • The sources of protein in the product shall be clearly shown on the label.
List of ingredients	• A complete list of ingredients shall be declared on the label in descending order of proportion except that in the case of added vitamins and minerals, these ingredients may be arranged as separate groups for vitamins and minerals. Within these groups the vitamins and minerals need not be listed in the descending order of proportion. • The specific name shall be declared for ingredients of animal or plant origin and for food additives.
Declaration of nutritive value	• The declaration of nutrition information shall contain the following information which should be in the following order: (a) The amount of energy, expressed in kilocalories (kcal) and/or kilojoules (kJ), and the number of g of protein, carbohydrate and fat 100 g^{-1} or 100 mL^{-1} of the food as sold as well as 100 mL^{-1} of the food ready for use, when prepared according to the instructions on the label. (b) The total quantity of each vitamin, mineral, choline and any other ingredient 100 g^{-1} or 100 mL^{-1} of the food as sold, as well as 100 mL^{-1} of the food ready for use, when prepared according to the instructions on the label.
Date marking and storage instructions	• The date of minimum durability (preceded by the words 'best before') shall be declared by the day, month and year in un-coded numerical sequence except that for products with a shelf life of more than 3 months, the month and year will suffice. The month may be indicated by letters in those countries where such use will not confuse the consumer. • In addition to the date, any special conditions for the storage of the food shall be indicated if the validity of the date depends thereon.
Information for use	• Adequate directions for the appropriate preparations and use of the product, including its storage and disposal after preparation, i.e. that formula remaining after feeding should be discarded, shall appear on the label and in any accompanying leaflet. • The directions should be accompanied by a warning about the health hazards of inappropriate preparation, storage and use. • Adequate directions regarding the storage of the product after the container has been opened, shall appear on the label and in any accompanying leaflet.
Additional labelling requirements	• Labels should not discourage breastfeeding. • The label shall have no pictures of infants and women nor any other picture or text which idealises the use of infant formula. • Information shall appear on the label to the effect that infants should receive complementary foods in addition to the formula, from an age that is appropriate for their specific growth and development needs, as advised by an independent health worker, and in any case from the age over 6 months. • The products shall be labelled in such a way as to avoid any risk of confusion among infant formula, follow-up formula and formula for special medical purposes (FSMP).

Data compiled from FAO/WHO (1991).

similarity in essential composition, the adequacy of infant formulae should be evaluated by comparing the physiological (e.g. growth patterns), biochemical (e.g. plasma markers) and functional (e.g. immune responses) outcomes of formula-fed with those of fully breastfed infants.

Recently, these principles have been applied by both the Codex Alimentarius Commission and the European Commission when defining the compositional criteria for infant formulae. The recent revision of the Codex infant formula standard requested advice from an international group of scientific experts in the area of infant nutrition (Koletzko *et al*., 2005). Similarly, the European Commission has requested advice on the essential composition of infant formulae from its Scientific Committee on Food (SCF) for the revision of its infant formula directive (SCF, 2003). With respect to the use of new ingredients in infant formulae, authorities request that scientific evidence demonstrates their suitability for the particular nutritional use by infants. Guidance on the design and conducting of appropriate studies has been published by expert scientific groups, such as the SCF (SCF, 2003) and the European Society for Paediatric Gastroenterology, Hepatology and Nutrition (Aggett *et al*., 2003). Such guidance should be taken into consideration when ingredients are introduced into infant formulae or follow-on formulae.

The essential components of the infant formulae contain carbohydrates, proteins, lipids, minerals and vitamins. The proportion and the quality of the ingredients used as well as the processing conditions determine the overall quality of the infant formula. Since an infant formula is intended to be the sole source of nutrition of a newborn, the choice and validation of these criteria is crucial (Nasipour *et al*., 2006).

Several legislations worldwide provide the regulatory frame for the essential composition of infant formulae as well as follow-up formulae. In 2007, the Codex Alimentarius adopted and published the revised Codex standard for infant formulae (FAO/WHO, 2007), which replaces the original standard published in 1981. The revised standard takes into account any new scientific finding and advice provided by an international group of experts (Koletzko *et al*., 2005). Similarly, the European Commission published a new Commission Directive 2006/141/EC governing infant and follow-on formulae (EU, 2006b). This new Commission Directive replaces the original Commission Directive 91/321/EEC (EU, 1991), and is based on a recent review by the Scientific Committee of Food of the available scientific evidence on infant nutrition made on behalf of the European Commission (SCF, 2003). The compositional criteria laid down in the new Commission Directive (see Table 9.7) are very similar to those in the revised Codex standard (FAO/WHO, 2007).

9.6.2 *Proteins*

The protein content of an infant formula is a crucial parameter during early life as it must provide both the need of nitrogen and of indispensable amino acids for maintenance of the body and for growth, that is, protein deposition. Therefore, the protein quality and quantity are very important when designing infant formulae. In addition, the protein content is generally calculated from the total nitrogen content by multiplying with a protein–nitrogen conversion factor. Historically, a protein–nitrogen conversion factor of 6.38 has been used for cow's milk protein. Recent scientific opinions concluded that for the purpose of determining the nutritional adequacy of infant formulae, the crude protein content of all types

Table 9.7 Essential nutrient composition of infant formulae by the Commission Directive 2006/141/EC.

	Units	Minimum	Maximum
Energy	kcal 100 mL^{-1}	60	70
Carbohydrate	g 100 $kcal^{-1}$	9	14
Lactose	g 100 $kcal^{-1}$	4.5	Not specified
Sucrose of total carbohydrates (only applicable in formulae made with protein hydrolysates)	g 100 $kcal^{-1}$	Not specified	20
Glucose (only applicable in formulae made with protein hydrolysates)	g 100 $kcal^{-1}$	Not specified	2
Starch (gelatinised/precooked, max 30 g 100 g^{-1} of total carbohydrates)	g 100 mL^{-1}	Not specified	2
Fructo-oligo + galact-oligo saccharides (ratio 10:90)	g 100 mL^{-1}	Not specified	0.8
Total fat	g 100 $kcal^{-1}$	4.4	6.0
Linoleic acid	mg 100 $kcal^{-1}$	300	1200
α-Linolenic acid	mg 100 $kcal^{-1}$	50	Not specified
Linoleic/α-linolenic acid	Ratio	5	15
Protein (cow's milk formula)	g 100 $kcal^{-1}$	1.8	3
Protein (soya milk formula)	g 100 $kcal^{-1}$	2.25	3
Iron (cow's milk formula)	mg 100 $kcal^{-1}$	0.3	1.3
Iron (soya milk formula)	mg 100 $kcal^{-1}$	0.45	2.0
Calcium (Ca)	mg 100 $kcal^{-1}$	50	140
Phosphorus (P) (cow's milk : soya)	mg 100 $kcal^{-1}$	25:30	90:100
Ca/P	Ratio	1	2
Magnesium	mg 100 $kcal^{-1}$	5	15
Sodium	mg 100 $kcal^{-1}$	20	60
Chloride	mg 100 $kcal^{-1}$	50	160
Potassium	mg 100 $kcal^{-1}$	60	160
Copper	μg 100 $kcal^{-1}$	35	100
Zinc	mg 100 $kcal^{-1}$	0.5	1.5
Manganese	μg 100 $kcal^{-1}$	1	100
Fluoride	μg 100 $kcal^{-1}$	0	100
Iodine	μg 100 $kcal^{-1}$	10	50
Selenium	μg 100 $kcal^{-1}$	1	9
Vitamin A (retinol equivalent)	μg 100 $kcal^{-1}$	60	180
Vitamin D (cholecalciferol)	μg 100 $kcal^{-1}$	1.0	2.5

Table 9.7 Continued.

	Units	Minimum for starters	Maximum for starters
Vitamin E (d-α-tocopherol)	mg 100 kcal^{-1}	0.5	5
Vitamin K_1	μg 100 kcal^{-1}	4	25
Vitamin B_1 (thiamin base)	μg 100 kcal^{-1}	60	300
Vitamin B_2 (riboflavin)	μg 100 kcal^{-1}	80	400
Vitamin B_3 (niacin)	μg 100 kcal^{-1}	300	1500
Vitamin B_5 (pantothenic acid)	μg 100 kcal^{-1}	400	2000
Vitamin B_6 (pyridoxine base)	μg 100 kcal^{-1}	35	175
Vitamin B_{12} (cobalamin)	μg 100 kcal^{-1}	0.1	0.5
Vitamin C	mg 100 kcal^{-1}	10	30
Folic acid	μg 100 kcal^{-1}	10	50
Biotin	μg 100 kcal^{-1}	1.5	7.5
Choline	mg 100 kcal^{-1}	7	50
Inositol	mg 100 kcal^{-1}	4	40
Taurine	mg 100 kcal^{-1}	0	12
Carnitine	mg 100 kcal^{-1}	1.2	2.0
Total nucleotides	mg 100 kcal^{-1}	0	5

EU (2006b)

of infant formula should be calculated as total nitrogen ×6.25 (SCF, 2003; Koletzko *et al*., 2005). This condition applies only to infant formulae, and by no means changes the factor of 6.38 when calculating the protein content of milk products (FAO/WHO, 2007).

The amino acid profile of human milk provides the 'golden standard' for any formula and reflects the protein quality. For an equal energy value, the infant formula must contain an available quantity of each of the essential and semi-essential amino acid at least equal to that of human breast milk (SCF, 2003; Koletzko *et al*., 2005). For calculation purposes, the concentrations of methionine and cysteine, and of phenylalanine and tyrosine may be added together when the ratio is <2. If the ratio is between 2 and 3, clinical data are requested. Table 9.8 illustrates the differences in essential and semi-essential amino acids between breast milk, cow's milk, whey, casein and soya protein. For comparison, the amino acid requirements as defined by the commission Directive 2006/141/EC, Annexe 5 (EU, 2006b) are also listed in Table 9.8. It should be noted that the Commission Directive uses the words 'indispensable and conditionally indispensible amino acids' instead of 'essential and semi-essential amino acids' as used in Codex Alimentarius.

Excessive amounts of dietary proteins cause renal solute overload, which is due to the formation of urea. The small stomach of newly born babies has limited gastric capacity, and the digestive and renal immaturity can lead to low tolerance to hyper-osmolar foods (Mace *et al*., 2006). In order to manufacture milk-based infant formulae, cow's milk has to be

Table 9.8 Differences of indispensable and conditionally indispensable amino acids from dairy products compared to breast milk.

Amino acids (AA) (g 100 g^{-1} of protein)	Source of data					
	EU (2006b)[a]	Human milk[b]	Cow's milk[b]	Acid whey[b]	Casein[b]	Soya protein[c]
Cystine + methionine	3.39	3.30	3.17	6.38	2.86	2.60
Histidine	2.22	2.82	2.54	2.42	2.64	2.60
Isoleucine	5.00	4.53	4.49	6.60	4.90	4.90
Leucine	9.21	9.77	9.08	14.81	8.62	8.20
Lysine	6.27	7.63	7.42	11.79	7.36	6.30
Phenylalanine + tyrosine	8.83	7.50	9.64	8.41	10.01	9.00
Threonine	4.27	4.99	4.23	6.74	4.22	3.70
Tryptophan	1.78	1.87	1.37	2.54	1.14	1.40
Valine	5.00	5.06	5.51	6.45	6.11	5.00
Total (semi-) essential amino acids	45.97	47.47	47.45	66.14	47.86	43.70

[a]Data calculated for 1.8 g protein per 100 $kcal^{-1}$.
[b]Jost (2007).
[c]Solae Proteins, The Solae Company, St. Louis, MO, USA, (http://www.solae.com/company/soyessentials/soyprotein.html).

considerably adapted. Indeed, the protein content of cow's milk is considerably higher than that of human milk, the whey to casein ratio is 20:80 for cow's milk and 60:40 for human milk and, as a consequence, the amino acid profile of cow's and human milk differ (Jost *et al.*, 1999). Therefore, the following adaptations are necessary:

- Reduction of the protein level
- Fractionation of the milk protein
- Enrichment of the milk with whey proteins
- Reduction of the casein fraction
- Enrichment with certain essential and semi-essential amino acids.

Soya protein is, after cow's milk protein, the most widely used protein for infant formulae. The protein has to be isolated from the soya bean in order to remove undesirable components, such as oligosaccharides, fibres, saponins and phytoestrogens. The amino acids profile of soya protein isolates is deficient in sulphur-containing amino acids, and supplementation with L-methionine is, therefore, required.

An important aspect, which needs to be considered when formulating an infant formula, is the potential allergenicity to milk or soya proteins. A reduction of the risk of allergic reactions to cow's milk protein can be achieved by hydrolysing the proteins to smaller peptides, namely so-called hypo-allergenic (HA) infant formulae (von Berg *et al.*, 2003).

Another aspect, which requires attention, is the potential of lysine residues of proteins to react with reducing sugars [e.g. lactose, high-dextrose equivalence (DE) maltodextrins] during heat processing or prolonged storage at high temperature. This reaction known as the Maillard reaction results in blockage of lysine and, as a consequence, a loss of the protein nutritional value and a lower protein digestibility.

9.6.3 *Lipids*

Dietary lipids are essential for normal growth and development. The dietary fat (i.e. predominantly triglyceride or triacylglycerol) is the predominant source of fuel energy for breastfed and formula-fed infants. Oxidation of 1 g fat yields about 38 kJ (9 kcal) of energy or twice as much as protein and carbohydrate. Besides the energy value, dietary lipids provide essential fatty acids and fat-soluble vitamins to the organs, and are also necessary for efficient absorption of the fat-soluble vitamins, carotenoids and cholesterol. Furthermore, fats are carriers of flavours in the diet, and contribute to its satiety value (Carey & Hernell, 1992).

The quality of the lipids is expressed by their fatty acid composition, the degree of saturation, the position of the fatty acids on the glycerol backbone (stereo-specific fatty acid distribution) and the content of *trans* fatty acids. The fatty acid composition is determined by the choice of fats and oils. The oils used to manufacture infant formulae are usually of vegetable origin (e.g. soya, sunflower, rapeseed and safflower). Milk fat is also used, but levels are usually low.

Linoleic acid ($C_{18:2}$ n-6) and α-linolenic acid ($C_{18:3}$ n-3) are essential fatty acids, from which the respective metabolites of the n-6 and n-3 series of fatty acids, the long-chain poly-unsaturated fatty LC-PUFA acids are synthesised by shared endogenous enzyme systems. Consequently, regulations lay down the criteria for linoleic and α-linolenic acids as well as for the ratio between these two essential fatty acids (EU, 2006b; FAO/WHO, 2007). Infant formulae are often enriched with (LC-PUFA) containing oils. Oils rich in LC-PUFA contain ARA and DHA; these two fatty acids have been identified to play a role in brain function and visual acuity. Due to the high degree of unsaturation of these oils, they are particularly susceptible to oxidation and, hence, special care has to be taken in order to avoid fat oxidation and rancidity. Regulations classify the supplementation of infant formula with LC-PUFA's as optional but, when added, there are specific regulatory requirements to comply with (EU, 2006b; FAO/WHO, 2007).

Finally, the specific quality criteria, which are laid down by regulations for lipids in infant formulae, are highlighted, such as (a) the use of sesame seed oil and cotton seed oil is prohibited; (b) lauric and myristic acid (separately or as a whole) shall not be more than 20 g 100 g^{-1} of total fat content; (c) the *trans* fatty acid content shall not exceed 3 g 100 g^{-1} of the total fat content; and (d) the erucic acid content shall not exceed 1 g 100 g^{-1} of the total fat content (EU, 2006b; FAO/WHO, 2007).

9.6.4 *Carbohydrates*

Human milk contains both digestible and indigestible carbohydrates. Lactose is the predominant digestible carbohydrate of human milk, 55 to 70 g L^{-1} or 8.2 to 10.4 g 100 $kcal^{-1}$.

In addition, human milk contains a complex mixture of non-digestible oligosaccharides, ~20 g L^{-1} in the colostrum and 5–13 g L^{-1} in the lactating milk (Coppa *et al*., 1999; Kunz *et al.*, 2000), the concentration in the latter ranges between 5 and 8 g L^{-1}. Digestible carbohydrates serve as essential sources of energy in the diet and, moreover, provide structural elements for the synthesis of glycolipids and glycoproteins. Disaccharides and polysaccharides from the diet are hydrolysed to monosaccharides which, after absorption in the upper small intestine, are converted to glucose in the liver. Human milk oligosaccharides were shown to be resistant to enzymatic digestion in the upper gastrointestinal tract (Engfer *et al*., 2000). Among other functions, human milk oligosaccharides may serve as substrates for colonic fermentation. It has been shown that human milk oligosaccharides induce an increase in the number of *Bifidobacteria* spp. of colonic microbiota (flora) in breastfed infants, accompanied with a significant reduction in the number of potentially pathogenic bacteria (Kunz *et al*., 2000). Complex oligosaccharides have the ability of inhibiting the binding of pathogens to cell surfaces because they act as competitive receptors on the host cell surface, thereby preventing adhesion of a number of bacterial and viral pathogens.

As in human milk, the carbohydrates fraction constitutes the major part of infant formulae (usually about 55 g 100 g^{-1} of the solids). In infant formulae, lactose is often the major carbohydrate, but other carbohydrates (maltodextrins, starches and/or maltose) are also being used. The addition of cooked starch results in a thickening of the reconstituted formula, and is perceived to give more satiety and less regurgitation.

9.6.5 *Minerals*

As for proteins and fats, minerals also play an important role in the nutrition of infants. Six elements (K, Na, Ca, Mg, P and Cl) are referred to as minerals, and are present in mg 100 $kcal^{-1}$, whereas the micro- or trace elements (Fe, Cu, Zn, Se, Mn and I) are present in μg or lower 100 $kcal^{-1}$. The nutritional relevance of the presence of minerals will not be discussed in this chapter, but the chemical form and the quantity of minerals can have a crucial influence on their bioavailability and the physical stability of the formula in which they are present. A few examples are given as illustration:

- Fe salts are commercially available in their ferric (Fe(III) or Fe^{3+}) and ferrous (Fe(II) or Fe^{2+}) forms, and each salt has a different solubility and bioavailability. Thanks to its high bioavailability, Fe^{2+} sulphate is generally used as Fe source in infant formulae. However, with Fe being a catalyst also for fat oxidative reactions, the presence of highly soluble Fe^{2+} sulphate in a formula with LC-PUFAs can result in a rapid oxidation of the latter. The processing conditions, the type and atmosphere of packaging are, therefore, of great importance to avoid oxidative reactions, which can result in rancidity and formation of free radicals.
- Ca and Mg are available as water soluble (e.g. chloride) and insoluble (e.g. citrate and phosphate) salts. As bivalent soluble cations, they strongly react with the proteins (milk and soya) to form coagulants after heat treatment, a fact that is well known in the cheese industry (e.g. Mozzarella and Quark). In their insoluble citrate or phosphate form, they hardly react with the proteins, but they sediment during processing or during

storage in the liquid (e.g. 'Ready-to-Feed' or concentrated formulae) to form a hard and difficult to reintegrate layer. As a consequence, making a heat stable formula without sedimentation of the calcium salts is a challenge for each infant formula producer, and the successful manufacturing procedures are, therefore, well protected.

9.6.6 *Vitamins*

Milk contains both fat- and water-soluble vitamins. The fat-soluble vitamins are retinol (vitamin A), β-carotene (pro-vitamin A), calciferol (vitamin D), tocopherol (vitamin E), and phylloquinone (vitamin K). Their absorption from infant formulae is related to the efficacy of formula fat absorption. These vitamins are stored in body fat depots, such as adipose tissue, and high intakes over longer periods of time may lead to their accumulation in the tissues. In addition to dietary intake, their transplacental supply during pregnancy determines an infant's stores of fat-soluble vitamins at birth, which may vary considerably within populations and may modulate the dietary requirements during the first months of life.

The water-soluble vitamins are thiamine (vitamin B_1), riboflavin (vitamin B_2), niacin (vitamin B_3), pantothenic acid (vitamin B_5), pyridoxine (vitamin B_6), cyanocobalamine (vitamin B_{12}), ascorbic acid (vitamin C), folic acid (vitamin B_9) and biotin (vitamin H or B_8). Excess intake of these vitamins is easily eliminated from the body as compared with fat-soluble vitamins.

9.7 Food safety

9.7.1 *Food additives*

Food additives are substances added to food products to provide a specific and well-defined action in the final product. Different classes of food additives are defined and each of which serves a specific purpose. Food additive classes used in infant formulae are thickeners, emulsifiers, acidity regulators, antioxidants and packaging gases. The use of food additives in infant formulae is strictly regulated (EU, 1995; FAO/WHO, 2007), whilst the safety of use of food additives is subject to a thorough risk assessment by scientific expert bodies prior to their incorporation into any legislation. For example, the Codex Alimentarius Commission refers to the FAO Joint Expert Consultation of Food Additives (JECFA), whereas in the EU, this risk assessment is performed by the European Food Safety Authority (EFSA).

9.7.2 *Hygiene and microbiological standards*

Ensuring consumer protection imposes a strict control of food hygiene, in particular, microbiological criteria. With respect to infant formulae, regulations lay down certain strict criteria for the presence of micro-organisms. The Codex Alimentarius lays down the provisions for micro-organisms with the specific standards. Hence, the infant formula standard recommends that it is prepared and handled in accordance with the appropriate sections of the Recommended International Code of Practice – General Principles of Food Hygiene (FAO/WHO, 2001) – and other relevant Codex texts such as the Recommended

International Code of Hygienic Practice for Foods for Infants and Children (FAO/WHO, 1979).

In the EU legislation, several regulations cover the hygiene of foodstuffs. Presently, a specific Commission Regulation 2073/2005 (EU, 2005) covers the hygiene requirements for different food items that include infant formula with specific recommendations (see also EU, 2006b, c).

9.8 Raw materials/ingredients

9.8.1 *General aspects*

Typical raw materials for infant formulae are as follows:

- Milk (skimmed or full fat, liquid or powder, cow's or buffalo's)
- Casein (acid or K/Ca neutralised)
- Whey protein (concentrate, isolate, partially demineralised), partially hydrolysed
- Isolated soya protein, locust bean seed protein and amino acids (elemental formula)
- Lactose, maltodextrins, corn syrup, saccharose (powder or syrup)
- Vegetable oils (unhydrogenated), butter oil, cream
- Emulsifiers/stabilisers [lecithin (soya), monoglycerides and diglycerides]
- Mineral salts (K, Na, Ca and Mg as carbonates, citrates, phosphates or chlorides)
- Micro-nutrients (e.g. vitamins, amino acids, KI, $FeSO_4$, $ZnSO_4$, $CuSO_4$)

However, 'optional' ingredients are added to more selective infant formulae with potential specific functional and nutritional benefits in accordance to regulatory requirement. Some optional ingredients, which can be added to infant formulae, are as follows:

- Prebiotic fibres (GOS, FOS and inulin) (Agostoni *et al*., 2004b; Bakker-Zierikzee *et al*., 2005; Moro & Arslanoglu, 2005; Brunser *et al*., 2006; Pérez-Conesa *et al*., 2006, 2007; SookHe *et al*., 2007).
- Probiotic micro-organisms (e.g. *Bifidobacterium animalis* sp. *lactis*, *Lactobacillus rhamnosus* GG, *Bifidobacterium longum* BL999 and/or other strains of *L. rhamnosus* (Agostoni *et al*., 2004a; Bakker-Zierikzee *et al*., 2005; Petschow *et al*., 2005; Puccio *et al*., 2007; Pérez-Conesa *et al*., 2007).
- Speciality oils (e.g. deodorised high DHA fish oil, LC-PUFA oils *ex* micro-algae extraction, medium-chain triglycerides, inter-esterified palm oil and sphingolipids) (Ribar *et al*., 2007; Koletzko *et al*., 2008).
- Carob bean gum and starch (corn or potato) (Aggett *et al*., 2002; HsunChin & Vandenplas, 2007).

9.8.2 *Milk*

When the milk source is fresh, it will be cooled after reception, and kept in intermediate storage tanks before standardisation of the fat content, homogenisation, heating and cooling, followed by cold storage in insulated silo tanks. From these tanks it will be pumped to the re-hydration station for mixing with the dry ingredients.

9.8.3 *Oils*

Oils are supplied either in drums or in bulk from insulated road tankers. Oils from the drums, which usually have been heated prior to emptying, are conveyed via double-jacketed piping to storage tanks that are fitted with load cells. The number of tanks used depends on the choice of the product and storage time. The tanks can be provided with a double jacket as well, making individual temperature regulation possible; this is especially important for coconut oil.

Anhydrous butter oil or anhydrous milk fat (AMF) is supplied and stored in 200 L drums. The vegetable oils, such as non-hydrogenated soya, corn, palm, palm olein, sunflower, rapeseed, safflower and coconut oils, are supplied by road tankers and stored in silos; whereas speciality oils, such as medium-chain triglyceride (MCT) oils and LC-PUFA oils, are stored in smaller tanks or pumped directly from the original packaging containers into the oil line or the product mix. In order to prevent oxidation of the poly-unsaturated oils, the tanks holding the oils are usually equipped with an inert gas (N_2) supply.

9.8.4 *Carbohydrates*

More recently, maltodextrins and glucose syrup are supplied as hot syrups (solids ~70 g 100 g^{-1}) by road tankers.

9.9 Manufacture of dried infant formulae (powders)

9.9.1 *Introduction*

In general, milk powders can be produced using either a 'dry mix' or a 'wet mix' process; the latter process is followed by spray drying. Each type of these processes has its specific advantages and disadvantages.

The advantages of the 'dry mix' process:

- The lack of water involved in the process makes it safer from a microbiological point of view as no growth can be expected.
- A much smaller investment, as less equipment and thereby, a smaller building is needed. The costs for energy and maintenance will also be considerably lower.

The disadvantages of the dry mix process:

- It does not allow incorporating oils.
- No heat treatment is included; thereby the physical and microbiological quality is defined by the quality of the raw materials used.
- Some of the physical powder properties, such as wettability and solubility, will be defined by the properties of the single ingredients unless a powder agglomeration process follows the mixing.
- The different ingredients have different densities, and will, therefore, segregate during transportation; in other words, the product can be inhomogeneous in appearance and composition.

The situation is different when it comes to infant formulae, which are exclusively produced by the use of wet mixing, emulsification, concentration by evaporation and spray drying. The possibility of using high-pressure homogenisation for the correct incorporation of oils in the protein matrix and the application of an adequate heat treatment of the milk base before drying ensure that all aspects of quality (i.e. microbiology, physical and chemical properties) can be controlled to a higher degree, and the resulting infant powder is of better quality. Obviously, the investment, as well as production costs, becomes higher with this process.

At present, the two processes are often combined by adding some of the dry nutrients (e.g. vitamins, trace elements or carbohydrates) after the drying process. Special attention should then be taken to the microbiological quality of these ingredients. As the 'wet mix' process is the most widely used for the production of infant formulae powder, the subsequent sections will only describe this process.

9.9.2 *The 'wet mix' processing line*

A typical production line for the infant formula using the 'wet mix' process method consists of following three main processing stages (Fig. 9.8).

Preparation of the mix – To achieve a homogeneous oil in water emulsion in the mix, the water soluble ingredients are carefully recombined in milk or water followed by in-line dosing of the oil-emulsifier mix at an adequate temperature (~60–70°C). The mixing is followed by high pressure homogenisation and cooling.

- *Evaporation* – Evaporation of the milk leads to the concentration of the solids content of the homogenised mix. The final heat treatment of the product is applied to kill all pathogenic organisms; this is typically done just before the evaporation stage of the mix in order to avoid excessive fouling.
- *Drying* – Drying of the concentrated mix takes place in a spray dryer using hot air to obtain a powder with good wettability, solubility, taste and nutritional quality.

In a modern plant, it must be taken into account that recipes and production methods are being continuously developed and improved. Furthermore, manufacturers of infant formulae have a natural interest not to reveal the specific processing details of their products. Therefore, each infant formula processing plant must be specially designed, and the following aspects should be considered before designing a processing line of infant formulae:

- Selection of raw materials, number of raw materials, as well as possible substitutes
- Determination of the tolerances of the composition of recipes
- Determination of process parameters, such as time/temperatures/pressures
- Training of personnel and level of automation
- Daily production output and cleaning routines, for example, prior to change of recipe
- Flexibility enabling changes if all conditions have already been determined

The following description of a complete wet mixing plant should be considered as an example of how a modern production line should be designed. The aim of the 'wet mix' process is to blend the liquid and powder (i.e. lipophylic and hydrophylic raw materials) into

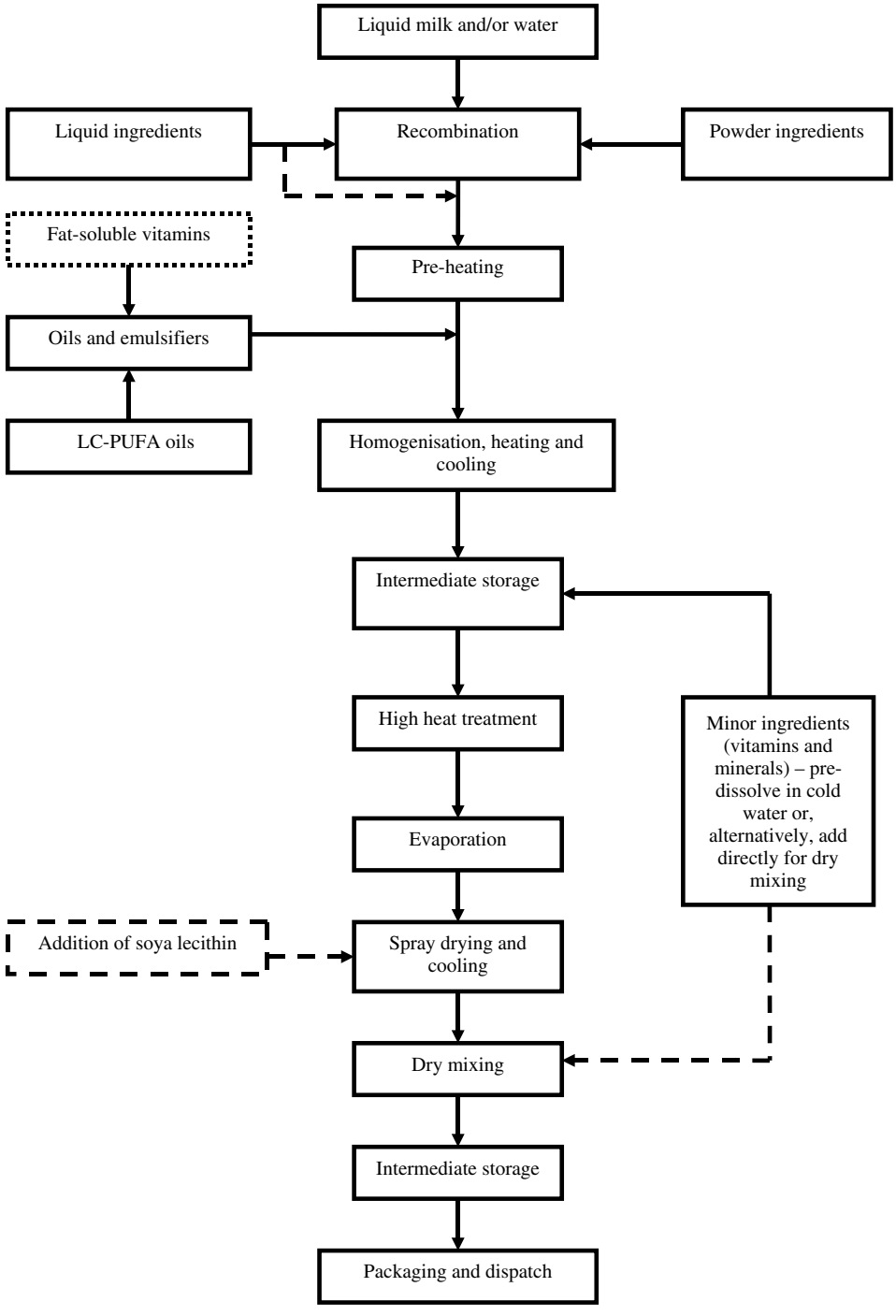

Fig. 9.8 The manufacturing stages of dried infant formulae. Note: dotted lines are alternative processing route.

a stable emulsion by homogenisation, and to inactivate all pathogenic organisms by heat treatment. Lots of variations are possible depending on the product, raw materials used and company traditions (Sørensen *et al*., 1992; Pisecky, 1997; Zink, 2003; Westergaard, 2004).

9.9.3 *Preparation of the mix*

The mixing method(s) is normally divided into a system for the water-soluble part, and a system for the oil-soluble part. The preparation can be continuous or batchwise. The water-soluble part is done by adding the powders into the milk or water using a high-shear mixer. The mix is circulated into a tank where the powder(s) and hot water are continuously added until the total batch volume has been achieved. As the mixer usually works under vacuum, the powder from the hopper easily enters the mixer when the valve is opened. After the dissolution of all the dry ingredients, the mix is stored in a hydration tank giving all the dry ingredients time for complete hydration. To this mix, the water-soluble minerals that are prepared in a separate tank by mixing the minerals with hot water can also be added. The pH of the mix may be adjusted by addition of an alkali (KOH or NaOH) or citric acid solution.

The pre-heated oils are usually added in-line into the pre-heated product mix followed by homogenisation and cooling. Sensitive speciality oils of the poly-unsaturated type can be added to the oil mix or to the oil stream just before the homogenisation in order to keep the oil protected as long as possible. Oil-soluble vitamins are normally solubilised in oil in a small separate tank and then added to the oil mix tank. However, more and more encapsulated fat-soluble vitamins are used. In this case, all the vitamins are supplied as a cold water-soluble premix with a certificate of analysis, which has the advantage that routinely only one tracer vitamin has to be analysed. This vitamin premix is either dissolved in water and added to the final storage tank before drying, or added and mixed as a dry premix to the spray dried powder that is added prior to dry mixing.

All powder supply pipes should afterwards be flushed by compressed air, and the complete mixing line should have cleaning-in-place (CIP) on a daily basis (see Chapter 3 for further technical details).

In the case of batch processing, the heat treated and cooled mix (5°C) is pumped to a final storage tank. To this tank, the water-soluble vitamins are added. At this stage of the process the quality of the mix (chemical analysis and microbiological examination) should be checked, as to whether it fulfils all specifications before resources are used for concentration and drying. If the mix does not fulfil these specifications, the plant should be designed in a way making corrective actions possible.

In the case of continuous processing, all controls (solids, fat, proteins and minerals) are done on line, and adjustments are done continuously. All equipment for mixing, hydration and storage as well as discharge lines should be CIP-ed at least once a day, whilst the heat exchanger should be CIP-ed every 8 h to keep the microbiological quality under control.

9.9.4 *Evaporation*

The raw materials used in the different formulations are often mixed to lower solids content than optimal for spray drying. This is because the recombination of the dried ingredients

is better at lower solids. Since it is cheaper to remove water by evaporation as compared with spray drying, concentration of the mix using an evaporator (i.e. falling film, multi-stage) is usually done before the final drying. This has the following additional advantages:

- Possibility to heat treat the mix effectively at a higher temperature because it is less viscous (i.e. contains lower solids content) and minimum fouling problems of the equipment.
- Deaeration of the concentrate results in a powder with minimal content of occluded air.
- Better drying economy due to the higher solids content of the concentrate, resulting in less water to evaporate per kg of powder.
- Using higher total solids of the concentrate improves the powder properties of the dried product by improving the agglomeration of powder particles.

Typically, the falling film and multi-stage vacuum evaporators are fed with the mix from one or two heating systems installed prior to the evaporator. The heat treatment is achieved through indirect heating of the mix from typically 5°C to 80°C, and direct contact or steam injection heating to 90–120°C with a holding of 5–30 s followed by flash cooling to ~78°C. The spent vapour from the evaporator and flash energy from the heat treatment system are used for product pre-heating from 5°C to ~78°C (i.e. regeneration). The falling film evaporator should be also equipped with a double heat treatment system to make a possible switch for CIP during production. Thermal vapour recompression (TVR) as well as mechanical vapour recompression (MVR) can be used for the falling film evaporator(s); for further technical details refer to Chapters 3 and 4.

9.9.5 *Spray drying*

The typical spray drying equipment consists of the following:

- High-pressure nozzle(s) or rotary atomiser/wheel for the atomisation of the concentrate
- Drying chamber and an air supply system with heaters and filters
- External or integrated fluid bed dryer for cooling of the powder
- Cyclones or bag filters as an air–powder separation system

The concentrate mix is pumped through a pre-heater/pasteuriser and a filter by means of a centrifugal pump to the atomising unit of the dryer. If a nozzle unit is used, a high-pressure pump is installed after the filter. The drying chamber can be equipped with an integrated fluid bed dryer for a lenient secondary drying/conditioning and agglomeration in order to improve the final product quality and drying economy. The powder is finally cooled in an external fluid bed dryer before sieving and further processing or filling. The drying air is passed through air filters, and heated in an indirect air heater before entering, via a specially designed air disperser, the spray dryer. The used air drying and cooling of the powder is also passed through air filters as well. However, for microbiological safety, these air filters will normally have a higher degree of filtration due to the lack of heating of the air. Furthermore, the cooling air may be de-humidified depending on the ambient

conditions. The exhaust air, from the spray drying chamber and the fluid bed dryer is discharged through typically two cyclones and a bag filter for collection of 'fine' powder particles entrained in the air. Afterwards, the powder ('fines fraction') from the cyclones is returned to the drying chamber, the atomising zone or the external fluid bed dryer to obtain the desired degree of agglomeration. The exhaust air from the cyclones can be filtered by a bag filter. The powder from the bag filter(s) is usually disposed of as animal feed.

In addition, the following points must be taken into consideration when designing a spray drying plant for an infant formula:

- Hygiene and production time between CIP or dry cleaning
- Desired powder structure (fine or agglomerated powder)
- Drying parameters (air temperatures, residence time of the powder in the spray dryer)

9.9.6 *Hygiene and production time between CIP cleaning*

All equipment have to be designed as per the strictest hygienic requirements. Hollow spaces in the drying plant should be avoided, and insulation material with double air gap sandwich panels, which can be removed from the drying chamber, should be standard. Alternatively, a hot room design can be used. This design transfers the chamber insulation to the building by insulating the room where the drying chamber is situated. The wall is typically quite close to the drying chamber. An important aspect in the manufacture of dried infant formula is a long continuous operation time between cleaning. Downtime means lack of production and risk of contamination during start-up of production.

The reason for cleaning is different in the separate parts of the plant. The wet feed system (e.g. plate heat exchanger) is CIP-ed more often in order to keep the microbiological quality under control and, if the feed system is CIP-ed more often than the evaporator, a double feed system can be used making it possible to keep the evaporator and spray dryer running for longer duration. The dryer is normally cleaned, if there is a change in composition of the product to be dried or build-up of powder deposits along the sides of the drying chamber.

9.9.7 *Structure of the powder*

To a great extent, dried infant formulae sold today are marketed as agglomerated powders (Fig. 9.9); the agglomeration process improves the reconstitution properties of the product. It is, therefore, important that the degree of agglomeration and the compactness of the agglomerated particles can be controlled. The typical density of a well agglomerated infant formula powder is between 400 and 500 g L^{-1}. In addition, by using nozzle atomisers in the spray dryer, the best powder structure is achieved and optimisation of the agglomeration process is done by adjusting the pressure of the concentrate, the spraying angle and the position of the nozzle(s) in relation to each other as well as to the returned fine powder particles from the cyclones. When using a rotary atomiser, the cyclone fraction is distributed to the atomiser zone.

For both spraying systems, the obtained degree of agglomeration is also a question of product composition. Thus, with increased content of carbohydrates, it becomes easier

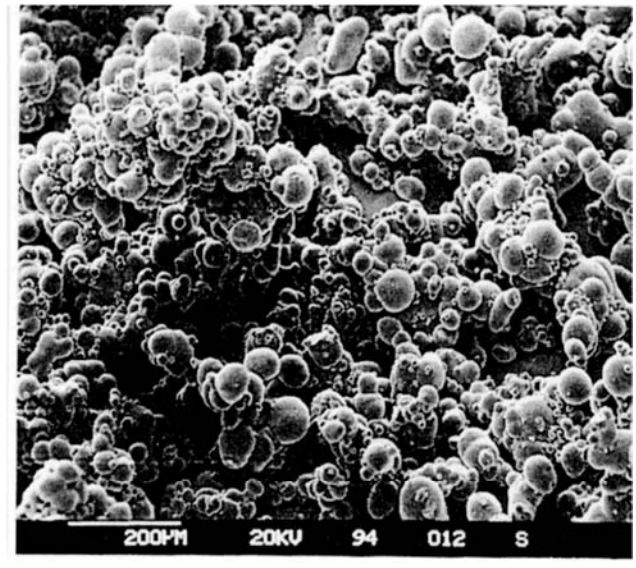

Fig. 9.9 Scanning electron microscope (SEM) image of agglomerated milk powder.

to agglomerate, whereas products with a high content of proteins will result in smaller agglomerates. Furthermore, if a very low bulk density is needed, CO_2 can be added to the concentrate before drying. This is a nozzle atomisation option only because the feed has to be under pressure to absorb the CO_2.

9.9.8 *Drying parameters*

In order to improve the production economy, the drying temperatures and solids content of the concentrate are selected to be as high as possible. The product composition, however, is the decisive factor as to what parameters can be selected. As a rule of thumb, the following aspects apply:

High fat content

- Allows high solids content of the concentrate
- Requires low drying temperature

High protein content

- Requires low solids content of the concentrate due to viscosity (except for hydrolysed proteins)
- Allows high drying temperature without sticking

High carbohydrate content

- Allows high solids content (except for starch) of the concentrate
- Depending on the type of carbohydrate added, the drying conditions(s) can allow low exhaust air drying temperature resulting in higher dryer capacity; generally, long-chain carbohydrate (starch, low-DE maltodextrins) containing formulae can be dried at higher

exhaust air temperatures, this is due to their high glass transition temperature, which results in less sticking in the drying chamber.

Typical temperatures (T) for an infant milk formula are as tabulated below:

• T-main (chamber) drying air	180–200°C
• T-integrated fluid bed drying air	50–60°C
• T-external fluid bed cooling air	30–20°C
• T-exhaust air from dryer	80–100°C

The temperatures will, as always for spray drying, be affected by the ambient air moisture. Higher ambient moisture requires lower drying air temperatures and higher exhaust air temperatures making the dryer less efficient. In some cases, dehumidification has to be applied to the drying air, which obviously increases the production costs.

A hopper can be installed to take the powder from the beginning and the end of the dryer cycle, as this powder can have slightly atypical powder properties. The powder is then mixed into the first grade powder from the dryer during production by a controlled discharge of the hopper to the drying chamber of the external fluid bed dryer. Thereby, all the powder produced will be of the same uniform quality. However, in order not to affect the quality of the total production too much, this addition of start-up or end of drying powder should not be higher than 10%.

9.9.9 *Finished powder conveying system*

The final product from the spray dryer is transported to a silo via a positive pressure dense phase transport system. From the bottom of each silo, the product is transported to either a blending area for mixing with other powder(s) and/or to the filling line by vacuum transport. Normally, the powder is packed in a N_2 atmosphere to prevent oxidation of the milk fat or vegetable (poly-unsaturated) oil. To make things simpler, the powder from the dryer can fall by gravity into a hopper and from the hopper directly into the bagging or metal can filling lines. Which option to choose depends on the overall approach to logistics, capacity and capital investment.

9.9.10 *Microbiological examination*

Infant formula powders are not produced in a sterile environment but, nevertheless, should be microbiologically safe. Since the powders are for very sensitive consumers and will, in most cases, be consumed without heating after re-hydration, they have to be of the highest microbiological quality. A good hazard appraisal (analysis) critical control points (HACCP) system must, therefore, be in place in order to guarantee upfront conditions, which guarantee a safe final powder at the consumer end. As mentioned elsewhere, the use of clean and boiled water for the preparation of the feeding bottle is of essential importance to avoid illness to infants due to microbial contamination.

Recently, health authorities have given considerable attention to the opportunistic pathogen *Enterobacter sakazakii*, which has been related to a health risk in infants and, particularly, in pre-term, underweight or immunocompromised infants. Consequently, Codex Alimentarius Commission is in the process of revising the Recommended International Code of Hygienic Practices for Foods for Infants and Children in order to address concerns raised by pathogens that may be present in infant formula (FAO/WHO, 2001). Similarly, the EU has recently reviewed their criteria for microbiological safety, including criteria for *E. sakazakii* (Zink, 2003; EU, 2005; WHO, 2005).

The infant formula powders are examined for the detection of the following:

- Aerobic mesophilic counts
- Enterobacteriaceae (especially *E. sakazakii*)

9.10 Manufacture of liquid infant formulae (Ready-To-Feed and concentrates)

Based on the manufacturing stages shown in Figure 9.10, the individual processing steps for the production of liquid infant formula will be described.

9.10.1 *Dissolving of ingredients*

In the mixing/dissolving device, the liquid and powder phases are mixed until a particle-free solution is achieved. Dissolution is facilitated at slightly elevated temperatures. There are batch and continuous mixing devices. For continuous mixing systems, all the ingredients have to be continuously and proportionally metered. A critical issue in this processing step is the formation of foam, which can create problems during further processing, and can lead to product losses. For this reason, some of these mixing devices operate under vacuum.

9.10.2 *First stage of standardisation*

In the batch production system, the mix is collected in the first standardisation tank. This allows preliminary control and, if necessary, adjustment of the total solids. In addition, this standardisation step allows the separation of the subsequent processing stages in order to facilitate process harmonisation.

9.10.3 *Oils and fat addition*

Different qualities of unhydrogenated vegetable and/or butter oils (or cream) are used for the manufacture of infant formulae. The vegetable oils are usually supplied as a blend and, at this stage, lecithin is added to facilitate the emulsion stability of the final product. Also, fat-soluble vitamins can be added at this point. The lipid materials are heated to $\sim 60^{\circ}C$ to ensure complete melting of all components, to reduce the viscosity of the mix and to facilitate their incorporation into the protein matrix. Some oils (highly unsaturated) have a tendency to oxidise when in contact with air. The handling and storage of such oils need special care and have to be carried out under constant protection with an inert gas.

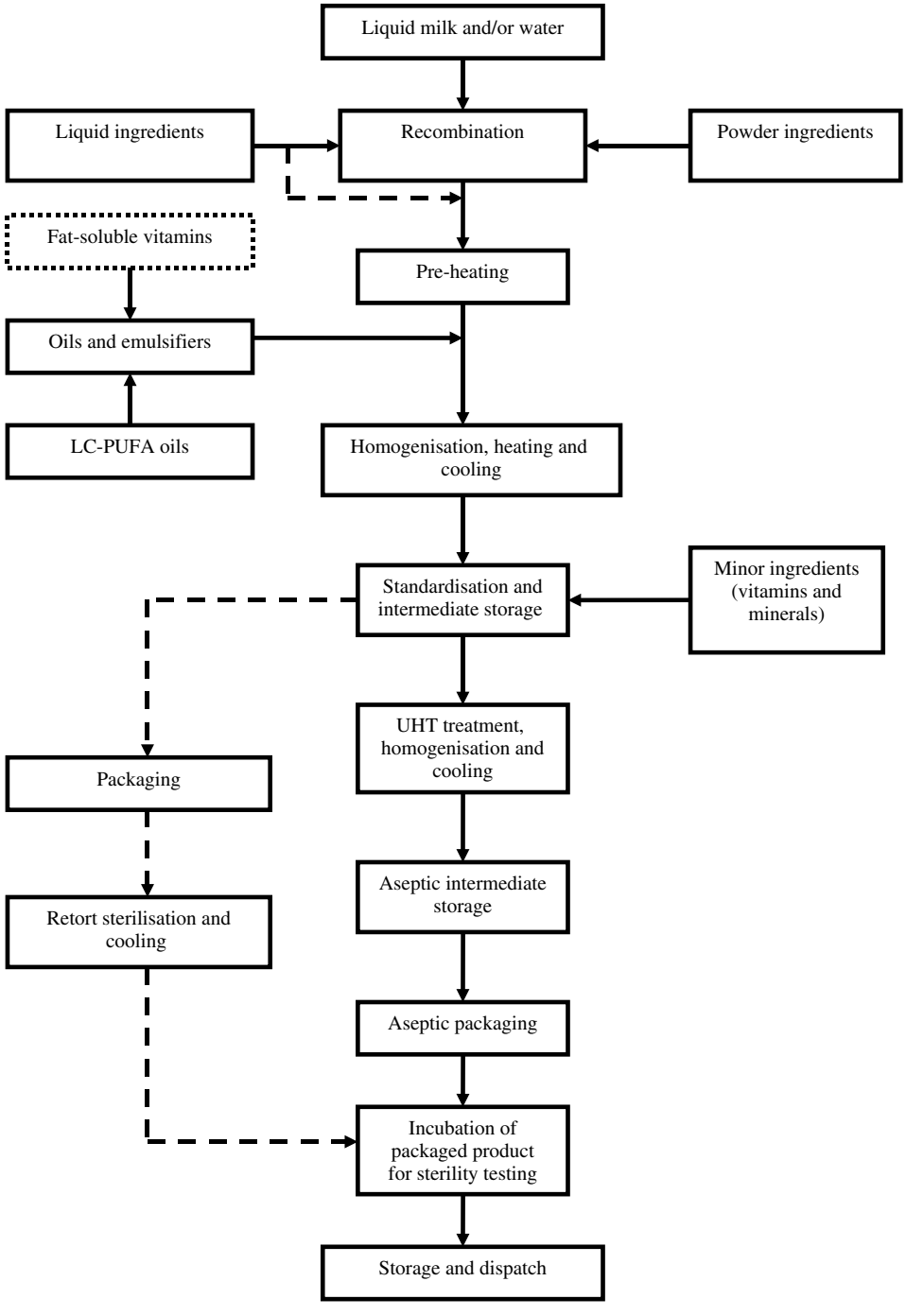

Fig. 9.10 The manufacturing stages of liquid infant formulae. Note: dotted lines are alternative processing routes.

9.10.4 *First heat treatment and fat emulsification*

This first heat treatment has several objectives: *firstly*, to obtain a stable emulsion between the oily phase and the water-soluble components; *secondly*, as a first treatment to reduce potential microbiological load from the raw materials; and *thirdly*, to inactivate possible unwanted enzymes (e.g. phosphatase, lipases and proteases). With the homogenisation at ~60°C and a pressure between 15 and 25 MPa, a stable fat/protein emulsion is achieved. The heating can be applied using an indirect or a direct system, and the temperatures applied range between 85°C and 120°C. When using a direct heating system, a subsequent flash cooling is included; this enhances homogenisation efficiency and results in an oxygen-free product.

9.10.5 *Second stage of standardisation*

After the emulsification step, the cooled product is collected in a second buffer tank. At this stage, all minor ingredients, such as water-soluble vitamins, trace elements, mineral salts and amino acids, are added. The correct final composition of the product is now adjusted based on quick laboratory test results, for example, solids, protein, fat and main minerals. The storage time in the standardisation tank has to be limited since the cold product under agitation absorbs oxygen from the atmosphere, which leads to a loss of some vitamins (especially vitamins A and C).

9.10.6 *Final conditioning*

All liquid infant formulae undergo a final thermal treatment to guarantee commercial sterility, which means that it does not contain any micro-organisms that can multiply (see Section 9.10.11 for more details). To fulfil this goal, the thermal treatment is defined in decimal reduction values (D-values) for specific heat-resistant spores. Depending on the sterilisation temperature used, the D-values of the most resistant spores in this range are used to calculate the necessary decimal reduction. An international standard for the temperature ~120°C is 12 decimal reductions for *Clostridium botulinum* spores. In the ultra-high temperature (UHT) range of 140 to 150°C, *Bacillus stearothermophilus* (now known as *Geobacillus stearothermophilus*) spores are generally used as the reference. The treatment of liquid infant formulae can be achieved by using (a) a retort steriliser after filling the product into cans or glass bottles or (b) a continuous UHT system (direct or indirect heating) before filling the product, followed by aseptic homogenisation and aseptic filling into sterile glass, metal cans, plastic or cardboard containers.

9.10.7 *Retort sterilisation*

The standardised product is filled into a container (i.e. non-aseptic condition), sealed and post-sterilised in a retort. The packaging container can be metal cans or plastic or glass bottles. The processing conditions in the retort consist of heating to 118–122°C for 15–20 min of the filled and sealed containers using pressurised hot water; this corresponds to an F0

value of >8. From the sterility point of view, this is a very safe process, particularly if metal cans are used. However, the quality of the product suffers due to long-time heating period, which can induce Maillard reactions, where the colour becomes brownish, the taste more cooked and the nutritional value and some vitamins can be reduced.

Post-sterilised (retort) liquid infant formulae are losing their importance and today, they are found mainly in hospitals to feed premature babies.

9.10.8 *UHT sterilisation and aseptic processing*

This process aims at achieving commercial sterility with minimal negative influence on the nutritional value of the product, the physical properties, the taste and also minimal fouling during the process (i.e. burnt product deposits on the heating surfaces and holding tubes) (Fig. 9.11). For these reasons, liquid infant formulas are preferably UHT treated (i.e. using direct steam injection or indirect heating systems). In the former sterilisation system, the product is pre-heated in-line in a heat exchanger to ~80°C, followed by a very fast increase of temperature by direct steam injection to 140–150°C and kept in a holding tube for a few seconds. Afterwards, cooling down to ~80°C is done instantaneously, which is achieved in a vacuum vessel. During this so-called flash cooling, the product is completely deaerated and the initially injected steam is removed. The final cooling to the filling temperature is carried out in standard indirect heat exchangers (e.g. tubes or plates).

When using the indirect UHT system (i.e. plates or tubes), the product is pre-heated in-line in a heat exchanger to ~80°C, followed by a fast increase of temperature to 135–145°C and kept in a holding tube for a few seconds. The subsequent cooling down to ~20°C is achieved in a standard indirect heat exchanger. After the first cooling step to 80°C, an aseptic two-stage homogeniser (5 and 25 MPa) is usually used. Both heat exchangers for pre-heating and cooling are combined in order to save energy. The heat load of the indirect systems on the product is higher than with direct systems, resulting in more intensive Maillard reactions, and more intensive fouling and decreased phase stability. For these reasons, direct UHT systems are more suitable for the production of liquid infant formulae.

Fig. 9.11 A logo illustrating the concept of aseptic production and packaging of food products.

9.10.9 *Intermediate aseptic storage*

The sterile and cooled product is normally stored in a tank under aseptic conditions. This storage is not mandatory, but has several advantages:

- The UHT steriliser may not have the same flow rate as the subsequent filling machines; intermediate storage allows for more flexibility for the filling with different machines or for different packaging volumes.
- Some filling machines, such as the Tetra Pak packaging machines, need a slight overflow if fed directly without intermediate storage, which leads to losses.
- The intermediate storage of a product allows the isolation of each production batch, which can be an advantage for traceability.

The aseptic storage of a sterile product needs specific installations and procedures, and requires complex computerised technology. It has to be guaranteed that no contamination of any kind can occur between product sterilisation and the filling machines. Therefore, this part of the production line has to be built fully hermetically sealed, thus excluding any contamination of the production line by the unsterile environment. It requires special valves and has to be cleaned fully automatically. Before any sterile product enters this line, all parts are heated with water or steam to $\sim 120^{\circ}C$ for 20–30 min to sterilise the processing line. Afterwards, the sterile processing line is cooled with sterile water or sterile air, and a positive sterile overpressure is kept all the time. For very oxygen-sensitive products, the aseptic tank can be pressurised with an inert gas.

9.10.10 *Aseptic filling machines and packaging materials*

After the intermediate aseptic storage of the product, the liquid infant formula is filled into the final retail container. During the intermediate aseptic storage, any potential recontamination of the product has to be prevented during filling. The filling machines and packaging material have to be sterilised before the product can be packaged. As such, the filling machines cannot be pressurised and, since both machines and packaging materials are of limited heat resistance, chemical sterilisation is mostly applied. A commonly used method is to cover the surfaces of the packaging container to be sterilised with a hot mist of hydrogen peroxide (H_2O_2). During the drying of the surface of the packaging material, the H_2O_2 is broken down into H_2O (i.e. vaporised) and O_2. The free oxygen destroys all micro-organisms, even spores. Other sterilisation methods and combinations of different systems exist, such as gamma irradiation or sterile bottle blowing. However, there is a wide range of different packaging types, and the most commonly used ones, including their typical properties, are shown in Table 9.9.

The shelf life of liquid infant formula ranges between 6 and 12 months, which is dependent on the phase stability (i.e. influenced by the raw materials and the manufacturing process), possible changes of the taste of the product and deterioration of the vitamin content during storage. Using packaging containers with good oxygen barrier properties and small and/or gas flushed head space, a longer shelf life of the product can be expected. In

Table 9.9 Some examples of contains used to package liquid infant formulae.

Packaging materials	Description	Main properties	Suppliers
Cardboard with layers of aluminium foil and PP	Square packs formed on the machine from a roll of packaging material	Light weight with different opening devices, big printing surface, good O_2 and light protection	Tetra Pak
Cardboard with layers of aluminium and PP	Pre-formed square packages	Light weight, different opening devices, big printing surface, good O_2 and light protection	Combibloc SIG
Multilayer of PP bottles with light and O_2 protection	Round bottles with an aluminium seal and plastic cap (i.e. re-closable)	Practical for use, but needs bottle blowing installation, acceptable O_2 and light protection	Ampak, Serac, Krones
Glass bottles	Multilayer pot sealed with aluminium foil	Like a yoghurt pot; mainly for viscous products, good O_2, but with limited light protection	Serac, Hamba, Jagenberg
Metal cans	Metal can with or without easy opening	Best product protection, thanks to optimal O_2 and light protection, but old fashion image	James Dole

PP = polypropylene.

order to guarantee the traceability of all products, each pack is coded during filling. From the printed text, the batch number, the filling machine and the date and time of filling can be identified.

Aseptically filled infant formulae are rigorously tested for composition and sterility before release for sale. 'Challenge' sterility tests are carried out by incubating a certain proportion of packages from each batch at an optimal temperature for microbiological growth, and then testing all the incubated packs for sterility. For some packaging and products non-destructive automatic sterility tests exist. Certain of these tests identify products with an altered viscosity that can be caused by microbial spoilage. Infections are possible only in rare cases, which do not change the physical properties of the product. The most frequently used test system is the so-called 'Electest'. It is based on controlled shaking of incubated packs followed by measuring the movements. Other systems use gamma irradiation or reflect rays.

9.10.11 *Microbiological examination*

Although specific safety issues are addressed in HACCP studies, the safety of aseptically filled UHT products is ensured by the application of prerequisite measures, such as good manufacturing practices (GMP) and/or good hygiene practices (GHP) including line validation, and a thorough and systematic training of dairy personnel. These prerequisite measures enable the manufacture of shelf-stable products with the avoidance of spoilage.

Incubation and testing of finished products alone are not sufficient to guarantee their absolute microbiological stability and safety. Results obtained from these tests represent

only a verification that the process is under control. They can be used for the release, but only in conjunction with strict control and registration of all process parameters, the identification of critical control points (CCPs) in the production line and systematic maintenance.

Commercial sterility, for example as specified by the Canadian Government in 2001–MFHPB-01 (http://www.hc-sc.gc.ca/fn-an/alt_formats/hpfb-dgpsa/pdf/res-rech/mfhpb01_e.pdf) is as follows: the condition achieved by the application of heat, alone or in combination with other treatments, to render a food free from viable forms of micro-organisms, including spores, which are capable of growing in the food at temperatures at which the food is designed normally to be held during distribution and storage. If the normal temperature for storage or handling of the product is higher than 40°C then analyse for thermophiles. Otherwise, analyse for mesophiles only. Sampling plans for incubation of test samples, which are destructive and non-destructive controls, have to be set up for each manufacturing line according to experience and any history of non-sterility events. Typically 1 sample per 1000 produced units is set aside for destructive testing. Incubation for laboratory samples is usually carried out for (a) mesophilic bacteria at 30°C for at least 7 days and (b) thermophilic bacteria at 55°C for at least 5 days. However, for the detection of spoilage organisms in non-acid liquid products after incubation, following methods are recommended:

Destructive methods

- pH determination
- Impedance method (Coppola & Firstenberg-Eden, 1988)
- Direct streaking on agar plates or enrichment in a nutritive medium
- ATP-bioluminescence

Non-destructive methods

- Visual examination of damaged, leaky or blown packs
- Electester (Tuomo Halonen OY, Finland) using the principle of a viscosity change of the product caused by curdling
- Vacuum determination measuring the deflection of lids
- Other methods, such as taptone, ultrasound, magnetic resonance, near infrared spectroscopy (NIRDAS) and colour change.

9.11 Conclusion

Breastfeeding is the ideal form of infant feeding during the early life of a child, but substitutes to breast milk existed as long as man. Technological and scientific developments led to the manufacturing in the late nineteenth century of standardised breast-milk substitutes or infant formulae. Continuous scientific research and technological progress have enabled procedures to improve considerably the nutritional composition of the early infant formulae.

Simultaneously with improved infant formulae, efforts to promote breastfeeding have been increased at international level and led to the adoption of the International Code of Marketing of Breast-Milk Substitutes by the World Health Assembly in 1981 (WHO, 1981).

Today, the essential composition, production and marketing of infant formulae are also controlled strictly by regulations, which make the infant formula one of the most regulated and controlled industrially produced foods. Regulations lay down the criteria for the essential composition of infant formulae, assuring that the particular nutritional requirements to enable normal growth and development of the infants are met. Innovations in infant formulae are a result of scientific research and are subject to approval by regulatory authorities based on scientific substantiation. It is reasonable to assume that evolving science and technology will enable (1) the manufacturers to continue with their innovations in infant formulae (e.g. essential composition, packaging, production and safety) and (2) the authorities in different countries to continue to adopt new regulations in order to assure the highest quality and safety to infant formula consumers.

In conclusion, improvements have been made in the safety and nutritional aspects of infant formulae (i.e. if used according to the instructions of the manufacturer) and will continue to be made; however, breast milk is still the ideal food for infants.

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10 Process Control in Evaporation and Drying

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10.1 Background

The processes used in the manufacture of dairy powders and concentrated products must be well controlled to ensure safe operation, compliance with regulatory requirements, appropriate product quality and low manufacturing costs.

Dairy processing is characterised by batch-continuous operation. Equipment is run for a few hours, or in the case of some spray dryers, a few weeks, before being shut down for cleaning. Process control systems should be able to cope with the gradual fouling of heat transfer surfaces throughout each run. They also should be able to ensure an effective cleaning of the equipment for hygiene and efficiency reasons. Minimisation of the fouling reduces the effluent loads, increases the product output, reduces energy consumption and provides cost savings due to reduced downtime.

Process control plays a role in protecting the environment in the vicinity of a spray drying operation. Examples include the control of cleaning-in-place (CIP) to avoid wastage of cleaning chemicals (Anonymous, 1997). Minimisation of fouling benefits the environment by reducing the effluent loads. Another way, process control protects the environment relates to energy use. Milk powder is the most energy intensive dairy product to manufacture. Good process control minimises energy use, with consequent minimisation of greenhouse gas production through the burning of coal and gas for steam raising and electricity production.

The control tasks are complicated by the lack of on-line sensors for many of the quality attributes of greatest interest. Instead, physical, chemical and microbiological laboratory analyses are used to assess quality. The results of these analyses are delayed by times ranging from minutes to hours, or in the case of some microbiological tests, days. The results are also subject to significant measurement error when compared with, for example, temperature measurements.

Dairy processing equipment is operated within a services infrastructure. The electricity, steam, hot, cold and chilled water, compressed air, building heating, ventilation and air conditioning and other services are typically shared by many items of equipment operating on different schedules. This provides a great deal of scope for different processes to interfere with one another through their demands on site services.

The control engineer is, therefore, faced with the challenge of operating quasi-steady state processes in a disturbed environment with delayed and relatively inaccurate feedback of many of the key performance measures. To make matters more challenging, the drive for energy efficiency has led to plants designed with high levels of heat regeneration. That is,

thermal energy is transferred backwards and forwards with respect to the milk flow. This has two deleterious effects on control performance: first, it allows disturbances to propagate at different speeds backwards and forwards through the plant, and second, it reduces the leverage of the control inputs. In extreme cases, excessive heat regeneration has prevented a plant from starting up at all (Bloore, 2007).

The primary task of the control system is to maintain constant levels of a number of process output variables measured on-line in the face of slowly drifting steady state system gains and process disturbances. The appropriate choice of set-point values to ensure that quality and throughput targets are met has traditionally been the responsibility of the staff. In recent years, high-level optimising control systems have been developed to assist, or in some cases, take over this role. The control system must also interface with the operating staff, providing them with information to assist them in interpreting causes of deviations from normal operation (Pisecky, 1997). The interface to staff may be a function of a plant supervisory system, separate from, but integrated with, the plant control system.

10.2 Control technology

The type of control technology used depends on the nature of the processes to be controlled. A simple way of analysing processes is to count the numbers and types of inputs and outputs (I/O). Dairy processes typically have thousands to tens of thousands of digital I/O, but only a few hundred analogues I/O. The number of single loop controllers will usually be a few tens to perhaps a hundred or so. This makes dairy processing different from assembly lines, where the huge preponderance of digital I/O has led to the universal adoption of programmable logic controllers (PLCs). Dairy processes are also different from petrochemical plants where analogue I/O dominates and distributed control systems have found favour. The dairy control engineer, therefore, has to make a decision on the most cost-effective control system architecture. The reader is referred to some publications for more complete discussion on automation and process control in the dairy industry (Tamime & Law, 2001; Anonymous, 2003; Lopez-Gomez & Barbosa-Canovas, 2005; Cramer, 2006; Elaisson, 2006; Heldman & Lund, 2007).

Most evaporation and drying processes in the dairy industry are controlled using PLCs that carry out sequential control of valves and implementing proportional–integral–derivative (PID) algorithms to control single input single output control loops. The human machine interface is most commonly supplied by one or more personal computers running supervisory control and data acquisition (SCADA) software. Some older installations still use individual single loop electronic controllers.

Several different higher level control systems have found commercial application. These use model-predictive control algorithms capable of handling multiple-input multiple-output control loops with time-dependent or non-linear steady-state process gains, and in some cases, with nonlinear dynamics. Neural net (e.g. Pavilion Technologies) or recursive least squares (e.g. Connoisseur) models are employed, and the software allows on-line process identification and loop tuning. Process optimisation is a feature of these systems. All rely, however, on low level, usually single loop, controllers to control the process inputs. The higher level control system provides set points to these lower level controllers. Some

research activity on the development of dynamic process models for the purpose of process control is presented in Sections 10.8.6 and 10.9.14 in respect of models with specific application to evaporator and dryer systems, respectively.

10.3 Measurement technology

All control loops require some form of measurement. At the simplest level, switches provide feedback on the position of valves, swing bends and key pieces on flow plates. These are connected to digital inputs on the control hardware. These switches are used to confirm that the selected pipe routes are available, and ensure that the mixing of the product and cleaning solutions is avoided, with obvious implications for food safety.

At the next level of complexity, sensors are available to measure temperature, pressure, volumetric and mass flow rate, conductivity, density, turbidity, humidity, refractive index and rotational speed. Coriolis effect mass flow -metres with a density measurement output have found widespread use to estimate the concentrate total solids in evaporators. These instruments are typically accurate to about 0.5 to 1.0 kg m^{-3}. The density of skimmed milk concentrate varies by about 5 kg m^{-3} *per* 1 g 100 g^{-1} total solids; whilst the density of full cream milk concentrate varies by about 3 kg m^{-3} *per* 1 g 100 g^{-1} total solids. This may limit the precision of control. Coriolis Effect mass flow metres are not usually as accurate as specialist density meters. Concentrate viscosity is an important variable, but none of the commercially available instruments is able to cope with the power-law shear thinning behaviour of milk concentrates while still being robust enough to take the mechanical, thermal and chemical shocks experienced in normal operation. Hygiene is also an issue with some viscometers. 'Home-made' capillary by-pass viscometers operating at constant flow rate have been used with some success.

The most elaborate sensors are adaptations of laboratory analysers for on-line or at-line use. Examples include analysers for measuring the fat, protein and total solids content of liquid milk during milk standardisation and analysers for measuring the fat, protein and moisture content of milk powders at the outlet of fluid bed dryers. These typically use infra-red or near infra-red spectroscopy to determine the concentration of milk components. The accuracy of measurements of the type just described depends on the frequent calibration against reference samples, since they are really inferential in nature, that is, they make inferences about chemical composition based on the measurements of optical properties. Such calibration must not be neglected in on-line operation.

A final category of measurement is the 'virtual' analyser or 'soft sensor'. The output signal from such analysers represents some quality or other product attribute deduced from a range of conventional sensor inputs using a mathematical model. A simple example is the measurement of concentrate viscosity by means of a capillary viscometer. The primary sensor readings of volumetric flow rate and differential pressure along the length of a dairy tube are used, along with the internal diameter and length of the tube to calculate viscosity using the Hagen–Poiseuille equation. More complex 'virtual' analysers use neural net or multiple regression models combined with first-principles physical models to provide real-time estimates of powder moisture. This approach also allows synthetic readings (inferential measurements) to be generated in place of faulty real sensors, providing redundancy (O'Callaghan & Cunningham, 2005).

10.4 Actuator technology

Pneumatically operated sanitary remote control valves are used to control the path of the product and the cleaning chemicals throughout the dairy processing plant. Regulations covering acceptable designs vary from country to country. Pneumatically operated sanitary control valves are often used in conjunction with centrifugal pumps for flow control. Most service flows of steam and water are controlled using pneumatically operated control valves. The control signals sent from the control system are usually electrical, so solenoid valves (for on/off operation) or electro-pneumatic converters (for modulating operation) are used to interface the electrical signals to the pneumatic ones.

Increasingly, positive displacement pumps fitted with variable frequency alternating current (a.c.) drives are being used in flow control applications. Variable frequency a.c. drives are also applied to the motors on the evaporator mechanical vapour recompression fans and the fans on the spray dryers and fluid bed dryers. These drives offer greater energy efficiency than the use of dampers and provide a more linear control response. Older variable frequency drives used 4–20 mA current loop interfaces, but most now take their instructions in digital form via a communications network. However, digital control valves are becoming available, but have been slow to find acceptance in dairy processing plants.

10.5 Communication technology

The earliest standard format used to transmit control signals was the 20–100 kPa pneumatic signal. This was followed by 4–20 mA electrical current signals. Both these signal types are analogous, in which a pressure or electrical current represents the value of the variable being transmitted. Pneumatic and early electronic controllers were also analogue devices, so an analogue communications medium was appropriate. Modern electronic controllers are predominantly digital, so there is a need to convert signals between analogue and digital forms, or to use sensors and actuators, which are digital in operation by themselves.

The integration of digital control systems (controllers, sensors and communications) offers some advantages over a hybrid mix of analogue and digital systems in process control. *Firstly*, the cumulative errors resulting from successive analogue-to-digital and digital-to-analogue conversions are eliminated. *Secondly*, the diagnostic capabilities of integrated digital systems can pinpoint problems for quick corrective action. *Thirdly*, connecting many sensors to one cable can significantly reduce hardwiring costs in a factory.

Thus, 'field area' networks are evolving that connect field devices, such as sensors and actuators, with controllers and man–machine interfaces. A specific requirement of such systems when used for process control is that they must be 'hard' real-time systems, like, giving a deterministic response, not subject to communications delay, and avoiding changes in the system dynamics, which potentially may have a negative impact on the process under control. Typically such real-time communication employs systems such as 'Industrial Ethernet' or 'Real-Time' Ethernet (Felser, 2005).

There are several internationally accepted standards for bus-based digital communication systems for process control. The most commonly used in food processing applications are

the PROFIBUS developed in Europe and the FOUNDATION fieldbus, developed in the United States. PROFIBUS is more commonly associated with PLC-based control systems while the FOUNDATION Fieldbus is more commonly used in distributed control system architectures. There are several proprietary bus systems that have also gained acceptance. These systems greatly reduce cabling requirements since many devices can be attached to a common cable rather than having to be individually wired back to a field cabinet or a central PLC.

Wireless systems are slowly gaining acceptance (Samad *et al*., 2007) and these may reduce cabling requirements even further. Some long-range wireless devices implement standard bus-based digital networks, while others use short-range systems, such as WiFi and Bluetooth to connect transducers and actuators to nearby field cabinets where a copper- or optical fibre-based network takes over.

Whatever communication technology is selected, it is important that the signals passing through are not corrupted in transit. For pneumatic systems, this requires a careful choice of pneumatic line sizes and lengths and the prevention of pressure fluctuations arising from other equipment that share the compressed air supply. Clean dry oil-free air is usually a requirement. For 4–20 mA loop based systems, good electrical grounding practices are vital to minimise noise in the signals. The current loop is popular largely because its low impedance makes it difficult to couple electromagnetic fields to the signal. Poor grounding can convert the instrument cabling into a giant radio antenna capable of picking up interference from variable speed a.c. drives, walkie-talkies and a host of other sources. Modern PLCs have both hardware and software filters available as standard features. It is common to find heavily filtered noisy signals compromising the performance of a control system, where the removal of earth loops would solve the problem and allow improved controller performance (Bloore, 2007). For networked or bus-based systems, the network cable itself can pick up electrical interference. Unless this is extreme, most modern transmission protocols will still get the messages through, but the variable delay time between transmission and reception may affect the controller dynamics. Fibre-optic cabling is not subject to electromagnetic interference. The security of networks against malicious attacks also needs to be addressed (Zurawski, 2005; Igure *et al*., 2006).

10.6 Control philosophies

There are two philosophies in designing a control system. The first is to maintain tight control of all input disturbances that could affect the process, making the task of controlling the process itself relatively simple. The second is to allow disturbances to affect the process, but to control their influence by the use of relatively complicated control algorithms. Control systems used in manufacturing concentrated and dried dairy products embody elements of both approaches, but generally tend to favour one or the other.

The relatively slow dynamics of most evaporator control loops make it attractive to concentrate on controlling the process infrastructure. Eliminating fluctuations in steam pressure, cooling water temperature and milk feed temperature reduces the standard deviation of concentrate total solids greatly, which is a key variable in the spray drying process.

10.7 Process dynamics

The dynamics of evaporators are relatively slow. Typical first order time constants range from 30 s to 5 min for most loops associated with evaporators. Direct steam injection heaters (DSI units) will have time constants of about 1 s. Excluding the pre-heating system, evaporators typically have pure transport delays of the order of 2 to 10 min. For spray dryers the corresponding time constants and delay times are shorter. For well-mixed fluid beds, such as are found on integrated fluid bed dryers, first order time constants range from 5 to 15 min and, for plug flow fluid beds, pure transport delay times may reach 30 min.

10.8 Evaporator control

There are four main controls on an evaporator: (1) the flow rate of milk to the evaporator; (2) the pre-heat temperature; (3) the supply of the energy, which drives the evaporation (e.g. steam); and (4) removal of the energy, which drives the evaporation (e.g. the condenser water flow rate). These are used to match the evaporator to the dryer, that is, to achieve the desired protein denaturation and the desired concentrate total solids.

It is important to maintain a consistent flow of concentrate to the spray dryer which equals the demand from the dryer so that the level in the balance tank between them can be kept as low as practicable. This will minimise the concentrate holding time, reducing the effect of age thickening on the concentrate viscosity. This brings further challenges to the control of the overall powder manufacturing process.

10.8.1 *Feed flow rate*

The feed flow rate is adjusted with a sanitary control valve or a variable speed positive pump. The flow rate is measured with a volumetric flow meter (usually a magnetic flow meter) or a Coriolis mass flow meter, which may also give a feed density reading. The objective is to ensure that the evaporator receives a consistent flow of product (to ensure adequate wetting of the tubes at all times in order to avoid burn-on) and that when the desired concentrate solids level is reached, the concentrate flow rate matches the dryer feed rate.

10.8.2 *Pre-heat temperature*

The pre-heat temperature is adjusted by varying the steam flow to the pre-heat system with a control valve. The objective is to ensure that the product receives a consistent heat treatment, to ensure consistent functional properties such as whey protein nitrogen index (WPNI).

10.8.3 *Energy input*

Adjusting the energy input to the evaporator controls the first effect boiling temperature, fixing the hot end of the temperature gradient through the evaporator. The design

of the evaporator determines which process variable is used to adjust the energy input. The objective is to control the amount of evaporation and, therefore, the concentrate total-solids.

For multiple effect evaporators, flow rate of the main steam is adjusted with a control valve. This may take the form of a simple single loop measuring the concentrate density and manipulating the steam valve position. Often, however, a cascade control system is employed. The steam pressure may be measured with a pressure transmitter, and a slave control loop may be used to maintain a constant pressure at a set point provided by a master loop measuring the concentrate density. Alternatively, the steam flow rate may be measured with a flow element, such as an orifice plate or annubar coupled with a differential pressure transmitter or with a vortex flow meter. A slave control loop may be used to maintain a constant steam flow rate at a set point provided by a master loop measuring the concentrate density.

For thermal vapour recompression (TVR) evaporators or finishers, the flow rate of the TVR steam is adjusted with a control valve. As for the multiple effect evaporator system described above, a single loop or a cascade system may be used. The most common practice is to control the TVR steam supply pressure with a slave loop and to provide the set point to this controller from a concentrate density controller.

For mechanical vapour recompression (MVR) evaporators, the fan speed is adjusted with a variable speed drive. The fan rpm is measured with a tachometer.

10.8.4 *Condenser water flow rate*

The condenser water flow rate is adjusted with a control valve. The flow rate may be measured with a volumetric flow meter, or the final effect pressure may be measured with an absolute pressure transmitter. The objective is to adjust the rate of condensation to control the vacuum and boiling temperature in the back end of the evaporator, fixing the cold end of the temperature gradient through the evaporator.

In older evaporators with mixing condensers and barometric leg vacuum seals, the final effect vacuum may be adjusted using a ‘snifter’ valve. This allows a controlled leak of air into the vacuum pump inlet. In these evaporators, the condenser water flow rate is usually uncontrolled. The final effect pressure may be measured with an absolute pressure transmitter. As with the use of the condenser water flow rate, the objective is to control the vacuum and boiling temperature in back end of the evaporator, fixing the cold end of the temperature gradient through the evaporator.

10.8.5 *Level of total solids in the concentrate*

The total solids content of the concentrate is usually estimated from the measurements of concentrate density. Vibrating tube densitometers or Coriolis Effect mass flow metres with a density output are the most common density meters.

Some combinations of fat, protein and lactose have densities close to that of water, making density measurement useless for total solids estimation. In these cases, the evaporator may be controlled on volume concentration factor (VCF) as for ultrafiltration (UF) plants. The VCF may be calculated from filtered measurements of the flow rates into and

out of the evaporator. The time constant of the filters must be long enough to smooth out short-term fluctuations, which would otherwise cause oscillation.

Milk powder plant capacity may often be increased by increasing the average total solids of the concentrate without increasing evaporator fouling. There is an upper limit to the concentrate total solids if normal run times between cleans are to be maintained. As the level of total solids content in the concentrate varies throughout a production run, there will be a maximum acceptable proportion of the time running in excess of the safe limit.

The distribution of concentrate total solids about the mean value is Gaussian. For such a distribution, 2.275% of the total solids readings will exceed 2 standard deviations (2σ) above the mean value. If σ can be reduced by better control, the mean can be raised by twice the change in σ for the same probability of exceeding the original upper limit.

For example, reducing σ of concentrate total solids control from ± 1.0 to ± 0.5 g 100 g^{-1} would allow set point of the total solids to be raised from 49 to 50 g 100 g^{-1} for the same probability of exceeding mean $+2\sigma$, that is, 51 g 100 g^{-1}, giving a 4.25 g 100 g^{-1} increase in throughput for the same evaporation in the spray dryer. This is illustrated graphically in Figure 10.1.

Many modern evaporators use MVR first effect(s) and TVR finishers. Some evaporator manufacturers run the TVR at constant steam pressure, and adjust the MVR fan speed to maintain an MVR outlet density that gives the required TVR outlet density. Others run the MVR at a constant fan speed (adjusting it upwards 10 rounds per minute (rpm) or so every few hours as it fouls) and control the TVR outlet density with the TVR steam control valve.

In practice, the principal disturbances in an evaporator control system arise from variation in the feed concentration (Winchester & Marsh, 1999). One strategy, which has been devised to reduce with the long delays between disturbances in evaporator feed and detecting the effects on concentrate total solids, is to utilise an intermediate solids concentration meter to monitor concentration *ex* the first effect (or first pass, if multi-pass).

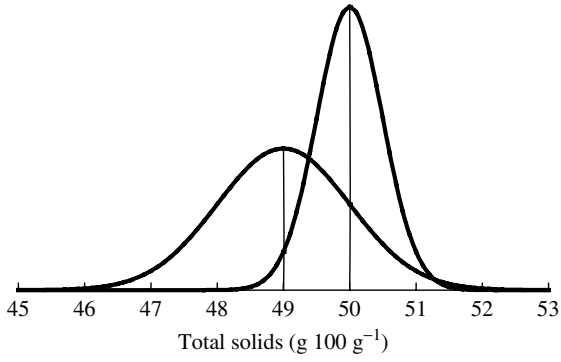

Fig. 10.1 An illustration showing the probability distribution of concentrate total solids (g 100 g^{-1}) for two controller performances; a mean value of 49.0 g 100 g^{-1} with a standard deviation of 1.0 g 100 g^{-1} and a mean value of 50.0 g 100 g^{-1} with a standard deviation of 0.5 g 100 g^{-1}. In both the cases, the probability of exceeding 51.0 g 100 g^{-1} is 2.275%.

10.8.6 *Modelling approaches for evaporator control*

Winchester & Marsh (1999) developed a first-principles model of a falling-film evaporator with MVR with the aim of understanding the ability of a control system to reject disturbances, for example, in feed temperature or dry mass fraction (solids content). They developed models for single- and two-pass designs, where the manipulated variables were milk feed flow rate, compressor speed and the condenser coolant flow rate and they were regulating the main process variables, such as the effect temperature, the product dry mass fraction and the product flow rate. Essentially, they examined the coupling and functional controllability of the three core control loops for effect temperature, product dry mass fraction (percentage total solids) and product flow rate, using a simplified model in which constant velocity was assumed along the calandria tubes and changes in product density, specific heat capacity and heat transfer coefficients with dry mass fraction were neglected. In spite of the simplicity of the model, significant coupling was found between the product dry mass fraction and product flow rate loops and it was concluded that the three-loop problem is best controlled by a multivariable controller. The authors concluded that the temperature loop is functionally controllable [using decentralised or single input–single output (SISO) control, that is, proportional–integral (PI) controllers], but that the product dry mass fraction loop is not functionally controllable, and that there are fundamental limitations on the rejection of disturbances in feed dry mass fraction. These limitations follow from the long residence times which are inherent in falling-film evaporators and this problem is accentuated in multi-pass/multi-effect evaporators.

Russell *et al*. (2000) compared three approaches to modelling: (1) the effect of temperature, (2) the effect of flow rate and (c) the effect of level of concentrate at the base of each effect, for a pilot-scale three-effect, falling-film evaporator. The following were the three modelling approaches:

- An analytical model
- A modular neural network model
- A linear regression model

The plant was represented as a matrix of subsystems, such as distribution plate, evaporation tube, feed, pre-heater and venturi condenser, after the earlier work of Quaak & Gerritsen (1990). Of the process parameters being modelled (temperature, flow rate and level of concentrate), the concentrate level was the most difficult to model, and the estimates become less accurate toward the downstream sections of the evaporator. The authors found that the linear model gave the best results, followed closely by the neural network model; however, one limitation of the study was that the models were only developed and validated for pure water as the product stream, that is, product concentration was not considered.

Bakker *et al*. (2006) devised a cascade control system, using PI controllers, for controlling product concentration in a two-effect industrial-scale falling-film evaporator with thermo compression. They employed intermediate total solids measurement after the first pass as a ‘surrogate process variable’. They showed by simulation that their approach gave improved immunity to disturbances in feed concentration and also gave a more rapid response to set-point changes.

10.8.7 *Control of evaporator cleaning systems*

Effective cleaning of continuous food processing equipment is vital to remove fouling, which can be a disturbance factor in process control during production, as well as for obvious food safety reasons. The trend towards the CIP systems of cleaning by pumping cleaning solutions and water in programmed cycles at controlled temperatures and flow rates along the filled pipes or through pressure nozzles or spray balls in vessels, as opposed to disassembling the plant and cleaning manually, is well established (see also Chapter 3; Tamime, 2008). It must be remembered that a plant has a different configuration in cleaning mode than in production mode and PLCs play a key role either in setting up the plant for the different modes of operation using mix-proof valves, or in confirming an appropriate configuration of pipe work through feedback sensors. The CIP control system may operate independently of the production control system, or may be integrated with it.

A CIP control system will normally monitor supply and return temperatures, flow rate, pressure, conductivity, turbidity and pH. It will control temperature of tank contents and will control supply and return pumps and will pulse tank outlet and mixing valves to ensure sanitation. It may also be required to control the makeup of cleaning solutions and to operate retractable spray nozzles. It will operate a washing cycle consisting of a programmed sequence of rinsing, circulation and recovery or drainage of water and acid and/or alkaline solutions.

In addition to achieving higher sanitary standards than could otherwise be attained, CIP control systems lead to reduced discharges of water and detergent to drain, decreased biological oxygen demand (BOD) and chemical oxygen demand (COD) loadings, reduced downtime of equipment due to shorter cleaning cycles, optimised quantity of detergent used for each cleaning cycle resulting in significant savings and reduced potential for operator error (Anonymous, 2006).

10.9 Spray dryer control

Spray dryers use heated air to evaporate water from droplets of atomised concentrate. In general, the aims of a spray dryer control system are to maintain the safe production of milk powder of a desired quality at a consistent rate, irrespective of disturbances in atmospheric conditions and feed concentration. The control system may also play a role in cleaning the plant.

10.9.1 *Controlling the evaporative demand*

The evaporative demand is controlled by varying the concentrate feed rate and total solids. During the start-up, the total solids coming from the evaporator will be low, so the dryer feed rate must also be low to avoid putting too much water into the dryer. As a corollary, safety interlocks are usually incorporated which switch the feed over to water in the event of high outlet temperatures to deal either with feed blockages or fire outbreaks (Masters, 2002).

10.9.2 *Controlling the energy input*

The main energy input is the sensible heat of the primary drying air, which can be controlled by adjusting the inlet air temperature and the inlet air flow. As the heating system is turned on prior to turning on the feed pump, there will be an interval during which there is no evaporative load. During this time the inlet temperature must be set low to avoid damage to the chamber through metal expansion and distortion. A secondary source of energy is the sensible heat in the concentrate, which can be adjusted by varying the concentrate temperature. For dryers with integrated fluid beds, the integrated bed inlet temperature and air flow may also be adjusted.

10.9.3 *Controlling powder moisture content*

The powder moisture achieved will be a balance between the evaporative demand and the energy input. The balance is affected by factors, which influence the drying efficiency, such as the following:

- The droplet particle size distribution (determined by the density and high shear rate viscosity of the concentrate)
- The droplet low shear rate viscosity

In most plants, powder moisture content is controlled indirectly by manual adjustment involving a combination of different aspects, such as (1) chamber outlet air temperature, which is regulated by balancing concentrate feed rate at a given total solids content with a set inlet air temperature at a given air flow rate and (2) fluid bed air temperatures at set air flow rates (clearly, only the former aspect applies in the case of a single-stage dryer). The relationship between all of these parameters and powder moisture is usually determined by experience backed up by periodic sampling and off-line analysis. The limitations of this approach are set out in Section 10.9.11. An approach to the estimation of outlet temperature from the spray drying stage in a two-stage or multi-stage plant is described in Section 10.9.12.

Direct on-line moisture control depends on the use of on-line moisture analysers for powder. Such analysers are available but they depend on indirect measurements of moisture, for example, by spectral analysis, usually in the infra-red band. The types of instrument available either use a matrix of filters or perform a chemometric analysis of a continuous spectrum, for example, by fast Fourier transforms. The interface with the product may be a non-contact optical beam or a fibre-optic probe in contact with moving powder. It is important to understand that the reliability of such instruments, and of any control loop of which they are a component, depends on periodic, rigorous calibration over the entire range of measurement with representative samples. In a plant where more than one product is made, specific calibration curves must be maintained for all products.

10.9.4 *Concentrate flow rate in disc atomising dryers*

For disk atomising dryers, the concentrate feed rate is adjusted to maintain a constant outlet temperature. As long as the flow rate does not change too much, the atomisation will not

be affected greatly. Low flow rates will give slightly finer particles, and higher flow rates will give slightly coarser particles.

10.9.5 *Concentrate flow rate in nozzle atomising dryers*

For nozzle atomising dryers, the concentrate feed rate is adjusted to maintain a constant outlet temperature, but this changes the atomising pressure approximately as the square of the feed rate. Low flow rates will give low atomising pressures and coarser particles, while high flow-rates will give high atomising pressures and slightly finer particles. This can be offset in multiple nozzle dryers by turning some nozzles on and off to keep the pressure roughly constant.

10.9.6 *Inlet air flow rate*

Varying the inlet air flow with dampers or variable speed drives will directly change the amount of energy transferred to the dryer. It will also change the air residence time, the efficiency of cyclones and the pressure drop across cyclones and baghouses. In general, the air flow should be maximised to get the highest powder throughput without applying excessively high inlet temperatures.

10.9.7 *Air-flow stability in spray dryers*

Southwell and Langrish (2001) investigated the ways in which inlet air swirl affects flow stability in a pilot-scale dryer, using a chamber of 0.8 m diameter and 1.61 m height. A significant finding was that the introduction of spray has a significant effect on flow behaviour, and that air-only studies do not adequately represent the flow conditions with spray. They found that, while no single swirl vane angle resulted in behaviour that was clearly steady throughout the dryer, a swirl vane angle of about 25°, corresponding to a swirl number of approximately 0.45, gave an appropriate compromise between the degree of stability and excessive spreading of the spray cloud.

Guo *et al*. (2003) simulated turbulent flow behind a sudden pipe expansion followed by a contraction, in order to investigate the effect of downstream contraction on flow instability in non-swirling and weakly swirling flows. The length of chamber was varied from 1 to 4 diameters, while the diameter ratio (chamber diameter:inlet duct diameter) remained constant at 5. Some of the dynamic features observed experimentally were captured in the simulation. The time scale of the flow oscillation, and the interacting location between the jet and the conical bottom were in reasonable agreement with the measured values. Application of the modelling technique to gas flow in a spray drying chamber predicted a self-sustained flapping oscillation, indicating that the chamber length was not short enough to eliminate the flow instability.

Langrish *et al*. (2004) applied a time-dependent simulation to predict the transient flow behaviour in the pilot dryer that Southwell & Langrish (2001) had used. The simulations were found to be useful in observing the flow behaviour that resulted under different swirl conditions, such as double rotation mechanism in the air core. The simulations were successful in predicting the precession (i.e. the effect is equivalent to the precession of a

gyroscope when perturbed) air flow patterns, including a fairly weak and unstable precession motion exhibited under no swirl conditions up to a stronger and more coherent precession motion at 25° inlet swirl. Accurate predictions of the dependence of the direction of precession of the air jet on the inlet swirl conditions also provided confidence in the technique. The effects of increasing the inlet swirl to the critical angle for vortex breakdown was shown in this study, with the prediction of bifurcation and on-axis recirculation zones at the axis.

This study showed the value of transient models to analyse flow mechanics in spray dryers. However, more development work would be needed to extend the simulation to include the effect of the spray on internal flow behaviour and to validate a wider range of inlet swirl angles. This would require extension of the modelling approach, as the boundaries at which the model breaks down are unclear.

10.9.8 *Inlet air temperature*

Varying the inlet temperature will directly change the amount of energy put into the dryer. It will also change the initial drying rate. The maximum inlet temperature will be determined by the capability of the air heater, by the potential to impair powder solubility through heat damage, or by low powder bulk density caused by the 'ballooning' of powder particles that have dried too quickly. For example, skimmed milk powder (SMP) can often be dried at higher inlet temperatures than whole milk powder (WMP), and caseinates can be dried at inlet temperatures of over 400°C.

The response time of the inlet temperature will depend on the type of air heater. Steam coils and direct gas-fired air heater are quick to react to changes. Indirect oil or gas-fired air heaters are much slower to react.

10.9.9 *Chamber pressure*

The chamber pressure is set by the relative speeds or damper settings of the inlet and exhaust fans. The chamber pressure should be slightly negative, typically −5 mm to −15 mm water gauge. If the dryer chamber is open to external fluid beds, their hood pressure should be slightly more negative to prevent cooler fluid bed air mixing with the humid air in the dryer cone, causing local areas of high relative humidity and sticky powder.

10.9.10 *Outlet temperature in dryers without static fluid beds*

The outlet temperature is a good measure of the evaporative work done in dryers without integrated fluid beds, as the sensible heat lost from the drying air in cooling it from the inlet to the outlet temperature is equal to the latent heat of evaporation. For this reason, outlet air temperature is most commonly used as the operational variable for (indirectly) controlling powder moisture (Masters, 2002).

It is common to control the outlet temperature in dryers without static fluid beds by varying the feed rate, although heat input regulation (via air inlet temperature) is also possible. One advantage of the feed regulation approach is that it gives a faster response (Masters, 2002).

In countries, such as France and the United States where direct gas-fired air heating is often used, it is usual to run nozzle atomisers at constant pressure and to adjust the outlet air temperature by varying the inlet air temperature. In the rest of Europe and in Australasia, direct firing is uncommon, and the inlet air temperature is usually kept constant.

10.9.11 *Outlet temperature in spray dryers with integrated fluid beds*

Spray dryers with integrated fluid beds can exhibit paradoxical behaviour, such as blocking with sticky powder at the same time as producing powder with excessively low moisture content. Understanding how the temperature and relative humidity of the drying air affects powder moisture and stickiness allows such dryers to be controlled effectively.

The outlet temperature alone is insufficient to estimate the evaporative work done in dryers with integrated fluid beds or external beds exhausting into the drying chamber. In these dryers the latent heat of evaporation is balanced by two components of sensible heat, that is, heat lost from the drying air in cooling it from the inlet to the outlet temperature plus the heat lost from or gained by the integrated static fluid bed (SFB) air in cooling or heating it to the outlet temperature. When secondary drying air mixes with the primary drying air within the drying chamber, as in Niro multi-stage dryer (MSD) and compact-style dryers (CSD) and Anhydro IFB dryers, the fluid bed inlet air conditions affect the behaviour of the spray dryer. The outlet temperature represents the combined effects of evaporation cooling the primary drying air and the SFB air inlet temperature.

Both the temperature and relative humidity of the outlet air must be used for control. Typically, the outlet temperature is controlled by varying the feed rate and the outlet relative humidity is controlled by varying the inlet temperature. If the SFB inlet temperature is set equal to the outlet temperature, these dryers may be operated the same as dryers without SFBs.

Dryers with integrated static fluid beds may be thought of as single-stage spray dryers and well-mixed fluidised bed dryers sharing a common chamber.

Heat energy enters the dryer through the following:

The primary inlet air

The SFB inlet air

The concentrate

Heat energy leaves the dryer through the following:

Evaporation

Heat losses

The powder

These energy flows are in balance when the dryer is operating in a steady state. The SFB air flow constitutes a larger percentage of the total air flow in Niro MSD and Anhydro IFB dryers than in Niro or Anhydro compact-style dryers (CSD).

10.9.12 *'Dummy' outlet temperature*

If it is assumed that all the heat energy used in evaporation comes from the primary air, and that the SFB air warms the air up (or cools the air down) to the outlet temperature, a crude "dummy" internal outlet temperature may be calculated. This represents the effective temperature at the end of the first stage of drying, and may be calculated as follows:

$$T_{\text{dummy}} = T_{\text{outlet}} + (G_{\text{SFB}}/G_{\text{primary}}) \times (T_{\text{outlet}} - T_{\text{SFB}})$$

where

T_{outlet} = measured outlet temperature (°C)
T_{SFB} = measured SFB inlet temperature (°C)
G_{SFB} = measured SFB air flow (kg/h)
G_{primary} = measured primary air flow (kg/h).

This very simple calculation ignores the changes in the specific heat capacity of air with changes in temperature and humidity. It also ignores any additional air flows associated with wall sweeps, roof cooling or nozzle cooling air. It is, however, a useful tool in understanding the balance of drying between the two dryers (primary and static) sharing the same chamber. The 'dummy' outlet temperature may be used to prevent the moisture of the airborne powder getting too high and causing blockages.

Because the SFB air flow constitutes a larger percentage of the total air flow in Niro MSD and Anhydro IFB dryers than in Niro or Anhydro CSD the 'dummy' outlet temperature changes more as the SFB inlet temperature is adjusted. Examples are given below, based on the SFB air flow being 15% of the total for a CSD and 30% of the total for an MSD or IFB dryer. The same inlet, outlet and concentrate feed temperatures have been used in all the examples. Only the SFB inlet temperature has been altered to show the effect on the 'dummy' outlet temperature.

Case 1: SFB inlet temperature > outlet temperature

If the SFB inlet temperature is higher than the measured outlet temperature, the dummy outlet temperature will be lower than the measured outlet temperature. It will be heated up to the measured outlet temperature by the hotter SFB air.

Case 2: SFB inlet temperature = outlet temperature

If the SFB inlet temperature is the same as the measured outlet temperature, the dummy outlet temperature will be the same as the measured outlet temperature. The dryer behaves like a conventional two-stage dryer.

Case 3: SFB inlet temperature < outlet temperature

If the SFB inlet temperature is lower than the measured outlet temperature, the outlet temperature of the pure spray dryer will be higher than the measured outlet temperature. It will be cooled down to the measured outlet temperature by the colder SFB air. Furthermore, numerical examples are presented in Table 10.1.

Table 10.1 Examples of outlet temperature in different types of Niro spray dryers with integrated static fluid bed dryer (SFB).

Case studies[a]	$T_{SFB} > T_{outlet}$		$T_{SFB} = T_{outlet}$		$T_{SFB} < T_{outlet}$	
Dryer type	CSD	MSD	CSD	MSD	CSD	MSD
Inlet temperature (°C)	200.0	200.0	200.0	200.0	200.0	200.0
SFB inlet temperature (°C)	110.0	110.0	75.0	75.0	50.0	50.0
Outlet temperature (°C)	75.0	75.0	75.0	75.0	75.0	75.0
'Dummy' outlet temperature (°C)	68.8	60.0	75.0	75.0	79.4	85.7

CSD = compact-style dryer; MSD = multi-stage dryer.
[a]Refer to text for further information.

10.9.13 *Moisture control*

Low powder moistures are often a problem when drying heavily protein standardised powders. Low SFB powder moistures mean that the external fluid beds act as expensive powder conveyors. When making instant whole milk (full cream) powders, it is necessary to heat the external bed air after adding the lecithin to obtain good wettabilities. In this case, the powder is inevitably dried further in the external beds, and getting the final moisture high enough can be difficult.

It is common to find in Niro MSD with 'dummy' outlet temperatures below 60°C experiencing blocked SFB outlets while the moisture content of the powder leaving the SFB is below 3.0 g 100 g^{-1}. The seeming paradox is resolved when the 'dummy' outlet temperature is considered. What is happening is that very moist airborne powder is landing on bone dry powder in the very hot SFB. The powder leaving the bed is over dried, yet the airborne powder is extremely sticky. The solution is to lower the SFB inlet air temperature. This lands dryer powder on a bed doing less work, allowing the stickiness to diminish while actually increasing the moisture of the powder leaving the SFB.

10.9.14 *A model-predictive approach to the control of a spray dryer*

Perez-Correa & Fariás (1995) put forward a thermodynamic-based spray dryer model as a basis for control of single-stage dryers with centrifugal atomisers. In their design, the liquid feed rate is manipulated to control the absolute humidity of the outlet air. A feature of their design is that frequent off-line measurements of product moisture are used in conjunction with a cascade control system to compensate for disturbances and eliminate drift in moisture content.

10.9.15 *The influence of the protein content of the powder*

As a general rule, the lower the protein content in the powder, the lower the SFB temperature should be, that is, the closer the dryer will be run to a single stage dryer. The higher the protein content of the powder, for example, milk protein concentrate (MPC), whey protein concentrates (WPC) and caseinates, the higher will be the SFB inlet air temperature.

Low-protein powders

For low-protein powders low SFB inlet air temperatures are used. The moisture of the powder landing on the SFB (and going to the cyclones or baghouses) will be lower, but the amount of drying in the SFB will be less, and the moisture leaving the SFB will be higher, leaving more work for the external beds to do. Note that the lower moisture in the airborne powder reduces the stickiness, and therefore the wall deposits, even though the powder leaving the SFB is higher in moisture.

High-protein powders

For these, high SFB inlet air temperatures are used. The moisture of the powder landing on the SFB (and going to the cyclones or baghouses) will be higher, but more drying will take place in the SFB, and the moisture leaving the SFB will be lower. It is to be noted that the higher moisture in the airborne powder does not cause stickiness and wall deposits when drying high-protein powders because the protein binds the water too tightly.

Summary

For low-protein powders, the following are used:

- High outlet temperatures
- Lower SFB inlet temperatures
- Higher 'dummy' outlet temperatures
- Lower moisture airborne powder
- Little drying in the SFB

For high-protein powders, the following are used:

- Low-outlet temperatures
- High SFB Inlet temperatures
- Low 'dummy' outlet temperatures
- High moisture airborne powder
- A lot of drying in the SFB

10.9.16 *Cleaning system control in spray drying*

The trend towards automated cleaning of dryers is more recent than for evaporators, due to the fact that (1) the dryers are physically bigger than evaporators and therefore automated cleaning is a more extensive project in a dryer and (2) the surfaces in contact with dried product do not require cleaning as frequently as those in contact with wet product. Liquid contact surfaces in a spray drying plant, for example, feed lines and atomisers require the most frequent cleaning leading to significant downtime and the automation can play a role in minimising this downtime, and thus increasing the overall production capacity. Some examples of the use of CIP systems to reduce downtime are dual feed systems

where cleaning of one feed system proceeds while the other is in production, and the use of retractable spray-ball nozzles. The points made in relation to evaporator cleaning in Section 10.8.7, also apply to dryer cleaning.

10.10 Conclusion

Effective process control is essential if dairy powders and concentrated products are to be manufactured safely, in compliance with regulatory requirements, with acceptable product quality and low manufacturing costs.

The key to success is the elimination of external disturbances. This can best be accomplished by careful design of the services infrastructure. Particular attention should be directed to steam, hot, cold and chilled water, compressed air and condenser cooling towers. Complementing this approach, tight regulation of all services flows supplying the evaporator and dryer is essential.

Process control systems have to control plant throughput and product quality while minimising fouling of heat transfer surfaces. They must also be able to ensure effective cleaning of the equipment for hygiene and efficiency reasons. Product quality control is complicated by the lack of on-line sensors for many of the quality attributes of greatest interest. The laboratory analyses used to assess product quality are subject to significant error and their results are not available in real time.

The control system human–machine interface must provide the operating staff with the necessary information to assist them in interpreting causes of deviations from the normal operation.

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11 Hazards in Drying

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11.1 Background

Combustible dusts and fine powders dispersed in air can burn so fast that they give rise to an explosion more properly called a *deflagration*. If the dust-air mixture is confined within a vessel such as a spray dryer or within a building, the result can be catastrophic. This chapter outlines the principles of combustion and dust explosions, describes the tests used to characterise dusts and powders and discusses various techniques that are used to detect, prevent and reduce the effects of fires and dust explosions. The causes of dust explosions are illustrated by case studies drawn from the experience of one of the authors (Bloore, 2007).

The production of spray dried milk powders inevitably carries with it the risk of dust explosion. A combustible material is suspended in air at a concentration within the explosible range inside spray dryer chambers, the hood space of fluid bed secondary dryers, in exhaust ductwork, cyclones, baghouses and the headspace of powder storage silos, bins and hoppers during powder production and transport operations. All that is missing is an ignition source. This chapter describes many of the potential ignition sources and suggests methods of reducing or eliminating their occurrence.

Abbott (1990) provides a useful guide to managing fire and dust explosion hazards in dryers. A New Zealand Department of Labour (Anonymous, 1993) publication is specifically aimed at dairy spray drying.

11.2 Combustion

Combustion is a chemical reaction in which a fuel combines chemically with oxygen to produce combustion products and heat. In spray drying dairy products, the fuel is the dried dairy product that is being manufactured. Oxygen is supplied from the air. Combustion products include inert solids (ash), gases (CO_2, CO and HCN), water vapour and pyrolysis products, which have a characteristic odour.

All three elements, such as fuel, oxygen and heat, must be present to initiate a fire or dust explosion. Removing any one will extinguish the fire or dust explosion. This relationship is well-known as the fire triangle.

- All food products are combustible, so fuel will be present within a milk powder plant whenever it is in operation. Good housekeeping is required to minimise the hazard from dust inside buildings but outside the processing equipment.

- There must be sufficient oxygen present to sustain combustion once ignition occurs. Air supply and extraction fans and the powder conveying blowers maintain a constant supply of fresh air whenever the plant is running.
- All that is missing from the fire triangle is heat, and careful plant design, good maintenance and alert operators can eliminate external sources of ignition; powders and dusts containing both proteins and sugars (such as dairy powders containing caseins and/or whey proteins and lactose) are capable of self-ignition.

11.2.1 *Smouldering combustion*

This is slow, inefficient burning without a flame. It may take place on the surface of powder or dust, or within a mass or layer of the powder. Smouldering powder may burst into flame hours or even days after ignition. Smouldering combustion typically gives off carbon monoxide (CO), which can be used to detect a fire at an early stage (see LiVun *et al.*, 2006).

11.2.2 *Flaming combustion*

Flames occur when sufficient vapour is generated as a product of combustion or when volatiles are driven off by the heat. The vapour burns with a luminous flame. Flames may have a stationary flame front fed by the vapour emitted from a burning mass of powder, but in a deflagration the flame front moves rapidly through a pre-mixed fuel-air mixture.

11.2.3 *Deflagrations*

A deflagration is a rapid fire where the exothermic reaction, involving air or some other gaseous oxidising agent, spreads throughout the burning material (the fuel), accompanied by the liberation of large amounts of gases (Bartknecht, 1989). Flame speeds vary from a few m s^{-1} up to several hundred m s^{-1}. The heat generated by the rapid combustion rapidly expands the air. If this takes place in a confined space, the pressure will rise sharply. The fuel may be a gas or vapour, a liquid mist, or a fine dust or powder. In the present context, the fuel is a dairy powder and air supplies the oxygen.

In a deflagration, the ignition source sets fire to a few nearby particles, which in turn set fire to a few of their neighbours. It is to be noted that this is a chain reaction, and anything that breaks the chain will stop the development of an explosion. When the particles are too far away from each other, the chain peters out. This is the case for dust concentrations below the lower explicable limit. Conversely, when the dust concentration is too high, the closely packed particles absorb the heat from the ignition source without enough of them getting sufficient energy to ignite. If inert particles are mixed with the dust immediately after the initial ignition, they will absorb sufficient heat to prevent the chain reaction propagating. This is the basis of dry powder explosion suppression systems.

11.2.4 *Detonations*

When the flame speed exceeds the speed of sound, the explosion is called a detonation. Shock waves compress the unburned fuel-air mixture ahead of the flame front, causing the flame front to accelerate. Detonations are extremely dangerous, but solid fuelled detonations are largely confined to metal dusts and are not known to occur in the manufacture of spray dried dairy products.

11.2.5 *Secondary explosions*

Primary explosions often disturb powder or dust deposits in the vicinity, blowing them up into a cloud. The flame front from the primary explosion then ignites the fresh fuel. Secondary explosions are often more violent than the primary one. For example, a primary explosion in a fluid bed might vent a cloud of burning dust into a building. The pressure wave will disperse dust from beams and ledges into the air. The mixture may then explode, destroying the building.

11.3 Dust characteristics

Materials differ in their combustion and deflagration behaviour. In order to assess the hazards and to design a plant to process, handle and store powders and dusts, the relevant characteristics of the particular material must be determined. Standard laboratory tests are used for this purpose.

11.3.1 *Combustibility/explosibility*

The material making up the dust or powder must be combustible. There are laboratory tests for this, but all food products are combustible, so combustibility can be assumed. All combustible materials will undergo deflagration if they are sufficiently fine, are suspended in air at an appropriate concentration and are exposed to a sufficiently energetic ignition source. The rate of reaction will depend on the chemical composition, the particle size of the material and its temperature.

11.3.2 *Upper and lower explosible limits*

The upper and lower explosible limits are the highest and lowest concentrations that will ignite and propagate a flame, that is, the richest and leanest fuel-air mixtures which can deflagrate. They are tested in a vertical tube apparatus. A known quantity of powder is placed in a dish and dispersed in a puff of compressed air. It is ignited by an electrical spark or a hot filament. The lower explosible limit is referred to as the minimum explosible concentration (Bond, 1991).

For most milk powders, the lower explosible limit is 60 g m^{-3}. This corresponds to 12 teaspoons of powder in a volume of 1 m^3. For comparison, icing sugar has a lower

limit of 15 g m^{-3}, or only 3 teaspoons dispersed in one m^3. The upper limit is less well-defined, but is usually between 2000 and 6000 g m^{-3}, that is, 2–6 kg m^{-3}. This is about 100 times lower than the concentration of powder found in the bed of a fluid bed or in a powder silo. Such dense concentrations of powder will not explode unless they are dispersed in air at concentrations between the upper and lower explosible limits.

11.3.3 *Minimum ignition temperature*

The minimum ignition temperature, sometimes referred to as *autoignition temperature*, is the lowest temperature at which a sample will spontaneously ignite. It is not an exact temperature and depends on the type of apparatus used (Bond, 1991). In particular, it depends on the source of energy used to trigger combustion, for example, an electrical spark or a hot surface, and whether the dust is in a static layer or suspended in air at the point of ignition. The Godbert–Greenwald furnace is used to measure the minimum ignition temperature of dust clouds. In this apparatus, a vertical cylindrical ceramic tube is heated to a known temperature and a sample of the powder is dispersed in the tube by means of a blast of air (Eckhoff, 1991). This is repeated at different temperatures until the minimum ignition temperature is identified. An alternative apparatus, working with the same principle, is known as the 1.2 L Furnace (Conti & Hertzberg, 1986).

Minimum ignition temperature of dust deposits or layers can be determined either by a hot-plate test or in a furnace (or 'oven test'). In the hot-plate test, a layer of dust of a defined height, typically 5 or 15 mm, is placed on a hot-plate surface which is brought to a controlled temperature and held for a period of time, during which the powder temperature is monitored and recorded. This is repeated at different temperatures until a thermal runaway reaction is observed. For a result to be recorded as an ignition, the dust temperature must exceed the hot-plate temperature by at least 20°C (Eckhoff, 1991).

As powder starts to burn, it releases heat. This heat will escape into the environment at a rate that depends on the temperature of that environment, and on how well insulated the powder is. For ignition to take place, the rate of heat release must exceed the rate of heat loss by enough to heat fresh fuel up to the ignition temperature.

This temperature will usually be much higher in a cloud of dust or powder than in a layer of the material. The thicker the layer of powder, the better insulated it will be, and the lower the temperature at which it will ignite. A dispersed cloud of skimmed milk powder (SMP) requires a temperature of 490–500°C, while a similar cloud of whole milk (full cream) powder (WMP) requires a temperature of only 440°C. Layers of powder will ignite at much lower temperatures. The greater the thickness of the layer, the lower the temperature at which it will self-heat to eventual ignition.

11.3.4 *Minimum ignition energy*

The minimum ignition energy is usually determined using an electrical spark as a source of ignition in a vertical tube apparatus, in which a pair of high-voltage electrodes is installed. A sample of powder is dispersed into the tube by a blast of air and a high voltage is applied to the electrodes, causing a spark to be discharged between them, via a capacitor. The energy stored in the electrical circuit, prior to discharge is generally taken as a measure

of the spark energy, for example, 0.5 CV^2. There are several spark discharge circuits in use for this purpose (Eckhoff, 1991), which result in slightly different ways of going about the test, for example, connecting more capacitors in parallel, and/or adjusting the applied voltage, to increase the spark energy until ignition is observed. Having observed an ignition, the test is repeated at progressively lower energies until ignition no longer takes place. Values below 15 mJ indicate a risk of ignition from static discharge sparks. Milk powders generally have minimum ignition energies above 50 mJ (Eckhoff, 1991; Anonymous, 1993), so static discharge sparks are not usually a major risk factor in dairy plants. Static electricity has been responsible for serious fatal deflagrations in other industries, so anti-static precautions, such as bonding and earthing equipment, ductwork and flexible connections, are important.

11.3.5 *Maximum explosion pressure and the rate of pressure rise*

Gas liberated during the combustion and the effect of the heat from the explosion in expanding the atmosphere cause a pressure build up if the explosion is confined. The maximum explosion pressure (P_{max}) and the normalised maximum rate of pressure rise (K_{st}) are determined using an explosion sphere. The most common size is 20 L, but 1-m^3 and 3-m^3 spheres are also used. The test sphere is partially evacuated to a pressure of about 0.04 MPa (0.4 bar) absolute. The powder sample is blown into a cloud inside the sphere by just sufficient compressed air to bring the pressure to 0.1 MPa absolute at the time the powder–air mixture is ignited. The time evolution of the pressure P within the sphere is recorded. From this record, two parameters can be derived; the peak pressure P_{max} and the maximum rate of pressure rise dP/dt.

P_{max} values are generally from 0.5 to 0.9 MPa gauge. A typical design P_{max} for dairy powders is 0.8 MPa gauge.

The K_{st} value is determined from the maximum rate of pressure rise. It is numerically equivalent to the maximum rate of pressure rise in a 1-m^3 vessel. It is defined by the formula:

$$K_{st} = (dP/dt \;.\; V^{1/3}) \text{ bar m s}^{-1}$$

where dP/dt is the maximum rate of pressure rise expressed in bar s^{-1} and V is the vessel volume in m^3.

It is a measure of the severity of the explosion, and it is used to classify dusts as follows:

Class	K_{st} value
St 0	0 MPa m s^{-1} (0 bar m s^{-1})
St 1	<20 MPa m s^{-1} (<200 bar m s^{-1})
St 2	20–30 MPa m s^{-1} (200–300 bar m s^{-1})
St 3	>30 MPa m s^{-1} (>300 bar m s^{-1})

A typical design figure for milk powders is 13 MPa m s^{-1} (130 bar m s^{-1}).

11.3.6 *Particle size*

The particle size of the powder or dust determines the surface area available for combustion. The smaller the particles of a given quantity of material are divided into, the greater is their collective surface area. Spray dryers exploit this to achieve rapid drying by atomising the feed into small droplets. Halving the average droplet size will increase the number of droplets eight-fold and double the total surface area. For example, if the particle density is 1000 kg m^{-3} and, if the powder has a mean diameter of 120 μm, it will have a specific surface area of 50 m^2 kg^{-1}. Halving the mean particle diameter to 60 μm will double the specific surface area to 100 m^2 kg^{-1}.

The total surface area of the powder airborne within a spray dryer chamber may be calculated as follows. Spray dryers typically have powder residence times of about 30 s, so about 1/120th of their hourly powder throughput will be airborne at any given time. A 1-tonne per hour dryer will, therefore, have about 8.3 kg of airborne powder, while a 20-tonne per hour dryer will have 166.7 kg of airborne powder. Multiplying these masses of powder by the specific surface area for 120 μm diameter particles gives total surface areas of 417 m^2 for a 1-tonne per hour dryer and 8333 m^2, or 0.83 hectares for a 20-tonne per hour dryer.

Dust clouds explode because the enormous surface area allows extremely rapid combustion.

11.3.7 *Moisture content*

The moisture content of the dust or powder must be sufficiently low to allow rapid combustion. Evaporation of the moisture absorbs heat energy otherwise used in propagating the fire through the cloud of powder. Moisture contents over 15 g 100 g^{-1} are required before the explosibility begins to decrease. Moisture contents over 30 g 100 g^{-1} are required to make a deflagration unlikely (Lunn, 1992). These are far in excess of milk powder moisture contents, so all parts of a milk powder plant will contain potentially explosible materials. Eckhoff (1991) suggests that the minimum ignition energy may increase 10-fold between 1 and 15 g 100 g^{-1} moisture for maize starch. Likewise, the explosion severity seems to decrease with increased moisture.

11.4 Ignition sources

Almost all of the following ignition sources have been responsible for initiating fires and/or dust explosions in dairy spray drying plants (Bloore, 2007).

11.4.1 *Flames*

Flames from burning alcohol used to sterilise powder sampling scoops have ignited dust explosions in fluid bed dryers. The flame is difficult to see, and in at least two cases, flaming scoops were plunged into the fluid beds, setting them on fire. In one case the dryer was destroyed.

Cigarettes are an obvious fire hazard and should not be used in the food processing plants. Moreover, hand-held gas torches have ignited powder deposits inside mild steel exhaust ducts and stacks during maintenance or equipment installation activities. The resulting chimney fires caused more embarrassment than damage because the fires were unable to burn back towards the dryers.

11.4.2 *Hot surfaces*

The temperature of hot surfaces in spray dryers and powder handling equipment are generally far too low to ignite a dust explosion directly. What usually happens is that a layer of powder builds up on a hot surface and then self-heats to the ignition temperature. Liquid dripping, splashing or sprayed on to hot surfaces in spray dryer air dispersers have caused many explosions once a layer of material has built up and self-heated.

A fairly common example is when the 'O' ring is left out of a union in the concentrate feed line to a rotary atomiser. Concentrate leaks out, runs down the atomiser well, past the rubber atomiser mounting ring and then runs down the outside surface of the atomiser. This surface is exposed to the full inlet temperature of the spray dryer, so the concentrate dries out and builds up in a layer, which chars and will eventually ignite. To minimise this risk, it is recommended that spillage detectors be fitted.

Another example, which has caused one major dust explosion incident in New Zealand and another in Australia, applies to certain types of nozzle atomising spray dryers. In these dryers, high pressure stainless steel tubes with a nozzle mounted at the end are installed in the air disperser, pointing vertically downwards. These tubes are variously known as lances, rods, wands or sticks in different parts of the world. The upward thrust on the lance is the product of the mass flow rate of the concentrate though the lance and the downward component of the velocity with which the spray leaves the nozzle at the end. This upward thrust is countered by the weight of the lance and its liquid contents. In some dryers, the thrust exceeds the weight by a substantial margin. Often the nozzle at the end of the lance is angled about 10° to allow the direction of the spray to be adjusted. If the lance is unfastened to adjust the direction of the spray while the dryer is running, the lance can rise unexpectedly and spray the concentrate on to hot surfaces inside the air disperser. In some dryers, the spray will impinge on the central fines return pipe, while in others it will impinge on the air disperser wall. In either case, the concentrate will dry, self-heat and then provide an effective ignition source for a dust explosion. Safety clips have been devised by some manufacturers and dairy companies to allow lance adjustment without the risk of the lance rising.

Lights fitted to dryer sight glasses have caused ignition when a layer of product built up on the inside of the sight glass and self-heated to ignition temperature. Halogen lamps generate intense heat, and the insulating properties of a layer of milk powder on the inside of the glass allow the glass to get extremely hot. All sight glass lights should be fitted with a time switch so that they cannot be left on for long periods.

Some dryer installations heat the explosion vent ducts of their chamber with a fan recirculating air through the electrical heating elements. In at least two cases, the powder has found its way into the explosion vent duct through leaking explosion doors. Enough powder accumulated to be drawn into the heating system where it caught fire and initiated

a dust explosion. In one case, the explosion blew into the dryer, setting off a deflagration in the chamber.

Hot surfaces in heat sealers present potential sources of ignition, if a layer of product is allowed to build up and self-heat to ignition temperature. Good dust extraction systems in packing equipment should prevent this from causing problems.

Particular attention should be paid to the location of hot air hand dryers within spray drying plants. They must be kept away from any area in which a dust cloud could form in an accident. In one such instance, a packing room was destroyed by a dust explosion when about a tonne of powder fell into the room through a perished rubber flexible connection between a hopper and a packing head. There was a hand basin and electrical hand dryer at the far end of the room. The dust cloud generated by the falling powder was dense enough to break the light beam to the photocell used to detect the presence of hands. The fan and the heating element turned on, drew the powder into the element and ignited a dust explosion. All the walls were blown out and the roof lifted momentarily before falling back on to its supporting columns. Fortunately, the room was unoccupied at that time, so there were no injuries.

Overheated electrical equipment to which the dust has access can ignite a fire. Observance of the relevant electrical safety standards should remove this hazard.

11.4.3 *Mechanical friction*

Following are the examples of mechanical friction that can cause ignition of powder and result in explosion:

- Hot bearings in bucket elevators and belt conveyors are common causes of fires and dust explosions in many industries, including the milk powder industry.
- Overloaded rotary valves and screw conveyors can generate intense heat. If these are left to run under layers or heaps of powder, fires can be started.
- Slipping fan belts can set fire to the belts and paintwork on belt guards. This may lead to fires external to the equipment if housekeeping is poor.
- Flooding of rotary atomiser distributors can lead to frictional heating of congealed concentrate, causing a fire and/or fracture of the atomiser shaft.
- Stray bolts or other metal objects can get caught in rotary sifters. They may stay in the sifter long enough for much of their material to be worn away before the stainless steel mesh burns through from the friction.

11.4.4 *Impact sparks*

Foreign objects, such as tools, tramp metal and dislodged equipment parts, can generate impact sparks in mills, sifters and conveyors, and in falling into silos, hoppers and other equipment. This is not a major cause of fires in dairy plants because most of the materials are stainless steel, which do not spark easily. However, the impact of aluminium spanners on rusty mild steel will cause an extremely energetic thermite spark.

Sparks from the angle grinders will not usually have sufficient energy to initiate a dust explosion in milk powders, but they may cause a smouldering fire in a heap of powder if they land on it for an extended period.

11.4.5 *Electrical sparks*

Electrical equipment can provide both hot surfaces and electrical sparks from contactors, switches and motor brushes, which are capable of igniting dust explosions. The techniques are used to prevent this are as follows:

- Intrinsic safety
- Dust-excluding ignition-proof (DIP) enclosures
- Isolation of equipment

Electrical standards cover the requirements for electrical equipment for use in the presence of combustible dust. It should be noted that a 'flameproof' equipment is NOT necessarily suitable for use in dusty environments.

Some electrical insect killers use ultra-violet light to entice insects to fly between highly charged metal grids, where they are electrocuted by a spark discharge. These must be kept away from packing rooms and any process area where dust clouds could form in an accident.

The use of portable electric power tools and vacuum cleaners must be restricted, and pneumatic or totally enclosed motor powered equipment must be provided for use in dusty areas, or areas where dust may be present in the event of an accidental spillage.

11.4.6 *Electrostatic discharge sparks*

Electrostatic sparks are usually too low in energy to ignite milk powders, but good electrical bonding of all pipes and ducts joined by flexible connectors is still important. Thick gauge, insulated multi-strand copper wires are preferred over open braided earth straps, which present hygiene and product contamination hazards. Conductive filter bags are available for baghouses. When the powder falls across long distances, such as when a powder silo is being filled by a pneumatic conveying system, a considerable static charge may build up.

11.4.7 *Hot work*

Hot work such as welding, cutting and grinding can ignite dust explosions and may also ignite flammable material which can smoulder for hours before bursting into flames. Good hot-work practices and good housekeeping are essential to prevent this.

Buildings made from expanded polystyrene sandwich (EPS) panels are particularly susceptible to ignition by welding operations. Several alternative materials, such as polyphenolic resins and polyisocyanurate, are now available for use in lightweight sandwich panels. These char rather than melt, and have fire ratings. Fires caused by hot work near the EPS panels have destroyed or severely damaged several dairy factories.

Welding operations on the outlet chute from an integrated fluid bed dryer set off a dust explosion within the dryer. The explosion-venting doors and the deluge system limited the damage and the loss of production from the dryer. A hot-work-permit system was in operation in the factory, but it failed to prevent the incident. It is helpful if the only access to the dryer building is through the control room, as staff members are more likely to notice the presence of an unauthorised worker.

11.4.8 *Self-ignition*

At high temperatures, dairy powders containing both protein and lactose degrade through Maillard browning. Fat-containing powders also undergo oxidation. Both these chemical reactions are exothermic, releasing heat at a rate which increases exponentially with temperature. This is known as *self-heating*. Powders are good insulators, slowing the rate of heat loss to the surroundings through the exposed surface. The thicker the layer of powder on a surface, the less will be the surface area available for heat loss relative to the volume of powder. The higher the temperature of the surroundings, the slower will be the heat loss.

When the rate of heat generation exceeds the rate of heat loss, the temperature of the powder rises. This further increases the rate of heat generation. The result is thermal runaway, followed by smouldering combustion, ignition and a fire and/or an explosion. As an example (Beever, 1985), for SMP,

a 150-mm thick layer will self-ignite at 100°C;

an 11-mm thick layer will self-ignite at 200°C.

Self-ignition is a very common cause of explosions because staff members are usually careful to avoid external ignition sources. It has been reported (Steenbergen, 1991) that in Europe, 80% of spray dryer fires were initiated by the smouldering of powder.

Self-ignition can take place whenever powder is allowed to build up in a thick layer in warm or hot surroundings. An example of a fire and explosion caused in this way follows an incomplete clearance of a blocked cyclone. The 3-m diameter cyclone was left with a plug of powder, perhaps 2-m thick, sitting in it at an initial temperature of 70°C. Over a 6-h period during which the dryer was off, the centre of the mass of powder self-heated to ignition temperature. The smouldering powder was completely surrounded by cooler powder, giving no external signs of a problem. When the dryer was restarted, the cyclone powder was pneumatically conveyed to a fluid bed mounted under the chamber. A lump of hot powder was dislodged from the previously blocked cyclone and found its way to this fluid bed, where it was exposed to a draft of warm air which fanned it into flames. As soon as the first powder landed on the bed, a small dust explosion took place. Three successively larger explosions followed over the next 25 s as powder deposits on the interior of the dryer chamber were disturbed and formed fresh dust clouds. The sequence is illustrated in Figure 11.1.

Another example of self-ignition occurred when a particularly sticky powder known colloquially as *gunpowder* was dried on a tall-form spray dryer fitted with an external fluid bed secondary dryer. On five different occasions, a large lump of powder formed in the fluid bed over a period of several hours. The centre was heated by self-ignition while the outside remained at the fluid bed air temperature. When the lump eventually bumped against the weir at the end of the bed, it broke open, exposing the red hot centre to the dust cloud caused by the cyclone fines being pneumatically conveyed to the end of the bed. The resulting explosion blew back into the chamber, opening the explosion doors.

A further example of self-ignition illustrates the importance of controlling the temperature of the dryer roof. The photographs in Figure 11.2 show how quickly self-ignition can

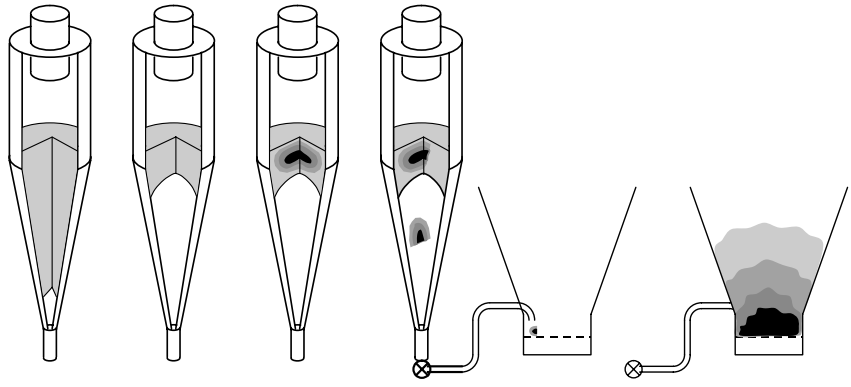

Fig. 11.1 Sequence of diagrams showing powder in an incompletely cleared blocked cyclone self-heating to ignition and causing a dust explosion.

begin. These show a general view and a close-up view of heavy powder deposits formed on the roof of a clean spray dryer within 30 min of starting on the product. A baffle had become detached and allowed cold roof cooling air to impinge on part of the roof. The relative humidity of the air immediately next to the roof increased because of its low temperature. This caused airborne powder to stick to the roof. As the powder layer built up, it insulated the roof, allowing it to cool still further, with the temperature of the roof eventually cooling to the dew point of the air within the powder layer. Within 30 min, part of the powder deposit became over 50 mm thick and turned to a dark chocolate brown. This time the potential ignition source was prevented from causing an explosion, but an over-cooled dryer roof of a nutritional products dryer caused three explosions over a 6-week period before the cause was identified and the fault rectified.

If large quantities of powder are held for long periods in filters and baghouses, self-ignition can occur. The finer particle size of the baghouse powder can encourage self-ignition at air temperatures below 100°C.

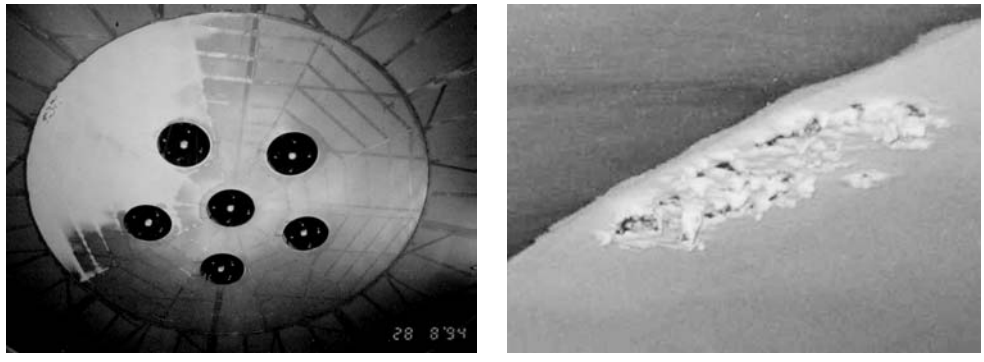

Fig. 11.2 Self-heated roof deposits caused by excessively low roof temperature.

11.5 Hazards of dust explosions

A dust explosion in a plant can kill or injure in several ways:

- The flame will burn the exposed two-pot epoxy paint back to the bare plaster on concrete walls and to the galvanising on handrails, so normal work clothing will be of limited value in protecting human flesh.
- The blast can blow the personnel off work platforms, walkways and stairs, causing gravity-induced injuries.
- The blast can pick up hatches, blast shields and loose equipment items and propel them to considerable distances with great force, causing impact injuries.
- The smoke ejected from the hatches, flexible connectors and the ruptured equipment items can fill even a large building in seconds, choking the personnel and reducing the visibility hindering timely evacuation.
- The over-pressure can cause structural failure in a vessel or building. Damage may include rupture of equipment, loss of cladding from a building and partial or total collapse of the building structure. Falling debris creates an obvious hazard.

It should be noted that it is entirely possible that the explosion will expose the personnel to two or more of these hazards at the same time.

11.6 Fire detection

All fire sensors and their alarm systems must be completely independent of the process control system with their own independent uninterruptible power supply. Regular tests of the systems are essential.

11.6.1 *Fast-acting temperature sensors*

These are the most common means used to detect fires in spray dryers and fluid beds. Multiple sensors are used to ensure a high probability of fire detection. Correct location is important. For example, if there is no sensor on the exhaust duct from an external fluid bed, a fire in the bed will not be detected. By the time the temperature sensors detect a fire, it will be well established, and a deflagration may follow before the dryer can be shut down and a deluge initiated.

11.6.2 *Infra-red optical detectors*

Infra-red detectors can sense sparks or hot spots with a reaction time of only a few milliseconds. They are very effective in dilute powder suspensions, or where the detection distance is short. Trials have demonstrated that the infra-red detectors are unable to see through the powder cloud inside the dairy industry spray dryers. They have been used in conjunction with the raised rate of pressure sensors to trigger explosion suppression systems in fluid bed dryers. They have also been used to protect sifters by sensing the embers dropping from the fluid beds.

11.6.3 *Carbon monoxide detectors*

Smouldering powder often gives off carbon monoxide (CO) because of inefficient combustion in the early stages of a fire. This can be detected in the exhaust air (Steenbergen *et al*., 1991; see also LiVun *et al*., 2006). If 1 kg of powder smoulders completely in 1 h in a dryer having an air flow of 100 000 kg h^{-1}, the CO production will be 1 mg kg^{-1}, which can be detected reliably by sensitive detectors.

These detectors are easily triggered by, for example, vehicle exhaust fumes drawn into the building ventilation system, high background CO levels due to air pollution, or CO from nearby fires from burning off the stubble or other materials. For this reason, the CO levels are measured in both the dryer inlet and outlet airstreams. Any CO entering the dryer will mix with the air already in the chamber, so its concentration in the outlet air will be spread out over time. This means that simply taking the instantaneous difference between the inlet and outlet CO concentrations will not be appropriate for use in the alarm system. Different systems use different techniques to address this issue.

It is not uncommon for conveying air blowers and fluid bed air handling units to draw their air from within the building. This air leaves through the exhaust system, so any CO present will register on the outlet, but not necessarily at the main inlet. A fire or an internal combustion-powered vehicle inside the building may trigger an alarm. The CO detection system must take account of these factors. Allowance must be made for the air residence time in the dryer and the effect of mixing the externally and internally generated CO in the outlet air when comparing the inlet and outlet CO concentrations.

If direct gas-fired air heaters are used, some CO may be present if the burner is not correctly adjusted, and this, too needs to be allowed for.

Air is sampled by means of valves and filters, and is delivered to a central measurement unit. The instrument looks for short duration ‘spikes’ in CO level, typically lasting for only a minute or two. This corresponds to the initial smouldering releasing CO followed by a more complete combustion to CO_2, with less CO being generated.

CO monitoring units have proved very successful in detecting fires early enough to allow the plant to be shut down in time to avoid a dust explosion. In spray dryers with capacities of 20 tonnes per hour and above the measurement of minute increases in CO level becomes difficult due to the diluting effect of the enormous quantities of air. This has led to attempts to use other components from the gas phase for detection. Notwithstanding the great dilution factor, a CO monitoring system was able to detect an incipient fire in a 25-tonne per hour spray dryer in New Zealand.

11.6.4 *Pressure sensors*

Pressure sensors can detect the initial pressure rise associated with a deflagration in time to trigger suppressions systems. There are typically two or more sensors arranged so that the chance of simultaneous activation by accident is minimised. Some sensors simply have a pressure threshold above which they trigger a suppression system, but others calculate the rate of pressure rise. These sensors must be installed so as to avoid accidental triggering by, for example, the impact of CIP solutions from spray balls.

11.6.5 *Operator observation*

Early indications of a potential fire and explosion are as follows:

- Burning smell
- Scorched or charred particles on pads
- Dark coloured crunchy lumps in the sifter of over-sized products
- Flames inside the equipment

Spray dryers run with a negative chamber pressure, so any smell of burning will have to leave the dryer exhaust stack, be wafted around the building and enter the ventilation intakes before any one inside the building will have a chance to notice it. Prompt action when a burning smell is noticed is vital.

Powder may take 30–40 min to travel through the fluid beds before it reaches the sifter, where hourly samples are usually taken for scorched particle pads. Taking a sample from the chamber base helps cut down this delay, but prompt action is still vital if dark pads are observed. Running out for a re-test sample is not advisable, as the smouldering fire will have been going for some time, and the re-sampling puts the staff at risk and wastes precious time.

11.7 Explosion suppression

Because a deflagration is a chain reaction in which the burning particles ignite each other, it is possible to interfere with the flame propagation and break the chain. All suppression systems rely on the early detection of the deflagration.

Explosion suppression is an attractive alternative to explosion venting when protecting fluid bed secondary dryers, which are usually installed too far from the exterior walls to be vented outdoors. Venting inside a building requires that the building itself be vented, and that staff be physically prevented from accessing areas in the path of the flame. This can make maintenance access difficult, and may encourage dangerous practices to circumvent the safety systems in order to accomplish a maintenance task without having to stop the plant.

11.7.1 *Dry powder suppression*

Sodium bicarbonate or another food-grade inert powder is used in dry powder suppression systems in food dryers. The powder is stored in a container pressurised by nitrogen gas. Rapid-acting pressure sensors trigger a valve, which discharges the powder into the spray dryer or fluid bed. The inert particles mix with the dust or powder particles and do not pass on the deflagration flame front, halting the spread of the deflagration. The dry powder will settle relatively quickly after it has been discharged, so immediate shutdown is essential to prevent the re-ignition. The system must be disarmed before the personnel enter the vessels protected by such equipment.

11.7.2 *Chlorinated fluorocarbon compounds*

Chlorinated fluorocarbon compounds (CFCs) were once widely used as suppressants because they interfere with the free radical reactions within the flames. They have been phased out because of their adverse effects on the ozone layer.

11.7.3 *Pressurised hot water*

Pressurised hot water explosion suppression systems use sealed bottles of water heated to about 180°C, which corresponds to a pressure of 1 MPa gauge. When the start of an explosion is detected by a pressure sensor, a valve opens allowing about 15% of the water to flush into the steam, which propels the rest of the water into the developing fireball as a fine mist of boiling water. The water droplets cool the fireball by evaporation and the mist absorbs infra-red heat energy, further helping to suppress the explosion.

Because the density of water is higher than the bulk density of dry powders, the recoil from the discharge of a pressurised hot water system is significantly greater, and care must be taken to allow for this when installing these systems. Pressurised hot water systems are simple to reinstate after use and they can be made safe by allowing the water to cool below 100°C.

11.8 Explosion venting

Explosion venting is a last resort measure. The objective is to prevent the pressure within the vessel or building from exceeding the design strength. There are two approaches to venting:

- Explosion pressure-resistant design, the design of the vessels and equipment is such that they can withstand the expected explosion pressure without any permanent deformation.
- Explosion-pressure-shock-resistant design, the design of the vessels and equipment is such that they may be permanently deformed by the explosion but will not rupture.

11.8.1 *Venting principles*

If there is no venting, the explosion over-pressure will head towards the maximum pressure P_{max}, bursting the vessel as it exceeds its strength. With a door or panel arranged to open at a static opening pressure P_{stat}, the explosion pressure overshoots to a reduced maximum explosion pressure P_{red}, which is typically two-thirds of the burst pressure.

The size of a vent required may be determined using methods set out in the standards and codes of practice, such as the German Verein Deutscher Ingenieure VDI 3673 (Anonymous, 2002) and the United States National Fire Protection Association NFPA 68 (Anonymous, 2007). These codes are updated every few years, and the most recent edition should be used when designing the explosion-venting systems.

11.8.2 *Vent ducts*

Any duct downstream of a vent will restrict the exhaust of combustion gases, and the unburnt dust may be ejected into the duct, where it will burn and produce a 'head wind' for the escaping explosion. In such cases, the vent area must be increased to compensate. The more complete the combustion in the vessel before it vents, the less unburnt powder will be ejected. This reduces the effect of the vent duct on the reduced explosion pressure when strong vessels with high P_{red} values are vented.

Vents must discharge to a safe location. The fireball emerging from a vented deflagration can reach as far as 60 m and be over 10 m in diameter. It is usual to install large plant items like spray dryer chambers and baghouses close to the external walls of buildings. This means that short vent ducts can be used. Vent ducts must be kept reasonably short to keep the reduced maximum pressure within the vessels within practical limits. Other items, such as cyclones and fluid bed secondary dryers, are usually installed further away from exterior walls, making ducting of the explosion difficult or impossible. Fluid beds are often vented inside dryer buildings and, when they discharge, they can fill a dryer building with smoke within 2 s. Suppression systems are gaining in popularity as an alternative to venting in these applications.

Explosion-venting requirements can have a significant influence on the building design. Particular care must be taken to avoid venting explosions close to walkways and fire escapes. If this cannot be avoided, the walkways must be shielded from the blast. It is unwise to vent explosions immediately below building air intakes, as there is a very real risk of 'inhaling' the explosion products. This may burn out the intake filters, fill the building with smoke, or even cause a second explosion in the dryer. Powder storage bins are often vented into a bin room, which is then vented to the outside of the building. This ought to make the upper part of the bin room a 'no-go' area, but staff will occasionally need access during the powder transport operations. Recent New Zealand factory designs have allowed bins to be vented externally, making it safe to work on the powder transport system at any time.

11.8.3 *Vent doors and panels*

Many older dryers have comparatively heavy hinged explosion doors installed in their cylindrical wall. Allowance must be made for the inertia of such doors when calculating the reduced maximum explosion over-pressure. They can be reused in all but the most extreme explosions. Doors are usually held closed by shear pins or spring loaded catches so that they will open at a pre-determined pressure. Doors have a tendency to leak, which may cause hygiene concerns. If an exhaust fan trips, the momentary over-pressure in the chamber may partly open an explosion door, blowing powder into the vent duct. This will not necessarily activate the proximity switches fitted to the doors. If this sort of problem persists, it is very tempting to replace the shear pins with bolts, rendering the doors ineffective in a real explosion.

Modern dryers and baghouses tend to have single-use explosion panels. These are extensively tested; they are more reliable in operation; and they do not leak. Panels are often insulated to avoid cold spots on the inner skin of the dryer, which would lead to powder

deposits. Domed panels are preferred for venting powder bins where the pressure will fluctuate as the pneumatic conveying system operates.

11.9 Containment

Small plant items like mills may be made strong enough to withstand the maximum explosion pressure P_{max}. This is not practical for large items like spray dryers, fluid beds, baghouses and powder silos.

11.10 Isolation

Explosion-proof rotary valves, quick-acting slide valves, extinguishing barriers, explosion diverters and chokes, such as screw conveyors, may be used to isolate various parts of the plant from each other in the event of an explosion. This prevents a fire or explosion in one area from spreading.

A good example of the value of isolation was seen when the burning powder deposits originating in the air disperser of a large New Zealand spray dryer fell into the static fluid bed and passed through a rotary valve into a series of external fluid beds. The burning powder ignited a dust explosion at the end of the first external bed, where returning fine powders had created an explosible mixture of powder and air. Two of the three external fluid beds were damaged, their cyclones were set on fire and the building was extensively damaged by fire, but the rotary valve prevented the explosion from passing back into the spray dryer, thereby preventing more catastrophic damage.

It is vital that rotary valves are stopped and valves are operated immediately the fire or explosion is detected. If not, they will simply meter out the ignition sources to adjoining plant so that the whole plant is affected. Stopping all fans and blowers as soon as a fire is detected will slow the spread of the fire and also reduce the chance of a fire developing into a deflagration.

11.11 Inerting

Explosions can be prevented by modifying the atmosphere within plant items containing an explosible concentration of dust or powder. This usually means reducing the oxygen content to a level below which a deflagration will not propagate. This is not practical in the dairy industry, and it brings with it the risk of suffocation of the personnel.

11.12 Fire fighting

Fires in powder handling plant must be fought with great care to avoid disturbing the powder. Water should not be directed in a jet at the burning dust because it will generate

an explosible concentration of the dust in the presence of an ignition source directly in front of the hose operator. A gentle low pressure spray may be effective.

Often powder will block the exit from large vessels, such as spray dryers. The weight of accumulated water from the deluge systems may exceed the vessel's structural strength if the water cannot drain out. It is common practice to limit the time the deluge system can operate without being manually reset. Overflow valves are sometimes fitted to the dryer chambers to limit the amount of water they can hold.

Many fire fighting foams do not penetrate the dust, and can leave the smouldering powder under the foam layer. Foam can be used as a dust suppressant to allow water to be used safely to extinguish the fire. Alternatively, inert gases are effective, provided that they do not stir up the dust.

It is important to realise that all the normal hazards such as hot steam pipes, strongly corrosive chemicals, floors slippery with caustic soda and tripping hazards will still be present during a fire. Excessive preoccupation with the fire may place the staff or outside fire fighters in danger from these other hazards. Low visibility will compound the risk.

11.13 Conclusion

Milk powder spray dryers, fluid beds, cyclones, baghouses and ductwork contain an explosible dust-air mixture whenever the plant is running. The first line of defence is to prevent ignition. This involves eliminating external sources of ignition, and internal sources arising from self-heating of deposits within the plant. Fire detection systems such as temperature, infra-red and carbon monoxide detectors can often give warning of a fire in time to shut down the plant before a deflagration occurs. Once a deflagration is initiated, explosion suppression systems activated by pressure and/or infra-red sensors can be used to limit the extent of the pressure rise. Explosion venting is used as a last line of defence. This has the advantage of being a passive system with a very high reliability.

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Note: Page numbers in *italics* refer to tables or figures.