# 19 Packaging of Dairy Products

# 19.1 INTRODUCTION

Particularly in the Western world, milk from cattle (*Bos taurus*) accounts for nearly all the milk produced for human consumption. The composition of milk reflects the fact that it is the sole source of food for the very young mammal. Hence, it is composed of a complex mixture of lipids, proteins, carbohydrates, vitamins and minerals. The approximate composition of milk and the range of average compositions for milks of lowland breeds of cattle are given in Table 19.1. The water phase carries some of the constituents in suspension, while others are in solution. The fat is suspended in very small droplets as an oil in water emulsion and rises slowly to the surface on standing, a process often termed *creaming*.

Milk is processed into a variety of products, all having different and varying packaging requirements. The simplest product is pasteurized milk where, after a mild heat treatment, the milk is filled into a variety of packaging media and distributed. The shelf life of such a product varies from 2 to 15 days, depending largely on storage temperature and type of packaging. Ultra-high temperature (UHT) milk is subjected to a more complex process and the packaging is also more sophisticated; its shelf life can be up to 9 months. Cream is typically processed and packaged in a similar way to fluid milk and has a similar shelf life. Fermented dairy products, although being subjected to more complex processing operations, are also typically packaged in an analogous manner to fluid milk.

The other dairy products (butter, cheese and powders) are quite different in nature to fluid milk and their packaging requirements are, therefore, also quite different. Each of these groups of dairy products will be discussed in turn.

#### 19.2 FLUID MILK

# 19.2.1 PASTEURIZED MILK

Milk for liquid consumption is often standardized with respect to fat content and homogenized to retard the natural tendency for the fat globules to coalesce and rise to the surface. In response to consumer needs, a range of nonstandard fluid milk products has been developed in recent years. These products have varying (reduced) fat levels and additives such as calcium or other nutrients (e.g., the fat-soluble vitamins). From a packaging point of view, these nonstandard milks can be treated analogously to standard milks.

#### 19.2.1.1 Effect of Microorganisms

In virtually all countries, liquid milk for consumption must be pasteurized and cooled before it is packed. The primary purpose of the pasteurization is to destroy any pathogenic microorganisms present in the raw milk to make it safe for human consumption, while simultaneously prolonging the shelf life of the milk by destroying other microorganisms and enzymes that might ruin the flavor.

In general, only 90%–99% of all microorganisms present in raw milk are destroyed by pasteurization and pasteurized milk producers depend on refrigerated storage to achieve the required shelf life. Postheat treatment contamination (PHTC) with cold-tolerant Gram-negative spoilage bacteria is the limiting factor affecting the shelf life of commercial pasteurized milks at refrigerated storage temperatures. Although these bacteria are completely inactivated by pasteurization, they are

TABLE 19.1
Approximate Composition of Bovine Milk from Lowland Breeds

Average Content							
Component	(% w/w)	Range (% w/w)					
Water	87.1	85.3-88.7					
Solids-not-fat	8.9	7.9-10.0					
Fat in dry matter	31	22-38					
Fat	4.0	2.5-5.5					
Protein	3.25	2.3-4.4					
Casein	2.6	1.7-3.5					
Lactose	5.0	4.9-5.0					
Mineral substances	0.7	0.57-0.83					
Organic acids	0.17	0.12-0.21					
Miscellaneous	0.15						

Source: Walstra, P. et al., Dairy Technology: Principles of Milk Properties and Processes, Marcel Dekker, New York, 1999.

regularly found in pasteurized products. Inefficient sanitation of milk contact surfaces and contamination from the dairy plant atmosphere are the major causes of PHTC, with most problems arising in the filling line where open containers permit ingress of contaminants (Muir and Banks, 2003). Upgrading of pasteurized milk handling and packaging systems to ultraclean standard is an effective method for extending shelf life at refrigeration temperatures. It is also essential to control the number of stoppages on high-speed lines.

In the absence of PHTC, the shelf life of pasteurized milk products depends on the activity of heat-resistant organisms, which survive pasteurization, and their level of activity depends on the storage temperature. A shelf life of at least 8–10 days at 6°C–8°C is typical, while at 5°C, a shelf life of 18–20 days is realistic for pasteurized milk products processed using an ultraclean packaging system.

# 19.2.1.2 Effect of Temperature

#### 19.2.1.2.1 Thermization

Thermization is a heat treatment of lower intensity than low-temperature-long-time (LTLT) pasteurization (see following sections), typically 62°C–65°C for 10–20 s. The purpose is to delay spoilage of milk during storage in the factory before pasteurization, usually for manufacture into cheese or other products. Thermization is generally insufficient to destroy bacterial pathogens in raw milk with a reasonable margin of safety (Touch and Deeth, 2009) but it will destroy bacteria, especially psychrotrophs, because several of these produce heat-resistant lipases and proteases that may later cause deterioration of milk products (Walstra et al., 1999).

# 19.2.1.2.2 Pasteurization

All fluid milk, except for a small quantity of "certified" raw milk, is pasteurized at either 63°C for 30 min (referred to as the LTLT method or sometimes the Holder process) or at 72°C for 15 s (referred to as the high-temperature-short-time [HTST] method). The milk is then immediately cooled to 4°C or less. These heat treatments are designed to destroy microorganisms that produce disease and to reduce the number of spoilage microorganisms present. They do not sterilize the product.

Ultrapasteurization involves heating the milk at or above 138°C for at least 2s to destroy all pathogenic organisms. Ultrapasteurized products are also known as extended shelf life (ESL) or superpasteurized. There is no single definition for ESL milk but Rysstad and Kolstad (2006) defined ESL products as "products that have been treated in a manner to reduce the microbial count beyond normal pasteurization, packaged under extreme hygienic conditions, and which have a defined prolonged shelf life under refrigeration conditions." If packaged using aseptic filling machines into presterilized containers and kept refrigerated, ESL milk has a shelf life of up to 90 days with little effect on its nutritive value (Henyon, 1999). ESL milk generally has superior sensory properties compared with UHT milk as a result of a milder heat treatment and chill storage. However, limited data are available in the scientific literature on the safety, sensory qualities and shelf life of ESL milk (Rankin et al., 2011).

The disease-producing organisms of chief concern in milk are *Mycobacterium tuberculosis*, a non-spore-forming bacterium that causes tuberculosis and is frequently found in the milk of infected animals; Brucella species that cause brucellosis in animals and humans; and *Coxiella burnetii* that causes a febrile disease in humans known as Q fever. The most resistant of these three organisms is *C. burnetii*, which is characterized by a  $D_{65.6}$  of 30–36s and a z of 4°C–5°C. *M. tuberculosis* is characterized by a  $D_{65.6}$  of 12–15s and a z of 4°C–5°C, with the Brucella species characterized by a  $D_{65.6}$  of 6–12s and a z of 4°C–5°C. It is left as an exercise for the interested reader to calculate the number of decimal reductions achieved for each of the above-mentioned microorganisms by the LTLT and HTST pasteurization processes.

## 19.2.1.2.3 Shelf Life

One of the most critical factors affecting the shelf life of pasteurized dairy products is the temperature of storage. Attention has been focused on ways of predicting the effect of temperature on the growth of bacteria in foods. The shelf life of pasteurized milk is determined mainly by the level of contamination with Gram-negative psychrotrophic bacteria, although the microflora of pasteurized milk varies significantly with storage temperature. Thus, while spoilage at refrigeration temperatures is mainly due to the growth of *Pseudomonas* spp., Enterobacteriaceae and Gram-positive bacteria assume greater importance in the spoilage of milks stored at temperatures above  $10^{\circ}$ C. Recently, in order to increase the shelf life of pasteurized milk, processes such as bactofugation and microfiltration have been introduced in the dairy industry and are used to complement HTST pasteurization. Bactofuged pasteurized milk has a shelf life in excess of 10 days under refrigeration packaged in either PET bottles with a UV blocker or LDPE-coated paperboard cartons stored at  $4^{\circ}$ C  $\pm$   $2^{\circ}$ C (Kontominas, 2010).

In a study seeking to determine the maximum shelf life of fat-free pasteurized milk, Duyvesteyn et al. (2001) found no correlation between the microbial count at the end of shelf life and the sensory quality of the milk. The sensory shelf life of the milk stored in paperboard cartons at 2°C, 5°C, 7°C, 12°C and 14°C was 15.8, 13.7, 12.3, 4.6 and 3.9 days, respectively.

Ultrapasteurized ESL milk keeps longer (up to 90 days under refrigerated [<4°C] conditions but typically 45–60 days) but poses greater challenges than ordinary pasteurized milk (Rysstad and Kolstad, 2006). In pasteurized milk, spoilage organisms limit the shelf life and, as a result, pasteurized milk spoils before becoming unsafe. With ESL milk, the thermal process of ultrapasteurization destroys spoilage organisms along with pathogens. If the milk is contaminated after ultrapasteurization, then the product could potentially develop high levels of pathogens without the usual signs of spoilage.

## 19.2.1.3 Effect of Light

During processing, distribution, storage and marketing, milk may be exposed to natural and artificial light. Flavor changes as well as loss of vitamins and other nutritional components are attributed to chemical reactions induced in milk by light, particularly in wavelengths ranging from 420 to 550 nm (Skibsted, 2000). Taste panels have indicated that in the early stages of oxidation, milk loses its naturally fresh flavor and becomes quite flat in taste without being objectionable as in the more advanced stages of oxidation.

Riboflavin (vitamin  $B_2$ ) plays a central role as it is not only destroyed itself by light but, in addition, catalyzes the development of oxidized flavor and ascorbic acid oxidation by generating excited state (singlet) oxygen. Because of its absorption by riboflavin, light of wavelengths of 350–550 nm is the most damaging, the maximum damage occurring at about 450 nm. These wavelengths are contained in the emission spectra of white fluorescent tubes (Bossett et al., 1999). Although riboflavin has been assumed to be a prominent photosensitizer in dairy products, recent results indicate that porphyrins and chlorophyllic molecules might play an even more important role in the photochemical reactions in dairy products (Wold et al., 2009).

Riboflavin destruction by light is greater in low/nonfat milk than whole milk, because light of 400–500 nm wavelengths can penetrate 40–50% deeper into low/nonfat milk than whole milk (Allen and Parks, 1979). Destruction of nutrients by light could present legal problems in connection with nutrient standards and/or labels, because after storage in supermarket cabinets, the milk may not meet regulatory requirements. U.S. federal regulations require low fat and nonfat milk to be fortified with vitamin A, which may degrade from light exposure in a retail dairy case.

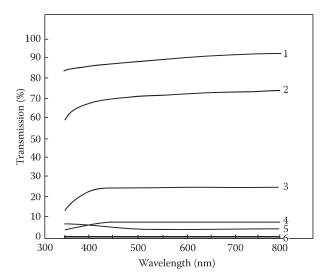
A study was conducted on the relative destruction of vitamin A and riboflavin in low-fat milk when exposed to fluorescent light at an intensity of 2000 lm m<sup>-2</sup> for 24 h (Senyk and Shipe, 1981). It was found that more than 75% of the added vitamin A was destroyed in glass, clear polycarbonate and HDPE containers. Paperboard containers provided the most protection, while gold-tinted polycarbonate, which blocks light of 400–480 nm, provided the second best protection. The presence of milk fat appears to protect against vitamin A degradation in fluid milk products, but adversely affects the flavor quality of milk after exposure to light. Even a brief, moderate light exposure (2 h at 2000 lx) can reduce the nutritional value and flavor quality of fluid milk products (Whited et al., 2002).

Pasteurized milk in LDPE bags without a light barrier suffered vitamin C losses in excess of 50% after 12h exposure to cool white light, whereas no loss was observed in milk packaged in paper-board cartons (Bossett et al., 1999).

The extent of nutrient loss in a supermarket depends on the proximity of the containers to the light source, the number and wattage of light bulbs in the display case, the exposed surface area of the package and the length of exposure. The detrimental effects of fluorescent light on milk can be alleviated by the following: (1) selecting packaging materials to minimize transmission of light, (2) reducing light intensities in display cases to 500 lm m<sup>-2</sup>, (3) use of yellow or yellow-green lamps or filters in display cases and (4) rotating packages at retail outlets to limit prolonged detrimental exposure to light (Senyk and Shipe, 1981).

Intawiwat et al. (2010) evaluated the effect of eight different colored filters based on PET on the photooxidation and quality of pasteurized milk (3.9% fat). Samples were stored in different atmospheres (air and  $N_2$ ) and exposed to light for 20 h at 4°C. The level of photooxidation was used to explain the results by studying degradation of the photosensitizers in the milk. For samples with high  $O_2$  concentration (packed with air in headspace), the red and green filters induced the least adverse effects, probably because of lower light transmission in these filters compared with the others. For samples with low  $O_2$  concentration (packed with  $N_2$ ) an unexpected, opposite trend was observed: red and orange filters induced the highest oxidation levels. The authors could not explain this phenomenon: why, with regard to photooxidation in milk, there apparently is an interaction between color of the light and the  $O_2$  concentration in the samples. A clear conclusion of the study was that complete blocking of light in the UV and the entire visible region is needed to avoid photooxidation and maximize shelf life for pasteurized milk.

Because it is clear that exposure of milk to light in the 400–550 nm wavelength region can result in the development of off-flavors and destruction of nutrients, packaging materials used for milk should ideally not transmit more than 8% of incident light at 500 nm wavelength and not more than 2% at 400 nm. Figure 19.1 shows the spectral transmission curves of six milk packaging materials and three of them (clear glass and clear and pigmented PET) exceed these guidelines by a



**FIGURE 19.1** Spectral transmission curves of milk packaging materials: (1) clear glass, (2) clear PET, (3) pigmented PET, (4) monolayer pigmented HDPE, (5) coated paperboard carton and (6) three-layer pigmented HDPE. (From Karatapanis, A.E. et al., *Int. Dairy J.*, 16, 750, 2006.)

considerable margin. Exposure to direct sunlight should be avoided under all circumstances as this also tends to increase the temperature of the milk, which accelerates microbial spoilage.

# 19.2.1.4 Effect of Gases

Oxygen plays an important role in the light-induced development of off-flavors in milk. Pasteurized milk at filling is generally saturated with  $O_2$  (about 8 ppm), but if no additional  $O_2$  can gain access, its content falls and the rate of adverse reactions slows or stops. However, additional  $O_2$  from the headspace or entering through a permeable container will maintain the  $O_2$  content and keep the rate of oxidative reactions high. The OTR at 4°C and 50% RH of a commercial 600 mL PET bottle is  $19\,\mu\text{L}$  day<sup>-1</sup> compared to  $390\text{--}460\,\mu\text{L}$  day<sup>-1</sup> for a commercial  $600\,\text{mL}$  HDPE bottle (van Aardt et al., 2001).

It has been known for many years that addition of  $CO_2$  to milk up to ~30 mM (mM is equivalent to mmol  $L^{-1}$ ) inhibits the growth of bacteria and extends the shelf life under refrigeration of both raw and pasteurized milk (Loss and Hotchkiss, 2003). Gram-negative bacteria are inhibited more than Gram-positives. The shelf life (defined as the time to reach  $10^6$  cfu m $L^{-1}$ ) of low fat milk packaged in high barrier plastic pouches and held at  $6.1^{\circ}$ C increased from 9.6 days with no added  $CO_2$  to 19.1 days when  $CO_2$  was present at 21.5 mM (Hotchkiss et al., 2006). Higher concentrations of  $CO_2$  enhance inhibition but also decrease the stability of the milk. In milk intended for consumption as a pasteurized fluid product, the  $CO_2$  must be reduced to a level similar to that found in untreated raw milk. Removal of  $CO_2$  can be achieved under vacuum, or the  $CO_2$  can be left to dissipate naturally. It is removed for sensory purposes, as it has a reported flavor threshold of  $10 \, \text{mM}$ , and prior to pasteurization to minimize fouling of heat exchanger surfaces.

 $\mathrm{CO_2}$  is being used for extending the shelf life of raw milk, particularly when it is necessary to transport milk in bulk over long distances. In such situations, extensions of shelf life of up to 14 days are possible if the milk is chilled to  $\sim\!2^{\circ}\mathrm{C}$  before being transported in insulated but nonrefrigerated containers.  $\mathrm{CO_2}$ -treated raw milk had lower microbial counts prior to pasteurization and exhibited a lower growth rate and a longer lag phase after pasteurization than non- $\mathrm{CO_2}$ -treated milk. It is also used, at  $\sim\!10\,\mathrm{mM}$ , for the shelf-life extension of fresh cheeses such as Cottage cheese and Quark (Deeth and Datta, 2011a). The quality of  $\mathrm{CO_2}$ -containing Cottage cheese packaged in PS tubs overwrapped with

a high barrier heat-shrinkable film can be maintained for 63 and 42 days at  $4^{\circ}$ C and  $7^{\circ}$ C, respectively. The use of  $CO_2$  in a range of dairy products has recently been reviewed (Singh et al., 2012).

# 19.2.1.5 Packaging Materials

Milk for retail sale was traditionally packaged in refillable glass bottles. However, today, single-serve paperboard cartons and plastic containers of various compositions and constructions dominate the market. The packaging material is central to the protection of the flavor and nutritional qualities of fluid market milk. The total amount of light passing through the container wall depends on the material from which the container is made, and also on the pigment either incorporated into the material or used in printing it. The nature of the pigment also determines the wavelength of the light reaching the milk.

Unpigmented HDPE milk bottles in the 350–800 nm spectral region have been found to transmit 58%–79% of the incident light. Light transmission was reduced by pigmenting with TiO<sub>2</sub> (1.6%), with the bottle being opaque below 390 nm. The use of a colorant with the TiO<sub>2</sub> pigment further reduced transmission of light with wavelengths less than 600 nm. The unprinted area of a paper-board carton had less than 1.5% transmission below 550 nm and was opaque to wavelengths below 430 nm (Nelson and Cathcart, 1984).

The effect of prolonged light exposure on the chemical changes in whole and 2% fat pasteurized milk stored at 4°C in clear PET bottles was compared with milk stored in green PET bottles, PET bottles incorporating a UV blocker, PET bottles with exterior labels, HDPE jugs and LDPE pouches (Cladman et al., 1998; van Aardt et al., 2001). The milk stored in the green PET bottles experienced less lipid oxidation and vitamin A loss than milk stored in clear PET bottles, HDPE jugs or LDPE pouches. The PET bottles with UV blockers slowed vitamin A degradation but had little effect on lipid oxidation. Blocking visible light with translucent labels helped to inhibit lipid oxidation and vitamin A degradation.

Vassila et al. (2002) studied changes in chemical and microbial quality parameters of whole pasteurized milk stored under fluorescent light at 4°C in 20 × 15 cm pouches made of (1) LDPE (clear and pigmented with  $TiO_2$ ) 60  $\mu$ m thick; (2) coextruded LDPE-PA-LDPE (clear and pigmented with  $TiO_2$ ) 60  $\mu$ m thick; and (3) coextruded LDPE + 2%  $TiO_2$ -LDPE + 2%  $TiO_2$ -LDPE + 4% carbon black-LDPE + 2% $TiO_2$ -LDPE + 2%  $TiO_2$ ) 60 and 110  $\mu$ m thick, with varying  $O_2$  (see Table 19.2) and light transmittance for a period of 7 days. Results showed good protection of milk packaged

TABLE 19.2

Oxygen Transmission Rate at 22°C and 60% Relative Humidity of Pouches and Carton for Milk Packaging<sup>a</sup>

Packaging Material	O <sub>2</sub> Transmission Rate (mL Package <sup>-1</sup> day <sup>-1</sup> )		
Clear LDPE pouch, 60 µm	38.3		
Pigmented (2% TiO <sub>2</sub> ) LDPE, 60 μm	38.8		
Clear LDPE-PA-LDPE, 60 µm	0.9		
Pigmented (2% TiO <sub>2</sub> ) LDPE-PA-LDPE, 60 μm	0.9		
LDPE + 2% TiO <sub>2</sub> -LDPE + 2% TiO <sub>2</sub> -LDPE + 4% carbon	39.5		
black-LDPE + $2\%$ TiO <sub>2</sub> -LDPE + $2\%$ TiO <sub>2</sub> , $60 \mu m$			
LDPE + 2% TiO <sub>2</sub> -LDPE + 2% TiO <sub>2</sub> -LDPE + 4% carbon	22.2		
black-LDPE + $2\%$ TiO <sub>2</sub> -LDPE + $2\%$ TiO <sub>2</sub> , $110 \mu m$			
LDPE-paperboard-LDPE carton, 450 µm	7.2		

Source: Vassila, E. et al., Int. Dairy J., 12, 715, 2002.

<sup>&</sup>lt;sup>a</sup> Pouch dimensions were 20 × 50 cm; all packages had a capacity of 500 mL.

in all pouches with regard to microbial and chemical parameters with the exception of vitamin retention. In both clear and TiO<sub>2</sub>-pigmented LDPE and LDPE-PA-LDPE pouches, vitamin degradation ranged from 50.9% to 73.6% for vitamin A and from 34.4% to 45.3% for riboflavin. In the coextruded pouches containing an inner layer of carbon black, the respective losses were 15.1% and 18.9%. No sensory evaluation was included in the study.

The shelf life of whole and low fat (1.5%) pasteurized milk stored at 4°C for 7 days in a variety of packages has been reported (Moyssiadi et al., 2004; Zygoura et al., 2004). The five packages evaluated were as follows: (1) 0.5 L multilayer pigmented (HDPE + 2% TiO<sub>2</sub>-HDPE + 4% carbon black-HDPE + 2% TiO<sub>2</sub>) 550–600 µm thick; (2) 0.5 L monolayer pigmented (HDPE + 2% TiO<sub>2</sub>) 550–600 µm thick; (3) 0.5 L clear PET 300–350 µm thick; (4) pigmented (PET + 2% TiO<sub>2</sub>) 300–350 µm thick; and (5) 0.5 L LDPE-paperboard-LDPE cartons 20–395–35 µm thick, which served as the control. Chemical and microbiological parameters showed satisfactory protection of whole milk in all five packages. For the whole milk, vitamin A losses were 8.8%, 10.5%, 50.9%, 29.8% and 14.0% and riboflavin losses were 18.4%, 20.6%, 47.1%, 30.9% and 19.8%. For the low-fat milk, vitamin A losses were 11%, 11%, 31%, 11% and 16% and riboflavin losses were 28%, 30%, 40%, 33% and 28%. The best overall protection was provided by the multilayer HDPE followed by the monolayer TiO<sub>2</sub>-pigmented HDPE bottles.

Karatapanis et al. (2006) investigated changes over 7 days in the volatile profiles of whole pasteurized milk stored under fluorescent light at 4°C and packaged in different containers in a study designed to differentiate between light-induced oxidative and purely autoxidative effects related to packaging material. Packaging materials tested were the same as those detailed in the preceding paragraph. Two distinct patterns of milk flavor deterioration were observed. In light-exposed samples, a light-induced oxidation mechanism prevailed, whereas in light-protected samples, an autoxidation mechanism was apparent. Microbiological data correlated poorly with both sensory and GC/MS data. Sensory data correlated well with selected volatile compounds, pointing to dimethyl disulfide, pentanal, hexanal and heptanal as potential markers of fresh milk quality. Based on sensory analysis, the optimal shelf life of the whole pasteurized milk used in this study was ~5 days.

Mariani et al. (2006) monitored sensory changes in pasteurized milk stored under fluorescent light in a supermarket refrigerator case for a period of 9 days in different packages (1) clear PET bottles, (2) cobalt blue PET bottles and (3) multilayer pigmented gable-top paperboard cartons. Milk packaged in both clear and cobalt blue PET bottles was affected by off-flavor between the first and second day of storage. Milk packaged in paperboard cartons did not develop any off-flavor during the entire storage period.

Clear PET bottles completely transmit light at wavelengths above 320 nm and thus offer no protection against light in the critical range for vitamin degradation. For this reason, pigmented PET bottles are required to protect pasteurized milk against vitamin A and vitamin  $B_2$  degradation. Saffert et al. (2006) stored pasteurized whole milk (3% fat) for 10 days under fluorescent light at 8°C in clear 1L PET bottles and three variants of pigmented PET bottles with different light transmittance. In clear PET bottles, vitamin A was reduced by 22% and vitamin  $B_2$  by 33%, while vitamin  $B_1$  remained almost constant. In all pigmented PET bottles, vitamin retention was significantly higher, with losses of 0%–6% for vitamin A and 11%–20% for vitamin  $B_2$ , depending on the pigmentation level. No impact of package light transmittance on the vitamin  $B_{12}$  content was observed. The use of highly pigmented PET bottles to reduce the light transmittance to below 10% at a wavelength of 450 nm appeared to protect vitamin  $B_2$  sufficiently within the normal shelf life period for pasteurized milk. However, light-induced sensory changes in pasteurized milk cannot be excluded under commercial storage conditions in packages with light transmission rates of 10% or even less at a wavelength of 450 nm.

The influence of storage temperatures from  $2^{\circ}\text{C}$  to  $16^{\circ}\text{C}$  on the microbial stability of homogenized whole pasteurized milk (75°C/15 s) packed in 1 L HDPE bottles and 1 L LDPE pouches pigmented with  $\text{TiO}_2$  was evaluated by Petrus et al. (2009). The microbiological shelf life for milk in HDPE bottles stored at  $2^{\circ}\text{C}$ ,  $4^{\circ}\text{C}$ ,  $9^{\circ}\text{C}$ ,  $14^{\circ}\text{C}$  and  $16^{\circ}\text{C}$  was 43, 36, 8, 5 and 3 days, respectively, and

for milk in LDPE pouches, 37, 35, 7, 3 and 2 days, respectively. Although no data was presented, it can be assumed that the HDPE bottle had a lower OTR than the LDPE pouch, which would explain the longer shelf life in the former.

## 19.2.2 UHT MILK

The International Dairy Federation has suggested that UHT milk should be defined as "milk which has been subjected to a continuous-flow heating process at a high temperature for a short time and which afterward has been aseptically packaged. The heat treatment is to be at least 135°C for one or more seconds."

The development of UHT milk processing methods for sterilizing in a continuous flow has brought about the need for aseptic packaging of the product. It is only through the use of aseptic packaging that the benefits of UHT processing can be fully realized.

The quality of the raw milk used in UHT processing is of utmost importance. It is arguably more important than for pasteurized products because of the long periods of storage of UHT products at ambient temperature when even very slow development of defects may lead to end of shelf life. In practice, some manufacturers select milk of the highest quality to use in UHT processes in order to minimize processing difficulties and the incidence of storage-related defects (Datta and Deeth, 2007).

# 19.2.2.1 Process Description

## 19.2.2.1.1 Sterilization

UHT processes can be classified as either directly or indirectly heated according to the kind of heat exchangers used (see Deeth and Datta [2011b] for details). Most European regulations rely on spoilage data as a measure of how well an aseptic system works. The U.S. FDA, however, requires microbiological (challenge) and chemical tests to document whether an aseptic system provides an adequate margin of safety.

# 19.2.2.1.2 Packaging

The various aseptic packaging systems available commercially have been described in Chapter 13 and Robertson (2011). Although all the different systems can be used for UHT milk, the most widely used are the paperboard/alufoil/plastic laminate carton and the plastic container.

# 19.2.2.2 Microbiology

Destruction of microorganisms during UHT processing has been well documented. The spores capable of surviving the UHT process are mainly *Geobacillus stearothermophilus*, *Bacillus subtilis*, *Bacillus megaterium*, *Bacillus sporothermodurans* and *Paenibacillus lactis*. *G. stearothermophilus* has a high survival potential, but is unable to grow below ~30°C and is a major problem only in tropical climates. In temperate climates, *Bacillus coagulans*, *B. subtilis* and *Bacillus licheniformis* are the most important spoilage species, although some heat-resistant strains of *Bacillus cereus* have also been implicated (Touch and Deeth, 2009).

The quality of raw milk selected for manufacture of UHT milk is critical. UHT milk prepared from raw milk containing more than  $5 \times 10^6$  cfu mL<sup>-1</sup> psychrotrophs is at risk of spoilage due to heat-resistant enzymes. For UHT milk to have a shelf life of 1 year, the raw milk must contain less than 0.1 unit of protease mL<sup>-1</sup> and some high protease-producing bacteria can easily synthesize this amount within a day (Touch and Deeth, 2009).

Massive contaminations of entire commercial lots of UHT and sterilized milk with a then unknown mesophilic aerobic spore former (subsequently identified as *B. sporothermodurans*) were first reported in Italy and Austria in 1985 and in 1990 in Germany. This organism was provisionally called a *highly heat-resistant spore former* (termed HHRS or HRS), as the causative organism survived the UHT process; it occurred more frequently in indirect UHT than in direct UHT processing.

The problem subsequently spread to other countries in and outside Europe. Affected milk products included whole, skimmed, evaporated and reconstituted UHT milk, UHT cream and chocolate milk in different kinds of containers and also milk powders (Scheldman et al., 2006).

*B. sporothermodurans* does not appear to cause spoilage other than a slight discoloration of the milk and may reach a maximum of 10<sup>5</sup> vegetative cells and 10<sup>3</sup> spores mL<sup>-1</sup> milk after 15 days' incubation at 30°C of unopened packages of consumer milk. These levels do not affect the pH of the milk and usually do not alter its stability or sensory quality. However, this contamination level far exceeds the sterility criterion of 10cfu (0.1 mL)<sup>-1</sup> specified in EU regulations. Several HRS strains have been tested and none showed pathogenic potential. Despite its poor growth characteristics in milk, UHT milk can be regarded as a new ecological niche for *B. sporothermodurans* because of the lack of competition from other organisms in this product (Scheldman et al., 2006).

The current hypothesis is that highly heat-resistant spores are adapted by sublethal stress conditions (e.g., the  $H_2O_2$  used to sterilize packaging material) in the industrial process and selected for by the heating step. As a result, considerable problems may occur through recirculation (reprocessing) of UHT milk that has passed its use-by date, leading to contaminated lots of milk and milk products. It is extremely difficult to remove from contaminated equipment and has caused the closure of some UHT plants. Reprocessing of UHT milk should not be permitted.

## 19.2.2.3 **Nutrition**

The nutritive value of UHT milk can be reduced at two stages: during the UHT treatment and during storage after packaging. The nutritive values of milk components such as fat, fat-soluble vitamins, carbohydrates and minerals are essentially unaffected, whereas values of other components such as the water-soluble vitamins and proteins are adversely affected. Nutrient loss during storage is a function of the temperature of storage, the initial  $O_2$  content of the milk and the nature of the packaging material (in particular, its opacity and permeability to  $O_2$ ).

#### 19.2.2.3.1 Vitamins

In general, it appears that vitamins are more stable under UHT processing conditions than under pasteurization or other low temperature heat treatments. Fat-soluble vitamins (A, D and E) as well as some water-soluble vitamins (riboflavin, nicotinic acid and biotin) are heat stable and are not adversely affected by UHT processing. Vitamins such as folic acid, C and  $B_{12}$  are lost to different extents (Rosenberg, 2011). Considerable variation (from 0% to 100%) has been observed in the losses of water-soluble vitamins during UHT processing and subsequent storage. If the amount of  $O_2$  dissolved in milk is limited, then ascorbic acid losses are minimal. The nutritional value of UHT milk can deteriorate during storage to an extent that is highly dependent on the  $O_2$  level in the product, the temperature and exposure to light. A high nutritional value of UHT milk can be attained by achieving low  $O_2$  levels, selecting packaging materials with effective  $O_2$  barriers and preventing exposure to light by using opaque containers. Low  $O_2$  levels can be achieved either by the use of a deaerator before heating or evaporative cooling after processing, the latter being an essential part of the direct heating process.

# 19.2.2.3.2 Proteins

The milk constituents that undergo the greatest change during UHT processing and storage are the proteins. Alterations in proteins are related to many technological problems with UHT products such as flavor, gelation, sediment formation, fouling of heat transfer surfaces, loss of nutritional value and browning.

Severe heat treatment causes considerable denaturation (up to 80%) of the serum proteins of milk, especially  $\beta$ -lactoglobulin. Direct heated UHT milk has less serum protein denaturation than indirect heated milk. Although available lysine levels in milk are reduced due to the Maillard reaction, the decrease is small and does not represent a significant loss in nutritional value. No significant changes in other amino acids occur, either during processing or storage.

#### 19.2.2.4 Biochemical and Physical Aspects

Extensive research has reported the presence and characteristics of heat-resistant enzymes in milk and their effects on UHT products during storage (Burton, 1988). Proteases and lipases are of greatest concern. Although phosphatase activity is always zero after milk has been sterilized, it may be reactivated after prolonged storage, where the extent of reactivation increases with storage time and temperature.

The shelf life of UHT milk is sometimes limited by age gelation, an irreversible phenomenon, which is characterized by an increase in viscosity during storage and ultimately formation of a gel, similar to that of custard. It is considered the most important quality problem associated with this type of product, because once the milk has gelled, it has reached the end of its shelf life. The gel is a three-dimensional network of whey proteins and caseins that binds water and engulfs casein micelles and fat globules. The skeleton of the matrix is a protein complex formed between heat-denatured  $\beta$ -lactoglobulin and  $\kappa$ -casein (Datta and Deeth, 2007).

Many factors affect the rate at which gelation occurs, but the major ones are proteolysis, severity of heat treatment, storage temperature, bacteriological quality of the milk and the milk solids content. A widely held view is that gelation is caused by proteolysis of casein caused by the natural milk proteinase, plasmin and/or heat-stable proteinases produced by psychrotrophic bacterial contaminants in the raw milk before processing. The more severe the heat treatment, the longer age gelation is delayed. Thus milks sterilized by indirect heating methods are less susceptible to gelation than milks treated by the direct steam injection and infusion methods. The rate of gelation of UHT milk is markedly influenced by the temperature of storage. Storage at refrigeration temperatures (~4°C) and "high" temperatures (35°C-40°C) delays gelation, while gelation occurs at a maximum rate at ~25°C-28°C. Milk of poor bacteriological quality at the time of processing is much more susceptible to gelling than good quality milk. UHT skim milks are more susceptible to gelation than whole milks. This is attributable to the reduced rate of proteolysis in whole milk caused by partial masking of the protein by fat, which prevents access to the caseins by the proteases (Datta and Deeth, 2007). Recently, a method for detecting very low levels of protease activity and predicting progress of proteolysis in stored UHT whole milk was developed (Button et al., 2011). Although the assays require up to 14 days to complete, this is not an excessive time, compared with the time required for microbiological clearance and total shelf life of the product.

The two main practical ways of preventing gelation or reducing its incidence are to use high quality raw milk and an indirect heating system for the high heat treatment. Addition of sodium hexametaphosphate (SHMP; 0.05% and 0.1%) to milk before heat treatment is effective in retarding gelation. SHMP does not inhibit proteolysis but prevents proteolyzed milks from gelling and is used where food regulations permit.

#### 19.2.2.5 Flavor

The flavor of UHT milk is different from pasteurized milk, with the former generally having a flatter or "purer" taste due to the removal of most of the feedy or barny odors. Fresh UHT milk is characterized by a poor flavor, described as a noticeable "heated" flavor, and by a sulfurous odor note. The initial sensory characteristics of UHT milk disappear within a few days of storage, and a characteristic UHT milk flavor develops with storage time (Rosenberg, 2011).

A U.S. committee on flavor nomenclature (Shipe et al., 1978) has hypothesized that there are four kinds of heat-induced flavors: cooked or sulfurous; heated or rich; caramelized; and scorched. Milk that has been heated to 135–150°C for several seconds exhibits a strong sulfurous or cooked flavor. After several days of storage, this flavor disappears to leave a rich or heated note. Volatile sulfides are believed to contribute to the cooked flavor and it has been suggested that the Maillard nonenzymic browning reaction causes the caramelized flavors. The compounds responsible for the rich or heated note have not been clearly elucidated, and it is possible that what many researchers describe as "stale" is a combination of "rich or heated" and "caramelized" (Mehta, 1980).

Oxidized or rancid off-flavors can also develop in UHT milk, with the extent depending on the level of  $O_2$  and the storage temperature. There is a direct relationship between the level of dissolved  $O_2$  in a product and the headspace volume of a sealed container.

# 19.2.2.6 Packaging Materials

The most common packaging material used for UHT milk is the paperboard laminate carton, although increasing quantities of plastic-based packages are now being used. The various types of packaging systems suitable for UHT milk were described in Chapter 13 and will not be repeated here.

The sorption of dairy flavor compounds (aldehydes and methyl ketones) by LDPE and PP films has been investigated quantitatively in an attempt to assist aseptic processors select appropriate packaging materials for maximum flavor stability. PP sorbed these compounds to a greater extent than LDPE. Headspace analysis of UHT-processed milk packaged in aseptic paperboard cartons revealed a loss of higher MW flavor compounds after 12 weeks storage, owing to interaction between the LDPE packaging material and the milk (Hansen and Arora, 1990).

Saffert et al. (2008) evaluated changes in the vitamin content of UHT whole milk stored for 12 weeks at  $23^{\circ}$ C and three different light intensities in PET bottles with a range of light transmittance. Losses of vitamins A and  $B_2$  were more pronounced in transparent PET bottles exposed to the highest light intensity. In these bottles, a reduction in light intensity reduced vitamin A loss from 88% to 66%, whereas in the case of vitamin  $B_2$  its complete decomposition was delayed from 4 to 8 weeks. Vitamin  $D_3$  losses in clear PET bottles were almost independent of light intensity. For the pigmented PET bottles, an increase in package light transmittance and light intensity was found to critically affect vitamin  $B_2$  stability. For vitamin  $D_3$  only, the increase in light intensity was found to be of relevance, whereas for vitamin A stability, no clear effect of light transmittance and light intensity was observed. No sensory evaluation was carried out in the study.

In a similar study, Saffert et al. (2009) stored UHT low fat (1.5%) milk for 12 weeks at  $23^{\circ}$ C under light of 700 lx in four PET bottle variants representative of those used for milk on the European market in terms of their light transmittance. In clear PET bottles, vitamin A declined by 93% and vitamin  $D_3$  by 66%, whereas vitamin  $B_2$  was completely degraded. In all pigmented PET bottles, the vitamin retention was only slightly higher, with the losses ranging between 70% and 90% for vitamin A, 63% and 95% for vitamin  $B_2$  and 35% and 65% for vitamin  $D_3$  depending on the pigmentation level. In the dark-stored "control" sample, a 16% loss was observed for vitamin A, but the levels of vitamins  $B_2$  and  $D_3$  remained almost stable. On the basis of these findings, the authors concluded that light barrier properties comparable with the high-pigmented PET bottle seemed to be sufficient to protect the light-sensitive vitamins A,  $B_2$  and  $D_3$  in milk for realistic periods on the retailer's shelf. However, they considered that light-induced sensory changes in UHT milk under commercially relevant storage conditions can only be excluded in light-tight packages.

Despite their widespread use, there are virtually no peer-reviewed papers on the shelf life of UHT milk in aseptic cartons. Recently, de Longhi et al. (2012) analyzed five brands of UHT milk sold in Brazil for up to 120 days, which is considered the end of shelf life in that country. None complied with all the physicochemical standards and regulations in Brazil although two brands met all applicable microbiological requirements. Gelation was noticed in all brands after 90 days.

# 19.3 FERMENTED PRODUCTS

Fermented milks are products prepared from milks (whole, partially or fully skimmed milk, concentrated milk or milk reconstituted from partially or fully skimmed dried milk) homogenized or not, pasteurized or sterilized and fermented by means of specific microorganisms.

Yogurt, the most important of the fermented milk products, is a coagulated milk product obtained by lactic acid fermentation through the action of typically *Lactobacillus bulgaricus* and *Streptococcus thermophilus*. It may contain added fruit or fruit flavors as well as carbohydrate

sweetening matter. Kefir has an alcohol content of 0.5%–2% and koumiss of 2%–3%, and both contain considerable quantities of  $CO_2$ , which is formed by the heterofermentative, aroma-forming lactic acid bacteria.

The shelf life of yogurt products is determined by the time the product remains safe to eat, the time its functional claims remain true to label or to regulatory requirements and the time its sensory properties remain acceptable to consumers. Fresh yogurt is at its best in the first few weeks of shelf life, after which there is a discernible reduction in sensory characteristics. For example, Salvador and Fiszman (2004) compared storage at 20°C for 21 days with storage at 30°C for 3 days (accelerated) during a study of refrigerated storage (10°C for 91 days) on whole milk and skim milk flavored, set yogurts. They found a gradual deterioration in sensory properties over a 91-day period such that the probability of consumer acceptance was around 40% for the whole milk yogurt and only 15% for the skim milk yogurt after 91 days' storage at 10°C.

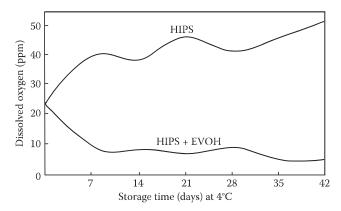
A wide range of packaging materials is used for yogurt products (MacBean, 2010). The most popular material by far in current use for spoonable yogurt (either set or stirred) is thermoformed HIPS in the form of small cups or larger tubs, with either an aluminum foil/plastic laminate or a paper/plastic laminate heat seal lid or closure. For example, 6–8 gsm of EVA is applied to foil intended for heat sealing to PS or PP (Özer, 2010). These containers may be produced in form-fill-seal packaging machines or be delivered preformed from packaging material suppliers. It is normal to add pigments such as TiO<sub>2</sub> to the HIPS in order to improve the appearance of the package and to provide some barrier to light (Table 19.3). This also helps in heating and softening the HIPS sheet for thermoforming when radiant heating is used. Rectangular paperboard cartons or cups (with or without an aluminum foil layer), glass containers, PP and blow molded HDPE containers are also used, and for some specialty products in some markets, ceramic containers have been used. A study of the effect of light-induced changes in plain yogurt concluded that PLA provided better protection against photodegradation processes than PS (Frederiksen et al., 2003).

For pasteurized, spoonable yogurt products, laminated materials are desirable if a long shelf life is needed, with some having shelf lives of 4–6 months at ambient temperatures. For these products, a low WVTR is required to stop the yogurt losing water during shelf life. A good O<sub>2</sub> barrier will help to protect the product from oxidation, and a good light barrier will help to delay fading of light-sensitive colors and avoid light-induced oxidation (MacBean, 2010).

TABLE 19.3
Summary of Protective Effects of Different Packaging Materials for Yoghurt

	Protectio	n Against	Rank in Order	
Materials	Light	Oxygen	of Decreasing Total Protection	
Transparent brown glass	Good	Perfect	1	
Unpigmented glass	Moderate	Perfect	4	
Transparent brown PS	Good	Moderate	3	
Unpigmented PS	Bad	Moderate	5	
Paperboard and PS	Excellent	Bad	2	

Source: Bossett, J.O. et al., Influence of light transmittance of packaging materials on the shelf life of milk and dairy products—A review, in: Food Packaging and Preservation, Mathlouthi, M. (Ed.), Aspen Publishers, Gaithersburg, MD, pp. 222–268, 1999.



**FIGURE 19.2** Dissolved O<sub>2</sub> levels in yogurt stored for 42 days at 4°C in HIPS (upper curve) and HIPS with an EVOH barrier layer (lower curve). (From Miller, C.R. et al., *Packag. Technol. Sci.*, 16, 61, 2003.)

A recent study into the effect of some packaging materials on the flavor of strawberry-flavored, stirred yogurts of 0% and 4% fat content during shelf life showed interesting differences between glass packaging as a reference, PP and 50:50 PS-HIPS (Saint-Eve et al., 2008). In common with an earlier study by Salvador and Fiszman (2004), 0% fat yogurt was found to deteriorate faster than 4% fat yogurt regardless of the packaging type. It was also concluded that PS-HIPS seemed to be preferable to PP for avoiding the loss of fruity notes and for hindering the development of odor and aroma defects, particularly for 4% fat yogurts.

Today, it is common to add probiotic bacteria such as *Lactobacillus acidophilus* and *Bifidobacterium* spp. to yoghurt to impart health benefits to consumers. These bacteria require a low  $O_2$  environment for maximum viability, because dissolved  $O_2$  has a negative effect on their viability. A study by Miller et al. (2003) found that HIPS was ineffective as an  $O_2$  barrier because, during the normal shelf life of commercial yoghurt, the mean dissolved  $O_2$  content increased from 20 to 50 ppm. The use of a packaging material with an added  $O_2$  barrier layer (HIPS-tie-EVOH-tie-LDPE) had a lowering effect on the dissolved  $O_2$  content in the commercial yoghurt, with an initial  $O_2$  content of 20 ppm decreasing to 8 ppm over 42 days (Figure 19.2).

Photooxidation of sour cream packaged in PS cups with different light barrier properties was investigated by Larsen et al. (2009). Three types of cups were evaluated (1) standard PS cups with  ${\rm TiO_2}$ , (2) PS cups with  ${\rm TiO_2}$  and a barrier layer of  ${\rm Al_2O_3}$  and (3) PS cups with  ${\rm TiO_2}$  and a barrier layer of carbon black and  ${\rm Al_2O_3}$ . An area  $5\times7$  cm on one side of each cup was exposed to light at 56101x for 36h. Based on sensory evaluation, it was concluded that only cups incorporating a layer of carbon black protected the sour cream from degradation when exposed to light.

# 19.4 BUTTER AND SPREADS

#### 19.4.1 Composition

Butter is a fat product obtained from milk and has the following main characteristics: a minimum milk fat content of 80%–82%, total fat-free dry milk solids of 2% and a maximum moisture content of 16%. Approved additives include food colorants (annatto,  $\beta$ -carotene and curcumin), NaCl and cultures of harmless lactic acid-forming bacteria. The pH can be adjusted, if required, by the addition up to a maximum level of  $2 \text{ mg kg}^{-1}$  of approved neutralizing salts (Fearon, 2011).

During the production process, the milk fat is concentrated by two successive physical steps: separation of the milk and churning of the cream, which is subject to phase inversion by physical disruption of the natural milk fat globule membrane. After washing with clean water to remove milk solids, the granules are physically worked into a uniform mass that is called *butter*. Thus, butter is

basically a high fat-content product in which the fat phase is the continuous one. However, the water phase (consisting mainly of small droplets) can migrate slowly. Butter is normally classified into the following four groups:

- 1. Unsalted butter (i.e., butter not flavored with salt) prepared from sweet cream
- 2. Salted butter prepared from sweet cream (salt content in the range of 0.2%–2.0%)
- 3. Unsalted butter prepared from sour cream
- 4. Salted butter prepared from sour cream

The most common type of butter in the United States is salted butter prepared from sweet cream. It typically has a moisture content of 15.8% w/w and a fat content of 82% w/w.

The shelf life of butter is influenced by microbial, enzymic and chemical reactions. In addition, the susceptibility of butter to readily absorb odors from the surrounding environment can also limit its shelf life. Therefore, the choice of packaging material is usually made with the intention of delaying or preventing the undesirable reactions.

The chemical composition of butterfat plays an important role in oxidation. Heavy metals or their salts (particularly copper) have a very strong catalytic effect, with low pH promoting oxidation. Free fatty acids, fat-soluble amino acids and carotene also promote oxidation, although under the influence of light, carotene acts as an antioxidant. A natural antioxidant in butter is tocopherol (vitamin E). Butter is best stored at  $-25^{\circ}$ C, and sweet cream, salted butter keeps satisfactorily for several years at this temperature. Although slightly oxidized flavors are expected by many consumers and are disguised by salt addition, the shelf life of butter can be usefully prolonged by exclusion of  $O_2$  during packaging and subsequent storage (Muir and Banks, 2003).

The chemical composition of the water phase varies depending on the type of butter in respect of pH and salt, protein and lactose content. Consequently, different microfloras can develop in, or are inhibited by, the various butters. For example, salted butter made from sour cream inhibits the development of certain microorganisms.

Dairy-based spreads are manufactured using similar technology to that used to produce margarine and may have fat contents of 37.5%–76.3%. In contrast to butter, the amount of fat present is low but high levels of milk protein may be incorporated to stabilize the product. Because of the high water content, the water in oil emulsion may have limited stability and this limits shelf life, especially when the product is subject to temperature cycling. A further problem is the potential for bacterial growth and spoilage as a result of the higher water content, limiting the shelf life of spreads especially when stored above 4°C (Muir and Banks, 2003).

#### 19.4.2 PACKAGING REQUIREMENTS

## 19.4.2.1 **Oxidation**

Butter is very susceptible to light-induced flavors, which are mostly accompanied by oxidation defects. Light-induced oxidation of butter may occur when it is inadequately protected under illumination. The degree of deterioration depends on factors such as light source, wavelength of light, exposure time, distance of the butter from the light source and  $\beta$ -carotene content of the butter (Hansen and Skibsted, 2000). Butter is generally exposed to fluorescent light during storage in retail display cabinets.

The effect of varying headspace  $O_2$  concentration and color of light on photooxidation and degradation of photosensitizers in butter was reported by Wold et al. (2009). In butter, at least six different photosensitizers are present: riboflavin, protoporphyrin IX, hematoporphyrin, a chlorophyll a-like substance and two unidentified tetrapyrroles.

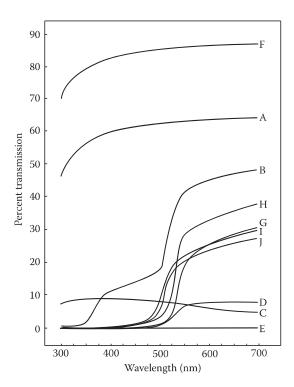
Butter samples were stored at 4°C under 0%, 0.4%, 0.8%, 1.6%, 3.0%, 5.0% and 21%  $O_2$  and exposed to violet, green or red light for 36h. The higher the  $O_2$  concentration, the more sensory

degradation of the samples. Violet light resulted in slightly higher degrees of photooxidation than green and red light for low O<sub>2</sub> concentrations.

In light of the aforementioned points, it is no surprise that the packaging material has a marked effect on the intensity of the oxidized flavor that develops, depending largely on the amount of light transmitted. Butter packaged in various types of conventional parchment papers develops objectionable levels of oxidized off-flavor after a few hours exposure in a supermarket display cabinet. However, aluminum foil laminates are satisfactory even after 48 days of continuous exposure. In a study (Emmons et al., 1986a) that investigated the influence of light on butter packaged with various materials (five mica-filled HDPE-based plastics, some containing yellow pigments; two parchment papers; two aluminum metallized papers; and aluminum foil glue laminated to bleached sulfite paper), oxidation occurred on the light-exposed butter surface with all the materials except the aluminum foil laminate when exposed to the three strongest light intensities.

Various packaging materials differ markedly in their transmission of light as shown in Figure 19.3. The transmission of parchment (A) ranges from 46% to 64%, while the presence of yellow pigments in (B) markedly reduces transmission to 1%–17% in the 300–500 nm range and to less than 50% in the higher range. However, the reduced transmission below 500 nm does not appreciably reduce oxidation, indicating that longer wavelengths are important. Metallized papers transmit less than 10% of light and the foil paper laminate transmits no measurable light. Oxidation is greater at lower intensities of light and at shorter times of exposure than expected from that observed at higher intensities and longer exposure times.

Metallic contamination of butter from dairy processing equipment is now minimal due to the widespread use of stainless steel. In light of the fact that surface oxidation has been documented as the major flavor defect in butter at the retail level (Emmons et al., 1986b), butter should be packaged in laminates containing aluminum foil, which effectively block out all the light.



**FIGURE 19.3** Spectra of light transmission by paper-based (A, B, C and D); foil laminate (E); and polyethylene-based packaging materials (F, G, H, I and J). (From Emmons, D.B. et al., *J. Dairy Sci.*, 69, 2248, 1986b.)

# 19.4.2.2 Water Vapor Permeability

The surface desiccation of butter results in discoloration, a defect known as *primrosing*. However, if the packaging material is completely impermeable to water vapor, then there might be an increased risk of surface mold growth in areas where pockets of moisture have developed. The surface drying, which occurs in butter packaged in parchment would thus be an advantage if the butter were susceptible to mold growth.

The WVTR requirements of a satisfactory packaging material for butter vary from country to country but a maximum of 3 g m<sup>-2</sup> day<sup>-1</sup> is generally agreed upon.

# 19.4.2.3 Odor Permeability

The susceptibility of fats to absorb odor compounds means that a satisfactory packaging material for butter should protect the butter from such odors. One simple but common test is to store the packaged butter for 24h at 18°C over clove oil, after which time the butter should have no odor or taste of clove oil.

# 19.4.2.4 Packaging in Current Use

Retail packaging of butter is commonly in aluminum foil (0.009 mm thick) laminated either to 40 gsm greaseproof paper or vegetable parchment, or sometimes just the paper or parchment alone. Other types of packaging for butter and dairy spreads include plastic tubs thermoformed from white-pigmented HIPS or PVC with a tight-fitting lid of the same material. To fill tubs with butter, it must be packed directly from the churn or reworked immediately prior to packing so that it will flow into the package and fill it efficiently.

#### **19.5 CHEESE**

## 19.5.1 CLASSIFICATION

Cheese is the generic name for a group of fermented milk-based foods produced in at least 500 varieties around the world. Although some soft cheese varieties are consumed fresh (i.e., without a ripening period), production of the vast majority of cheese varieties can be subdivided into two well-defined phases: manufacture and ripening. Despite differences in detail in the manufacturing processes used for individual varieties, the conversion of milk into cheese generally comprises four stages:

- 1. *Coagulation*: physicochemical changes in the casein micelles due to the action of proteolytic enzymes and/or lactic acid lead to the formation of a protein network called coagulum or gel
- 2. *Drainage*: separation of the whey, after mechanical rupture of the coagulum, by molding and, in certain cases, by pressure, to obtain a curd
- 3. *Salting*: incorporation of salt by deposition on the surface or within the body of the cheese, or by immersion in brine
- 4. *Ripening*: biochemical changes in the constituents of the curd brought about by the action of enzymes, mostly of microbial origin

Cheese manufacture can be viewed as essentially a dehydration process in which the fat and casein in milk are concentrated between 6- and 12-fold, depending on the variety. The degree of hydration is regulated by the extent and combination of the three steps listed earlier in addition to the chemical composition of the milk. In turn, the level of moisture in the cheese, the salt content and the cheese microflora regulate the biochemical changes that occur during ripening and thus determine the flavor, aroma and texture of the finished cheese. Although the nature and quality of the finished cheese are determined in very large measure by the manufacturing steps, it is during the ripening phase that the characteristic flavor and texture of the individual cheese varieties develop.

With the exception of some soft cheeses, most cheese varieties are not ready for consumption at the end of manufacture but undergo a period of ripening (also referred to as *curing* or *maturation*), which varies from about 4 weeks to more than 2 years. The duration of ripening is generally inversely related to the moisture content of the cheese, although many varieties may be consumed at any of several stages of maturity depending on the flavor preferences of consumers.

#### 19.5.2 MICROBIOLOGY

The microflora in cheese can be divided into two main groups: starter and secondary flora. The starter flora, added at the beginning of manufacture or naturally present in the milk, is responsible for acid development during cheese production. Mesophilic and thermophilic starters, with optimal growth temperatures of ~30°C and 45°C, respectively, are used. The secondary flora is composed of complex mixtures of bacteria, yeasts and molds and contributes significantly to the specific characteristics of a particular cheese variety (Poças and Pintado, 2010).

A great variety of microbial species are involved in the ripening of cheese, the total population generally exceeding 10° organisms per gram. The principal bacterial groups involved in ripening are the lactic acid *Streptococci*, *Leuconostoc*, *Lactobacilli* and *Propionibacteria* species. *Micrococci* and *Corynebacteria* species may also be involved; they are aerobic and salt tolerant and thus grow especially on the surface of cheeses.

Yeasts are widely distributed in nature and are found in raw milk and some cheeses, with the basic flora in the majority of cheeses being species of the genus *Kluyveromyces*. Yeasts produce enzymes capable of degrading the constituents of the curd and so can contribute to modifying the texture of the cheese and to the development of flavor and aroma. They are capable of converting lactose into CO<sub>2</sub> and may also take part in lipid degradation.

Among the fungal flora found in or on mold-ripened cheeses, species of the genus Penicillium are of particular importance. Penicillium camemberti is the original mold of Camembert and Brie and has a single habitat—the surface of a few cheeses and the environment of cheese factories. By contrast, Penicillium roqueforti, a microaerophilic mold, is widely distributed and is the mold of mold-ripened cheese; it grows very well at quite low  $O_2$  levels (5%). Geotrichum candidum is present on certain soft cheeses where it forms a characteristic grayish-white crust on the surface. The role played by molds in ripening is a major one for soft and mold-ripened cheeses.

Fermentation of lactose to lactic acid during manufacture and the metabolism of residual lactose during the initial stages of ripening reduce the pH of cheese to around 5 depending on variety; at this pH, the growth of many pathogenic bacteria is inhibited. During ripening, the pH of the cheese rises due to the formation of alkaline N-containing compounds and/or the catabolism of lactic acid (Fox, 2011).

The influence of pH on microbial growth and enzyme activity is particularly decisive. Only lactic acid bacteria, yeasts and molds can grow at pH values below 5. Enzymes are also very sensitive to variations in pH, with most microbial proteases having greatest activity in the pH range of 5.0–7.5, and lipases in the range of 7.5–9.0. Curd at the end of drainage has a pH of less than 5.5 and is the site of active lactic acid fermentation. This acidic character of the curd is a necessity in that it slows down enzyme action and impedes the development of a harmful bacterial flora.

Soft cheese curd has a pH below 5, with the pH of Camembert increasing from 4.5 to over 6.0 in the interior and over 7.0 on the surface by the end of ripening (about 30 days). Hard cheeses have pH values around 5.2–5.3, and neutralization of the cheese is undesirable. The pH tends to remain at about this level during ripening, partly because of the high degree of mineralization of the cheese, which gives it a high buffering capacity.

# 19.5.3 PACKAGING REQUIREMENTS

The two key parameters contributing to the stability of cheeses are pH and  $a_{\rm w}$ . However, neither of these parameters is low enough to ensure complete stabilization of the product, with the result that

cheeses as a class lie between perishable foods on the one side and intermediate moisture foods on the other. While the packaging will have no influence on the pH of the cheese, the  $a_{\rm w}$  of the surface (and ultimately the interior) of the cheese will be affected by the WVTR of the packaging material.

Two other key environmental factors that must be considered in the packaging of cheese (and indeed in the packaging of all dairy products) are light and O<sub>2</sub>. As discussed earlier in this chapter, light initiates the oxidation of fats, even at temperatures found in refrigerated display cabinets. In unripened cheeses, this gives rise to off-flavors, which have been described as "cardboardy" or "metallic." Although lipid oxidation has been the main focus due to its high impact on flavor and off-flavor formation, the increasing production and sale of low fat dairy products, including low fat cheeses, makes the oxidation of amino acids and proteins of interest due to their higher impact on off-flavor formation in products with limited fat content. Sulfur compounds have been suggested to be of major importance in protein oxidation in dairy products and dimethyl disulfide (DMDS) has been identified as a major protein oxidation product in milk, including low fat cheeses (Dalsgaard et al., 2010). The generation of DMDS is reduced in vacuum-packed cheese when compared with cheeses packed in air.

Photooxidation of cheeses may be reduced by (1) minimizing light exposure, (2) optimizing the package barrier to light and (3) minimizing both the headspace volume and its residual  $O_2$  levels (Mortensen et al., 2004). A review of light-induced changes in packaged cheeses concluded that integrating results and conclusions on the effects of light on the sensory characteristics of packaged cheeses is very complicated because published reports contain various levels of detail about the experimental setups and methodologies. It was suggested that there is an urgent need for a more systematic approach to shelf life testing with respect to time, temperature, light sources and their intensity (Mortensen et al., 2004).

The oxidation reactions initiated by light may continue even if the cheese is subsequently protected from light (Hong et al., 1995) and any metallic ions contained in the packaging material will catalyze the reactions. In addition, the ingress of  $O_2$  through the packaging film is undesirable as it will contribute to the oxidation of fats and the growth of undesirable microorganisms.

For the purposes of providing a framework to discuss packaging requirements, the following classification scheme for cheeses will be used: very hard and hard, semisoft and soft, fresh, and processed.

# 19.5.3.1 Very Hard and Hard

The very hard class of cheese (sometimes referred to as *grating grade*) is ripened by bacteria and is characterized by a moisture content (on a fat-free basis) of <51%, (e.g., Parmesan 42%; Romano 31%; and Mozzarella 45%). The hard class of cheese is also ripened by bacteria and is characterized by a moisture content of 49%–63%, where the range 49%–56% is categorized as hard, and the range of 54%–63% is categorized as semihard. Cheeses in this class include Cheddar (still the cheese produced in the greatest quantities worldwide), Edam, Gouda, Cheshire, Gloucester, Derby and Leicester, as well as those with eyes such as Emmental and Gruyere. Also included in this class are Provolone, Mozzarella and Kasseri.

# 19.5.3.1.1 Rindless Ripening

Rindless cheese may be defined as cheese that has been ripened under a plastic film that allows little or no evaporation into the atmosphere to occur. Cheeses to which the technique of rindless ripening can be applied are obviously those in which the rind does not play an essential role in ripening. Such cheeses include the cooked, hard cheeses such as Emmental, as well as the uncooked hard cheeses such as Cheddar, Edam, Gouda and Saint Paulin. A feature of all these cheeses is that they do not have surfaces covered with molds, bacteria or yeasts producing enzymes responsible for ripening.

The development of the technique of ripening under film (also known as *rindless ripening*) was motivated by various factors. One was the need to produce blocks better suited to a high degree of mechanization. Another was to increase cost-effectiveness by replacing earlier techniques of

coating, thus reducing handling during ripening and losses due to drying-out. The first techniques (which appeared around 1930 and were used for cheddar) involved coating the blocks with mineral oil. In 1950, J.B. Stine of Kraft Foods filed two patents concerning the use of a plastic packaging material for blocks of Swiss cheese; these patents have since passed into the public domain. A further factor was the development of prepacked, self-service, consumer portions of cheese for or by supermarkets, where the losses resulting from removal of the rind were considered unacceptable. This led to the idea of rindless cheese (Fradin, 1987).

There are three variables which may be used to adjust the process of ripening rindless cheese: the permeability of the packaging material; the ripening temperature; and the ripening time. Discussion will be limited to the first of these variables.

Mineral waxes used for coatings on cheeses consist of refined hard paraffin, petroleum jelly and microcrystalline waxes with various additives. Paraffin wax comprises mostly saturated aliphatic unbranched alkanes, and a range of different colored waxes are used to differentiate cheeses. For example, Gouda cheeses are typically coated with yellow or white waxes, whereas Edam cheeses are coated with red wax. Wax protects the cheese from mold growth and weight loss through moisture evaporation and prevents aerobic ripening because of its barrier to O<sub>2</sub>. Mineral waxes present a higher barrier to O<sub>2</sub> than acetoglyceride-based waxes, which are also used for cheese. Water-based dispersions are typically copolymers of ethylene and vinyl acetate. These coatings may be uncolored or pigmented with different colors and are used as carriers for antifungal agents such as natamycin (E235), calcium sorbate (E203) and potassium sorbate (E202). These coatings are applied in one or several layers before the wax layer (Poças and Pintado, 2010).

Plastic films are widely used for packaging rindless cheese, and the specifications of some retail packages used for rindless cheeses are presented in Table 19.4. Unfortunately, the package dimensions and test conditions for the transmission rates were not specified, making the data of limited applicability.

One of the major problems faced by consumers of packaged cheeses is mold growth once the package has been opened. The incorporation of zip-lock press-to-close resealable technologies featuring flanged interlocking zipper profiles on plastic pouches for cheese has solved this problem. Resealable packages can also be made using a slider that features an ergonomically designed "clip" to enable consumers to easily open and close a package. Both technologies are ideal for premade pouches and form-fill-seal applications, and can run on virtually all packaging formats and machinery configurations.

Analysis of the gas found in the holes of, for example, an Emmental cheese revealed that it was composed of 95%  $\rm CO_2$  and 5%  $\rm N_2$ . This atmosphere protects the entire cheese against the development of molds (Stehle, 1987).

## 19.5.3.1.2 Role of Oxygen

The fate of  $O_2$  in cheese is still not completely understood. What is certain is that the gas present in the space between the packaging material and the cheese (either produced as a product of enzymic action in the cheese, left in the package after sealing or diffusing through the packaging material) determines whether or not microbial growth will occur on the surface of the cheese.

The extent to which oxidation occurs largely depends on the storage temperature and time; also important are the surface area:volume ratio of the cheese and the  $O_2$  permeability of the packaging material. The latter will vary with temperature and may vary with RH, depending on the chemical nature of the material.

Packaging films for Cheddar cheese must be sufficiently impermeable to  $\rm O_2$  to prevent fat oxidation and mold growth. Data on the minimum partial pressure of  $\rm O_2$  necessary for the development of molds are scarce. In the absence of  $\rm CO_2$ , molds will grow at  $\rm O_2$  partial pressures below 0.133 kPa. In the presence of  $\rm CO_2$  at partial pressures of 8.7–15 kPa, microbial growth is inhibited up to an  $\rm O_2$  partial pressure of 2.0–2.7 kPa.

TABLE 19.4
Specifications of Some Retail Packaging Materials Used to Package Rindless Cheese

Laminate		Transmission Rate (mL m <sup>-2</sup> day <sup>-1</sup> atm <sup>-1</sup> )			
Composition	Thickness (µm)	$O_2$	CO <sub>2</sub>	$N_2$	H <sub>2</sub> O
Sliced cheeses (with no gas production, e.g., Cheddar):					
Lower films PET-LDPE or	300/50	12	60	7	1.5
PET-LDPE	250/50	14	42	9	1.0
Upper films PET-LDPE	36/70	35	128	21	2.0
or PET-PET-LDPE	12/23/54	40	160	20	8.0
Sliced cheese (with slight gas production, e.g., Gouda) usin	ng hard box pack (Re	Ca):			
Cover film (OPET-LDPE)	23/75	85	340	43	2.0
Trough film (PET-HMb-LDPE) with migration barrier	200/25/25	5	20	2.5	1.5
Tubular bag (OPA-LDPE)	15/40	60	240	30	4.0
Sliced cheeses (with high gas production, e.g., Emmental)	using hard box pack	(RCa):			
Cover film (OPET-LDPE)	40/60	550	2200	275	1.5
Trough film (PET-HM-LDPE) with migration barrier	200/25/25	5	20	2.5	1.5
Tubular bag (OPA-LDPE)	10/50	90	360	45	4.0
Cheese portions without gas production:					
Tubular bag (OPA-LDPE)	15/40	4	16	2	4.0
Cheese portions with low gas production:					
Cover film (OPA-LDPE)	15/40	60	240	30	5.0
Bottom film (PA-LDPE)	15/120	40	160	20	1.5
Tubular bag (OPA-LDPE)	15/40	60	240	30	5.0
Grated cheese (e.g., Emmental):					
Tubular bag (OPA-LDPE)	15/40	60	240	30	5.0

Source: Schneider, Y. et al., Packaging materials and equipment, in: *Technology of Cheesemaking*, 2nd edn., Law, B.A. and Tamine, A.Y. (Eds), Blackwell, Malden, MA, pp. 413–439, 2010.

It is likely that problems of mold growth on hard cheeses packaged in the films in common use today are a function of hygienic conditions in the packing room, the degree of vacuum inside the package and the integrity of the heat seal, rather than the  $O_2$  permeability of the packaging material used.

# 19.5.3.1.3 Modified Atmosphere Packaging

Modified atmosphere packaging (MAP) is used particularly for portioned and sliced hard cheeses as they are more prone to deteriorative changes because of a larger surface area exposed to light and  $O_2$ , and, therefore, high barrier packaging must be used. Vacuum packaging may have a negative impact on the appearance of some cheeses, particularly those with eyes, such as Emmental, Edam and Gouda, which may collapse under reduced pressure. For more resistant cheeses, vacuum skin packaging is very common. In MAP, the gas mixture should be optimized for each cheese; a recent review (Khoshgozaran et al., 2012) helpfully summarizes the reported optimal MAs for a range of cheeses. Some cheeses withstand mixtures with compositions richer in  $CO_2$ ; others suffer from sensory problems and package collapse and, therefore, MAs with a lower percentage of  $CO_2$  and more  $N_2$  are used. Multilayer films composed of combinations of PA or EVOH as gas barriers and

<sup>&</sup>lt;sup>a</sup> RC is reclosable.

b HM is not melt.

polyolefin-based materials such as LLDPE, EVA copolymer and ionomers as moisture barriers and sealing layers are used (Poças and Pintado, 2010).

In a study evaluating various gas mixtures (Favati et al., 2007),  $CO_2$  and  $N_2$  (30:70) best preserved portioned Provolone cheese by slowing down the proteolytic and lipolytic phenomena typical of cheese ripening. This packaging system extended the shelf life to more than 9 months at 8°C, an increase of 50% compared with that obtained under vacuum packaging. The packaging film was  $20\,\mu m$  PA-80 $\mu m$  LDPE with an  $O_2$  permeability at 23°C of 7.7 barrer. Although the study indicated that 400 g portions of cheese were packaged, unfortunately, the surface area of the packs was not specified making the findings nontransferable. In commercial applications, pillow pouches of portions of Provolone are given a 6 month shelf life when refrigerated at 4°C–8°C.

Graviera hard cheese had a shelf life of 9 weeks when stored in the dark at 4°C under atmospheres of 100% N<sub>2</sub> or 50:50 CO<sub>2</sub>:N<sub>2</sub>, compared with 2–3 weeks for unpackaged cheese. The packaging material consisted of 75 µm LDPE-PA-LDPE with an OTR of 11 mL m<sup>-2</sup> day<sup>-1</sup> and WVTR of 1.29 g m<sup>-2</sup> day<sup>-1</sup> (temperature and humidity test conditions unspecified). Samples packaged under 100% CO<sub>2</sub> developed a bitter score after 5 weeks of storage (Trobetas et al., 2008). Arvanitoyannis et al. (2011) investigated the shelf life of Graviera cheese stored at 4°C for up to 90 days in 90 µm PA-LDPE pouches under three different MAs: MA1 40% CO<sub>2</sub>:55% N<sub>2</sub>:5% O<sub>2</sub>; MA2 60% CO<sub>2</sub>:40% N<sub>2</sub>; MA3 50% CO<sub>2</sub>:50% N<sub>2</sub>. Control cheeses were packaged in air. Pouch barrier properties were given as OTR 40-50 mL m<sup>-2</sup>, CDTR 150 mL m<sup>-2</sup> and N<sub>2</sub>TR 10 mL m<sup>-2</sup>, but temperature and humidity test conditions were not specified, and neither were the pouch dimensions. The microbiological analysis revealed that there were no colonies of Staphylococcus aureus and Listeria monocytogenes, whereas both Escherichia coli and total viable counts increased strongly in control samples but were inhibited under all MAP compositions. After 10 days of storage, the sensory characteristics of the control cheeses were found to be unacceptable. The most effective mixtures for inhibiting the growth of E. coli were MA2 and MA3. Both MA1 and control samples had a very negative effect on sensory quality.

The effect of an oxygen scavenger combined with an ethanol emitter (EE) and a MA of  $100\% N_2$  in combination with a high barrier experimental  $SiO_x$ -coated PET-LDPE film on shelf life extension of grated Graviera cheese stored for 10 weeks at  $4^{\circ}C$  and  $12^{\circ}C$  was investigated by Mexis et al. (2011). Sensory shelf life was  $\sim 1$ , 1.5, 4.5, 6, 9 and at least 10 weeks for control samples ( $12^{\circ}C$  and  $4^{\circ}C$ ), for  $N_2$  packaged samples ( $12^{\circ}C$  and  $4^{\circ}C$ ) and samples packaged with the OA + EE ( $12^{\circ}C$  and  $4^{\circ}C$ ), respectively.

The introduction of pure  $CO_2$  into the package often produces the appearance of vacuum packaging after a certain period of storage. This is the result of absorption of  $CO_2$  by the cheese as well as some loss by diffusion through the packaging material. Because the permeability of polymer films to  $CO_2$  is approximately four times that of  $O_2$  or  $N_2$ , the rate of loss of  $CO_2$  is greater than the rate at which these other gases can permeate in from the surrounding atmosphere. As a result, there is a decrease in volume of the package and the packaging material collapses around the cheese. A similar phenomenon was described in Chapter 18 in connection with MAP of fruits and vegetables. The more  $CO_2$  which the cheese has "lost" at the time of cutting/slicing and packaging, the greater the absorption of  $CO_2$  from gas flushing and the greater the contraction of the package volume. Thus, in the gas flush packaging of cheese slices it is recommended that a mixture of gases ( $CO_2$  and  $O_2$  in the ratio 80:20 or 70:30) be used to avoid the slices being pressed against each other by atmospheric pressure (Alves et al., 1996).

Unattractive calcium lactate crystals (CLC) on Cheddar cheese have been documented since the 1930s. Agarwal et al. (2005) reported heavy CLCs on the surfaces of all gas-flushed but not on vacuum packaged cubes ( $1 \times 1 \times 4$ cm) after 12 weeks storage at 7°C, regardless of the gas composition. Although milk composition and the presence of nonstarter lactic acid bacteria can contribute to the development of CLC on cheese surfaces, gas flushing is the most important factor.

#### 19.5.3.2 Semisoft and Soft

This class of cheese is also ripened by bacteria and is characterized by a moisture content of 61%–69% on a fat-free basis and 43%–55% on a total basis. Soft cheeses are cheeses which, independently of lactic acid fermentation, have undergone other ripening processes. The body is neither cooked nor pressed and may contain internal mold.

Examples of semisoft and soft cheeses include those that have been ripened by bacteria such as Brick and Munster, those ripened by bacteria and surface microorganisms such as Limburger and Trappist, those ripened by surface mold such as Brie and Camembert and those ripened by internal (blue) mold such as Roquefort, Gorgonzola and Stilton. Other semihard cheeses include Havarti, Samsø and Edam.

# 19.5.3.2.1 Light

Sliced, rindless Havarti cheese packaged in an atmosphere of 25%  $\rm CO_2$  and 75%  $\rm N_2$  and stored under light for up to 21 days at 5°C showed a decrease in yellowness, an increase in redness and no significant changes in lightness (Kristensen et al., 2000; Mortensen et al., 2002). Sliced, rindless Samsø cheese packaged in atmospheres of  $\rm CO_2$  and  $\rm N_2$  (0:100; 20:80 and 100:0 with residual  $\rm O_2$  at time of packing <0.24%) and stored under varying light conditions for up to 21 days at 5°C also showed significantly decreased yellowness and increased redness (Juric et al., 2003). Cheese stored in 100%  $\rm CO_2$  had significantly lower lightness and was described by the sensory panel as having a rancid taste and odor, as well as a dry/crumbly texture.

In contrast to fresh cheeses, semisoft and soft cheeses require limited protection against light to maintain quality. When the cheese is ripe enough to be packaged, the influence of light on soft cheeses with a surface mold is of little importance. Although light can slow down or hinder the germination of conidia, there is no such hindering effect with thick layers of mycelia at an advanced stage of ripening. The same applies to red smear, and light does not even reach the internal mold of blue-veined cheeses unless they are sliced. Mold mycelia, as well as the rind, which forms during ripening, also provide protection against light.

# 19.5.3.2.2 Gases

The consumption of  $O_2$  and the production of  $CO_2$  are closely connected with the total bacterial count and the ripening conditions of temperature, humidity, pH,  $a_w$  and so on. In the case of Camembert, for example, there is an increase in the starter bacterial count to about  $10^{10}\,\mathrm{g}^{-1}$  after the first turning, declining more or less rapidly until ripe for packaging to stabilize at about  $10^7-10^9\,\mathrm{g}^{-1}$ . For a Camembert weighing  $100\,\mathrm{g}$ , the peak  $O_2$  requirement is about  $15\,\mathrm{mL}\,\mathrm{h}^{-1}$  with a  $CO_2$  release of  $10\,\mathrm{mL}\,\mathrm{h}^{-1}$ . On further ripening prior to packaging, this drops to about  $4.5\,\mathrm{mL}\,\mathrm{h}^{-1}$  for  $O_2$  and about  $3.6\,\mathrm{mL}\,\mathrm{h}^{-1}$  for  $CO_2$ .

Measurements taken of unpackaged and packaged Camembert have shown that the unpackaged cheese loses about 0.04% of its weight per hour during ripening, whereas packaged cheese loses about 0.006%  $h^{-1}$  as water vapor (International Dairy Federation, 1987). These figures were obtained at 10°C, and temperature clearly has an important influence. Thus, at 20°C, the  $O_2$  requirement is 11 mL  $h^{-1}$  and at 30°C it is 17.5 mL  $h^{-1}$ . The surface area:volume ratio is also important in determining the level of  $CO_2$  release, and the greater the surface area for a given cheese weight, the higher the  $CO_2$  release.

The effects of MAP on the growth of L. monocytogenes in mold-ripened Stilton cheese during refrigerated storage over a 6 week period have been studied (Whitley et al., 2000). When samples were inoculated with L. monocytogenes and stored under MAP at  $N_2$ : $CO_2$ : $O_2$  ratios of 80:10:10, 100:0:0 and 80:20:0, a significant decrease in count was found in samples stored in the 80:10:10 atmosphere. A greater inhibitory effect was achieved when  $CO_2$  concentration was increased to 20% than by reducing the  $O_2$  content. Results indicated that an 80:10:10 ratio is not suitable for use with blue Stilton cheese when L. monocytogenes may be present.

# 19.5.3.2.3 Humidity

The growth of microorganisms in and on soft cheese is dependent on the  $a_{\rm w}$  of the cheese, which is clearly influenced by the water vapor permeability of the packaging material. If the humidity of the

air under the package becomes too high, then the fungus mycelia begin to turn yellow and exude liquid in the form of droplets, finally showing autolytic symptoms. This results in a clear shift of the flora composition toward hydrotrophic bacteria such as *Brevibacterium linens*. Conversely, if the permeability of the packaging material is too low, the growth of mold or surface smear may be stopped, resulting in the appearance of facultative anaerobic bacteria with strong proteolytic activity.

Most polymeric packaging materials are too impermeable to be used for soft cheeses and must, therefore, be perforated before use. No definite rules can be laid down with regard to the number and size of the perforations as these must be found experimentally for the particular type of cheese. It should also be borne in mind that soft cheeses of the same type (e.g., Camembert) may behave differently depending on the method of production.

In some countries, it is permissible to treat the packaging materials with fungicide to prevent subsequent mold growth on the surface after packing. Typically, the packaging material is impregnated with sorbic acid or its salts, where the concentration needs to be sufficiently high (and the packaging material sufficiently close to the cheese surface) in order to prove successful.

# 19.5.3.2.4 Ripened by Internal Mold

The packaging material for internal molded cheese should allow for the passage of  $O_2$  to promote mold development in the curing channels of the cheese. The package should also allow for a certain permeability to  $CO_2$  and water vapor (Odet and Zachrison, 1982). However, blue-veined cheeses seem to be less dependent on the gas permeability of the packaging material than other soft and semisoft cheeses. For example, *P. roqueforti* grows in  $O_2$  concentrations as low as 5%, as found in the curd holes of the cheese. The presence of  $CO_2$  seems to stimulate growth, and therefore more impermeable packaging materials such as aluminum foil, PP film or thermoformed packages made from rigid PVC or PS with a transparent multilayer film lid for portions have proved effective (International Dairy Federation, 1987).

# 19.5.3.2.5 Ripened by Surface Mold

In surface mold-ripened cheeses such as Camembert and Brie, lactate is metabolized to  $CO_2$  and  $H_2O$  by the activity of *P. camemberti*. For these cheeses, it is important that packaging does not take place until the mold has grown to a certain extent. The packaging material must have a limited permeability to  $O_2$  to minimize the risk of development of anaerobic proteolytic bacteria, which can also develop if the permeability to water vapor is too low resulting in condensation inside the package. The material should not adhere to the surface mold of the cheese (Odet and Zachrison, 1982).

A suitable material for the packaging of these cheeses is perforated OPP, where the perforations are necessary to allow the passage of controlled quantities of water vapor. Paper is also used for packaging but, if it is in direct contact with the surface of the cheese, it must be coated with wax or laminated to perforated film to avoid decomposition of the paper by cellulase enzymes produced by certain molds. Aluminum foil is also used at a thickness of 7–9 µm if laminated to other materials and 12 µm if used alone. It must be given a protective coating of lacquer otherwise NH<sub>3</sub> (a metabolite in the cheese ripening process) many corrode the metal, turning it black. It is preferable not to perforate the inner layer in direct contact with the surface of the cheese in order to avoid mycelia growing through the perforation and becoming apparent on the outside of the package.

Thermoformed packages made from PS and PVC are used, sometimes extrusion coated with LDPE to reduce WVTRs. Special packages for the sterilization of Camembert are made from combinations, which consist of OPP-PVdC copolymer-PP, PET-PVdC copolymer-PP or OPA-PP-PVdC copolymer for deeper containers with higher puncture resistance (Stehle, 1987).

With few exceptions, soft cheeses are placed in an additional outer package prior to marketing. Traditionally, these consisted of wood, but the use of paperboard or a combination of a plastic base and a paperboard cover has become the norm. The plastic bases (PVC or HIPS) are designed in such a way that corrugation and air channels in the base ensure that sufficient gas and water vapor exchange occur.

Soft-mold (Camembert-type) cheeses are packaged early in the ripening process as soon as *P. camemberti* mycelium covers the cheese surface. The effect on cheese-ripening dynamics of four films with different WVTRs (ranging from 1.6 to 500 g m<sup>-2</sup> day<sup>-1</sup> at 38°C and 90% RH) was compared to unwrapped cheeses by Picque et al. (2010). The packaged cheeses were stored at 6°C and 75% RH. Water loss ranged from 0.5% to 12% on day 23, compared with 15% for unwrapped cheeses, and this appeared to be a key factor in controlling cheese-ripening progress. Low water losses (from 0.5% to 1% on day 23) led to over-ripening in the cheese underrind, which became runny as a result. Water losses of around 3%–6% on day 23 led to good ripening dynamics and the best cheese quality. This level of water loss appeared to be ideal in terms of cheese film design.

The shelf life of Stracciatella cheese (similar to Mozzarella) packaged in various MAs of CO<sub>2</sub>:N<sub>2</sub>:O<sub>2</sub> gas mixtures (50:50:0 [M1], 95:5:0 [M2], 75:25:0 [M3] and 30:65:5 [M4]) and stored at 8°C was investigated by Gammariello et al. (2009). Cheeses in traditional tubs and under vacuum were used as the controls. Results showed that MAP, in particular M2 and M3, delayed microbial growth of spoilage bacteria, which determined end of shelf life; the sensory quality did not limit shelf life.

Recently, Rodriguez-Aguilera et al. (2011) compared MAP of a soft surface mold ripened full fat (45%) cheese (St. Killian) under two atmospheres: A (0%  $O_2$ : 27%  $CO_2$ ) and B (2%  $O_2$ :19%  $CO_2$ ) at 12°C with an existing commercial packaging system (a wax layer in contact with the cheese surface, a paper layer and an outer layer of varnish, and inserted in an open cardboard box). The predicted shelf life was found to be 14, 6 and 17 days for control, MAP-A and MAP-B, respectively. It was concluded that MAP of surface-mold-ripened cheese with low levels of  $O_2$  (1%–3%) and relatively high levels of  $CO_2$  (17%–21%) can be used to extend the shelf life of soft cheese by 20%. However, the package has to be suitably designed, as total loss of  $O_2$  (as in MAP-A) would shorten the shelf life; a high barrier plastic container was suggested but not tested. The shelf life of the control is  $\sim$ 4 weeks when stored at 4°C.

# 19.5.3.2.6 Ripened by Smear Coat

The quality of smear-coated cheeses, such as Havarti, Limburger and Munster, is strongly dependent on the vitality of the surface culture (often  $B.\ linens$ ), which is related to the RH inside the package. These cheeses are sometimes wrapped in clear or orange pigmented OPP combined with greaseproof paper. Vegetable parchment is also recommended in view of its mechanical strength when wet, and its consequent moistening function for the surface flora of the cheese, which require a high  $a_{\rm w}$  for their growth. Although vegetable parchment is sometimes used alone, it is more often used in a combination laminated to aluminum foil or an aluminum/tissue paper laminate to give a shelf life of 6–10 weeks.

#### 19.5.3.3 Fresh

Fresh cheeses are slow drainage cheeses, which have been subjected to lactic acid fermentation; they are characterized by a moisture content of >80%. The three major types of fresh cheeses are Cottage, Quark and Petit Suisse.

# 19.5.3.3.1 Manufacturing Processes

19.5.3.3.1.1 Cottage Cottage cheese is normally prepared from pasteurized skim milk by the in situ production of lactic acid by starters consisting of Streptococcus lactis, Streptococcus cremoris and Leuconostoc citrovorum (for flavor). The desired pH is  $\approx$ 4.6, which may be reached in 5–16 h depending on the level (0.5%–5%) of starter addition and the set temperature (22°C–32°C). The coagulum is cut and cooked to 50°C–55°C over a 1.5 h period during which time considerable syneresis occurs and the curd assumes a firm, meaty texture. After removable of the whey and washing of the curd, salt ( $\approx$ 1% NaCl) is added, and the curd mixed with a creaming mix to give a level of  $\approx$ 4% fat in the finished cheese. A good quality product has a shelf life of 1–2 weeks at chill temperatures, which may be extended by packaging in an atmosphere of CO<sub>2</sub> (Fox, 2011).

19.5.3.3.1.2 Quark Quark is produced in a very similar manner to Cottage cheese, with Streptococcus diacetylactis replacing L. citrovorum for flavor development. After setting, the coagulum is broken and the whey removed from the uncooked curd/whey mixture by filtration or centrifugation. Compared to Cottage cheese, Quark has a smooth consistency and a significant lactose content since it is not washed. Quark typically has a moisture content of 82% (Fox, 2011).

19.5.3.3.1.3 Cream and Petit Suisse These are produced by in situ production of acid in cream and typically have a moisture content of 54%. After coagulation, the curds are separated from the whey by filtration or centrifugation. Gums may be added to cream cheeses to improve texture and consistency, and they may also be heat treated and homogenized to produce a product with a longer shelf life (Fox, 2011). Cream cheese has a shelf life of 3–6 months, while Petit Suisse has a shelf life of 3–4 weeks (International Dairy Federation, 1987).

# 19.5.3.3.2 Packaging Requirements

The packaging requirements for fresh and cream cheeses are basically similar to those for the other types of cheeses, namely protection against light,  $O_2$  and loss of moisture.

Due to their high moisture content, low salt concentration and high pH, fresh cheeses are susceptible to microbial spoilage and consequently have a limited shelf life. They are also very sensitive to dehydration, and most fresh cheeses keep draining slowly. Because of their high  $a_{\rm w}$ s, the adsorption of moisture from the atmosphere is of little importance but loss of water through evaporation, particularly from the surface, must be avoided. Although fresh cheeses do not seem to be as sensitive to the influence of light as milk, cream or butter, the cream cheeses with their greater fat content ( $\approx 34\%$ ) are. For all the cheeses in this class, the packaging must provide protection against light transmission. The  $O_2$  in fresh cheeses may either be present in the cheese as a result of the processing techniques used (e.g., centrifugation), in the headspace inside the package, or permeate through the package over time. Some of these cheeses are packed under a low  $O_2$  MA.

## 19.5.3.3.3 Packaging Materials

While a number of plastics have been used over the years, the standard material is HIPS, which is thermoformed on form-fill-seal machines. It is also coextruded or extrusion coated with PVdC copolymer to improve its barrier properties, and pigmented with TiO<sub>2</sub> to provide a better barrier to light. Injection molded containers made of HDPE or PP with slits in the side to allow drainage of whey are also used, with the fresh cheese being ladled directly into the containers. Outer packaging of PA-LDPE laminates make these containers gas tight.

The shelf life of Cottage cheese, without chemical preservatives, stored at 3–4°C is 14–21 days. Flushing the headspace (25%) of commercial packages of Cottage cheese with pure CO<sub>2</sub> extended the shelf life at 8°C by about 150% without altering the sensory properties or causing any other negative effects (Poças and Pintado, 2010).

Genuine vegetable parchment or greaseproof paper was frequently used in the past to package fresh cheese and is still used in some markets for Petit Suisse. It is usual for the paper to have a basis weight of 40–60 gsm. Paper coated with paraffin or PVdC copolymer is still sometimes used in the form of a banderole; for example, for packaging an unripened cheese intended for consumption within a short time (Stehle, 1987).

Aluminum foil with a thickness of 7–20 µm can be used, with the thicker foils (15–20 µm) formed into containers of either rectangular shape with straight walls, or cylindrical section with corrugated or pleated sides. In all cases, the aluminum must be protected against corrosion, either by applying a suitable enamel or by laminating with LDPE or PP. If this is not done, then the whey coming into contact with the aluminum will cause the formation of aluminum lactate and attack the walls of the container, sometimes perforating them (Stehle, 1987). MAP has been suggested to maintain the quality of Cottage cheese (Maniar et al., 1994).

#### 19.5.3.4 Processed Cheese and Analogues

#### 19.5.3.4.1 Manufacture

Attempts at the end of the nineteenth century to export hard cheeses from Europe to tropical countries were largely unsuccessful. This led to the development by the Swiss company Gerber in 1911 of "processed" cheese that was made by removing the rind from Gruyère or Emmental cheese and heating it to about 80°C while stirring in a solution of sodium citrate as an emulsifying salt. The cheese formed a "sol," which could be packaged in a metal foil while hot. On cooling, this gave a gel, which was pleasant to eat, had a taste reminiscent of the original cheese and (if the acidity was properly controlled) had good keeping qualities. In 1916, processed cheese was manufactured in the United States by J.L. Kraft from Cheddar cheese using a mixture of citrates and orthophosphates as emulsifying salts (Kapoor and Metzger, 2008).

Processed cheese is produced by blending natural cheese of different ages and degrees of maturity in the presence of emulsifying salts and other dairy and nondairy ingredients, followed by heating and continuous mixing to form a homogeneous product with an extended shelf life. Today, a wide range of processed cheeses is available containing a variety of flavoring compounds and sometimes fruits, vegetables and/or nuts. Preservatives such as sorbic and propionic acids and their salts may be added to prevent mold growth. Nisin may be added to prevent the growth of anaerobic spore-formers such as *Clostridia* spp. The addition of antioxidants is also permitted in many countries. Generally, the hot processed cheese is filled into packages such as pouches or polymer-coated aluminum foils, after which the packages are sealed and the product is cooled.

# 19.5.3.4.2 Packaging

A detailed discussion of packaging materials and equipment has been presented by Buys and Mostert (2011). Traditional packaging of processed cheese consisted of triangular portions (usually weighing 20–30 g) packaged in tinfoil (97% tin, 3% antimony and traces of lead, copper and iron) because of its resistance to corrosion by processing salts. For economic and technical reasons, tinfoil was replaced by heat sealable, lacquered aluminum. The thickness of the aluminum varies from 12 to  $15\,\mu m$ . An opening device consisting of narrow strips of PET film sealed onto the inner side of the aluminum foil is provided to facilitate opening a portion of the cheese. The strips extend several millimeters beyond the packaging material so that they can be grasped between two fingers. Their point of exit from the packaging must itself be sealed to avoid any possibility of leakage or contamination. The strips are normally colored red to attract the consumer's attention (Stehle, 1987). The triangular portions are often assembled into a circular paperboard carton or plastic container complete with lid.

Typical packaging systems used for spreadable processed cheese are (Poças and Pintado, 2010):

- 1. Squeezable nonbarrier tubes made of LDPE, high barrier tubes made of multilayer materials containing EVOH as a barrier layer, or metal tubes
- 2. Cups made of PP, PET-LDPE or PS-EVOH-LDPE heat sealed with alufoil or plastic laminate
- 3. Glass cups heat sealed with an alufoil plastic laminate or with an easy-open tinplate cap

Slices of processed cheese were first marketed in the United States in 1950, and were manufactured either by forming strips of cheese, which were then cut up and packaged, or by molding the cheese in the form of a tube around which a web of plastic was wrapped, with the whole assembly then being flattened and cooled. The films most often used are laminates of PET-LDPE, or if a more impermeable package is required, PET-PVdC copolymer-LDPE or OPP-EVOH copolymer-LDPE (Stehle, 1987).

A Brazilian processed cheese (Requeijão cremoso) was stored for 60 days at 10°C in five different packages: (1) glass cup with an easy-open tinplate cap, (2) glass heat sealed with aluminum foil-based laminate and plastic cover, (3) PP cup heat sealed with aluminum foil-based laminate and

plastic cover, (4) LDPE-HDPE squeeze tube and (5) coextruded LDPE-HDPE squeeze tube with an EVOH copolymer  $O_2$  barrier (Alves et al., 2007). When all the packages were stored under light (1000 lx), the shelf life in terms of overall sensory quality was only 17 days for the nonbarrier tube (4). Because of their shape, the squeeze tubes presented the largest surface area exposed to light, followed by the glass containers; the PP cups had the smallest surface area exposed to light. The stability of the product was similar for the coextruded tubes (5), PP cups (3) and non-vacuum-sealed glass cups (1) due to the combined effect of the initial  $O_2$  available and the package barrier to  $O_2$  and light. Among the types of packages studied, the vacuum-closed glass (2) preserved the initial quality of Requeijão cremoso for the longest period (32 days) as a result of the minimal amount of available  $O_2$  contained in its headspace. The results confirmed that it is not enough that a package has good gas barrier properties; it is also necessary to reduce the amount of headspace  $O_2$  and use materials that offer a barrier to light.

Some processed cheese is packaged in either tinplate or aluminum cans, both of which must be adequately enameled inside to prevent corrosion occurring. A small quantity of spreadable processed cheese is packaged in tubes, which used to be manufactured from aluminum but are now made of five-layer laminates containing aluminum foil as the central core. The tubes are filled through the unsealed base opening, which is then welded by a high-frequency current (Stehle, 1987).

# 19.6 MILK POWDERS

## 19.6.1 Manufacture and Properties

Milk and milk products are dried mainly by spray drying. This involves converting concentrated milk into a fog-like mist (atomizing) where it is given a large surface area and exposing this mist to a flow of hot air in a drying chamber. When the atomized product is in contact with the hot air, the moisture evaporates quickly, and the solids are recovered as a powder consisting of fine, hollow, spherical particles with some occluded air.

An important quality attribute of milk powder is the bulk density. It is obviously of considerable interest from an economic point of view as it influences the cost of storage, packaging and transport. The bulk density is governed chiefly by the total solids of the feed to the atomizer but also by the temperature of the drying air. Agglomeration also has a marked affect on the density, with a heavily agglomerated particle being very light.

Instantization produces milk powder with better rehydration properties (e.g., wettability, sinkability, dispersibility, solubility and rate of dissolution) by using two or three-stage drying. Instantization is based on agglomeration, which enables a larger volume of air to be incorporated between the powder particles, resulting in a characteristic coarse, cluster-like, agglomerated structure (Schuck, 2011a). The instantizing process for skim milk powder (SMP) consists of agglomeration of the particles into porous aggregates of sizes up to 2–3 mm. As a result, the amount of interstitial air (i.e., the air between the particles) is increased; reconstitution commences when the interstitial air is replaced by water. Powder agglomeration may be regarded as intentional caking as a result of forced compaction under controlled conditions.

Instantization results in a reduction of bulk density (e.g., for SMP from 0.64 to 0.55g mL<sup>-1</sup>, although the bulk density of agglomerated particles can be as low as  $0.35-0.40 \,\mathrm{g}$  mL<sup>-1</sup>). The free space volume has an important influence on the rate of oxidation of foods. If a food is packaged in air, a large free space volume is undesirable because it constitutes a large  $O_2$  reservoir. Conversely, if the product is packaged in an inert gas, then a large free space volume acts as a large "sink" to minimize the effect of  $O_2$  transferred through the package. It follows that a large package surface area and a low bulk density result in greater  $O_2$  transmission.

The instantizing process for whole milk powder (WMP) is more complicated due to the hydrophobic nature of the fat. Reconstitution of the agglomerates will not take place in water at temperatures below 45°C unless the powder particles have been coated with a surface-active or wetting agent.

The use of lecithin as a surface-active agent is accepted worldwide and it is mixed with butter oil and sprayed at 70°C onto the powder (which should be  $\sim$ 50°C) to give a final concentration of 0.2% in the powder. The best results are obtained when the powder is packaged at this temperature, but because the hot powder is very vulnerable to oxidation of the fat, it must be packaged with inert gas to reduce the  $O_2$  level in the package to a maximum of 2%.

SMP typically has a fat content of 1%, lactose 51%, protein 36%, mineral 8.5% and moisture content 3.5%, while WMP has a fat content of 28%, lactose 37%, protein 28%, mineral 6% and moisture content 3.0%. Buttermilk (the liquid remaining after the manufacture of butter) can be evaporated and spray dried, as can whey, which results from the manufacture of casein, quark and cheese. Infant milk formula contains added lactose and whey powders in order to increase the lactose content to that of human milk, which is significantly higher (7.0%–7.5%) than that of bovine milk (3.5%–4.0%). However, a higher lactose content presents greater challenges when drying such milks.

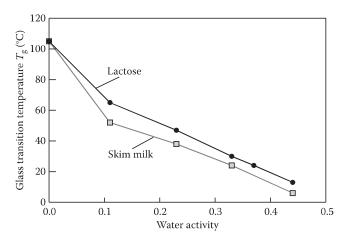
The physicochemical properties of free and bound water affect the physical state, transition temperatures, sticking temperature, reaction kinetics and stability of milk powders. The quality and shelf life of dairy powders are significantly dependent on the physical state of both lactose and other carbohydrates, which themselves are dependent on  $T_{\rm g}$  and  $a_{\rm w}$ . In fresh spray dried milk powders, lactose exists in a metastable amorphous state, because the rapid rate of concentration and spray drying does not allow sufficient time for it to crystallize. During storage, an increase in temperature or  $a_{\rm w}$  enhances an irreversible transition to stable crystalline forms which will occur if  $T_{\rm g}$  is reduced to below the powder temperature (Fitzpatrick et al., 2007).

Murrieta-Pazosa et al. (2011) studied water diffusion in SMP and WMP and related it to surface composition of the dairy powders. Their results suggested that powder microstructure and the chemical state of the key components could play an important role in determining water diffusivity. Silalai and Roos (2010) showed that  $T_{\rm g}$  can be used to describe time-dependent stickiness and crystallization phenomena in milk powders. The  $T_{\rm g}$ s of lactose and SMP as a function of  $a_{\rm w}$  at 24°C are shown in Figure 19.4.  $T_{\rm g}$  is usually well above the storage temperature for most dry powders but this may not be the case when powders produced in temperate climates are marketed in, or shipped through, tropical climates.

# 19.6.2 DETERIORATIVE REACTIONS

# 19.6.2.1 Oxidation

The detrimental effect of  $O_2$  on the flavor of milk products (especially those high in fat) has already been discussed earlier in this chapter. Lipid peroxidation is responsible for changes in the taste and



**FIGURE 19.4** Glass transition temperatures  $T_{\rm g}$  of lactose  $\bullet$  and skim milk powder  $\square$  as a function of water activity  $a_{\rm w}$  at 24°C. (Adapted from Silalai, N. and Roos, Y.H., *J. Food Sci.*, 75, E285, 2010.)

odor of milk powders through the development of off-flavors caused by the formation of secondary reaction products (alkanes, alkenes, aldehydes and ketones) and thus limit the shelf life of milk powders.  $O_2$ , light exposure, storage temperature,  $a_{\rm w}$  and concentration of unsaturated fatty acids are the most important factors that affect oxidation.

#### 19.6.2.2 **Browning**

Maillard or nonenzymic browning of milk powders during prolonged storage at moderate to high temperatures is initiated by condensation of lactose (functioning as a reducing sugar) with the free amino group of lysine in milk proteins (Thomas et al., 2004). In addition to the undesirable change in color, Maillard browning can also give rise to undesirable flavors.

# 19.6.2.3 Caking

Caking is a problem that arises when a low-moisture, free-flowing powder becomes lumpy, then agglomerates into a solid and finally transforms into a sticky mass. Many methods have been proposed for eliminating, minimizing or controlling caking, e.g., (1) controlling storage conditions below  $a_{\rm w}$  0.57 if no amorphous lactose is present, and below  $a_{\rm w}$  0.25 if it is present; (2) avoiding mixing of powders with different initial  $a_{\rm w}$ s and temperatures; (3) cooling the powder immediately to an appropriate temperature (well below  $T_{\rm g}$ ) before packaging; and (4) minimizing temperature variation during storage. Although these techniques delay caking, they do not prevent it.

# 19.6.3 PACKAGING REQUIREMENTS

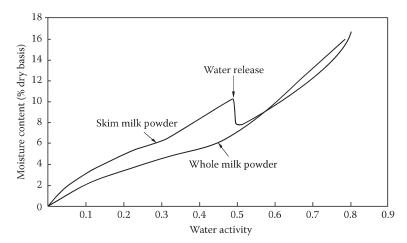
## 19.6.3.1 $O_2$ Permeability

The most effective method of extending the shelf life of milk powder is to package it in a high  $O_2$  barrier package from which air has been removed and replaced by an inert gas such as  $N_2$ . This is particularly true for WMP where the shelf life is governed to a large extent by the rate of oxidation of the unsaturated fats and the consequent development of objectionable flavors. The advantage of gas packing SMP is much less but it has been found to be worthwhile in preventing the development of stale flavors, especially where the storage period might be prolonged or the storage temperature is high. An alternative to gas packing is vacuum packaging and this is discussed in Section 19.6.5.2.

# 19.6.3.2 Water Vapor Permeability

The rate of development of oxidized flavor or oxidative rancidity during the storage of WMP depends on both the  $O_2$  concentration and the  $a_w$ . Therefore, if the maximum shelf life possible is to be obtained from milk powders (especially WMPs), it is important that the moisture content corresponds to the  $a_w$  at which the rate of lipid oxidation is a minimum. This is usually taken to be the  $a_w$  which corresponds to the monolayer value. The  $a_w$  of WMPs is mainly controlled by the moisture content of the nonfat solids, because fat has no influence. Thus, differences in  $a_w$  of different kinds of dairy powders are mostly the result of the state of the proteins and the physical state of the lactose (Schuck, 2011b).

A generalized MSI for SMP and WMP at 20°C is presented in Figure 19.5 and shows a break for SMP between 0.4 and 0.5  $a_{\rm w}$ , owing to lactose crystallization (Thomas et al., 2004). In the case of WMP, lactose crystallization does not occur until  $\geq$ 0.66  $a_{\rm w}$ , due to the role of milk fat, which is believed to act as a hydrophobic barrier and limits the diffusion of hydrophilic molecules and the growth of lactose crystals (Kelly, 2009). If the moisture content is calculated on a nonfat basis, the isotherms for SMP and WMP are almost identical. The  $a_{\rm w}$ s of WMP typically vary from 0.25 to 0.35 and for SMP from 0.32 to 0.43 (Tehrany and Sonneveld, 2010). During crystallization, the amorphous lactose initially absorbs moisture from the surroundings due to its hygroscopic nature, and subsequently releases moisture as it crystallizes. At low  $a_{\rm w}$ s, lactose generally occurs in the anhydrous form, the less hygroscopic  $\alpha$ -lactose monohydrate containing about 5% water as water of hydration.



**FIGURE 19.5** Generalized moisture sorption isotherms of skim and whole milk powders at  $20^{\circ}$ C. A break (water release) appears in the skim milk isotherm just below  $0.5~a_{\rm w}$  because of lactose crystallization. This break is not observed if experimental points are not close enough. (From Thomas, M.E.C. et al., *Crit. Rev. Food Sci. Nutr.*, 44, 297, 2004.)

The change in crystal structure from the anhydrous to the hydrated form can, therefore, only take place when the moisture content exceeds 5%. The proportion of hydrate increases as  $a_{\rm w}$  rises above 0.5.

In selecting a suitable packaging material for milk powders, three factors must be taken into account: the initial moisture content of the powder, the final acceptable (critical) moisture content of the powder and the shelf life required. Assuming that the powder equilibrates rapidly whenever moisture enters the package, the maximum quantity of moisture, which can enter the package can be calculated and the maximum water vapor permeability of the packaging material specified. This will vary depending on the surface area of the package and the weight of dry solids in the package, a point often overlooked when pack sizes are changed without any consideration of the resultant effect on product moisture content. These factors were discussed earlier in Chapter 12.

# 19.6.3.3 Light

It is necessary to protect milk powders (especially those made from whole milk) from light, otherwise oxidative reactions will be accelerated. The nature of these reactions and the light barrier properties of various packaging materials have been discussed earlier in this chapter.

# 19.6.4 PACKAGING MATERIALS

#### 19.6.4.1 Metal Cans

The traditional method for packaging milk powders for consumers uses three-piece tinplate cans where the atmospheric air is withdrawn from the powder and replaced with an inert gas such as  $N_2$  prior to seaming the base onto the can. When correctly seamed, the can is essentially impermeable to  $O_2$ , water vapor and light, and can be filled at high speeds. Its mechanical strength facilitates transport and handling, and the reuse possibilities of the empty can contribute to its popularity in many parts of the developing world. It is usual for the top of the can to have a lid, which can be levered off and, in order to provide a gas-tight seal under the lid, an aluminum foil diaphragm is sealed to the rim of the can. This is punctured by the consumer immediately prior to use. The use of an easy-open lid incorporating a ring-pull made from scored aluminum is now quite common; a plastic overclosure is supplied to provide a limited degree of protection once the metal end has been removed.

Milk powder has a long shelf life when packed in metal cans due to their excellent barrier properties. The exchange of moisture and  $O_2$  and the influx of light are not possible. Powders with a higher fat content are more susceptible to oxidation, and most powders are susceptible to deteriorative effects such as lumping and caking from moisture ingress. With adequately constructed cans, a shelf life in excess of 5 years is realistic, but national food safety authorities often adopt a conservative approach by reducing the nominated shelf life.

SMP and powdered whey beverages are available at the retail level in many countries packaged in no. 10 cans (157 × 178 mm with a capacity of 3108 mL) in a reduced- $O_2$  atmosphere to prolong shelf life (up to 54 months). Lloyd et al. (2004) found that in the 10 U.S. brands tested, a wide variation existed in headspace  $O_2$ , can seam quality, sensory quality and vitamin A (with 6 of 10 brands entirely lacking the vitamin). The  $a_w$ s of the brands ranged from 0.14 to 0.28, corresponding to 3%–5% moisture content. The brand that scored highest in overall acceptability had an average headspace  $O_2$  of 7% and poor can seams, calling into question the ability of the package to maintain product quality over an extended storage time.

#### 19.6.4.2 Laminates

In recent years, aluminum foil-plastic laminates have been introduced as a replacement for the tinplate can. The laminates can be formed, filled, gas flushed and sealed on a single machine from reel stock. Gas flushing is achieved by saturating the powder with inert gas. The main advantages associated with laminates are lower material cost and lighter material weight. The disadvantages are that laminates do not have the mechanical strength and durability of rigid containers, and there can be difficulty in obtaining a satisfactory heat seal because of contamination of the heat seal area by powder during filling at high speed.

A typical construction would be an inner layer of LDPE so that the pouch can be sealed and an outer layer of BOPP or PET with alufoil in the middle. Alternatively, with pouches for which a shorter shelf life is acceptable, the alufoil layer may be replaced with a high-barrier plastic layer such as EVOH or PVdC copolymer, and the PET may be coated with SiO<sub>x</sub>.

#### 19.6.4.3 Fiber Cans

Fiber cans or composites manufactured by spiral winding of paperboard strip are available with a wide variety of liners. They can give a similar degree of protection to that obtained from alufoil-LDPE-paper bags and have similar strength to a metal can. They have the added advantages of being lighter than metal cans and not corroding, a problem which can occur with metal cans under high humidity conditions. A typical specification for fiber cans for use with either WMP or SMP is  $0.9 \, \text{mm}$  board and a foil coating of  $5 \, \mu \text{m}$  with a nitrocellulose lacquer to protect the powder from the aluminum foil. An outer decorative label incorporating a fiber seal material gives increased protection against moisture penetration (Cummins, 1982).

# 19.6.5 Packaging Techniques

## 19.6.5.1 Gas Packing

The technique of gas packing is simple and, provided that the package is airtight, the  $\rm O_2$  content can easily be reduced from the 21% present in air to 1% or less immediately after the operation is finished. However, with spray dried powder, this reduction in  $\rm O_2$  content is limited mainly to the atmosphere surrounding the hollow powder particles. Up to 28 days may need to elapse before equilibrium is established between the gases within and around the particles, although most of the change will usually be complete after 7–10 days. During this desorption period, the  $\rm O_2$  content within the can may rise to as much as 5%; this is considerably above the level of 1%–1.5% required to ensure optimal keeping quality. When  $\rm O_2$  levels of the order of 1% are needed, there is no alternative in the usual gas packing technique but to store the powder after the initial gas packing long

enough for desorption to take place and then to gas pack the cans a second time. This is costly and time consuming, requires considerable storage space, increases the handling of the cans and is unsuitable for normal factory routine. Thus, any process that will remove the desorbed  $O_2$  as it is liberated is likely to receive close scrutiny.

Occluded air trapped in vacuoles within powder particles is not removed during gas flushing of cans or laminate packs. Depending on the volume of occluded air in the powder, the equilibrium residual  $O_2$  content of the pack could exceed the  $0.02-0.03\,\mathrm{mL}$  g<sup>-1</sup> normally tolerated. This problem can be alleviated by conditioning the powder under vacuum for 24–48 h prior to gas flushing to remove occluded air.

The U.S. industry standard for shelf life of whole WMP is 6–9 months at <27°C and <65% RH, although flavor changes have been detected after 3 months at ambient storage. Lloyd et al. (2009) evaluated the shelf life of WMP (initial  $a_{\rm w}$  0.17 and moisture 2.46%) packaged in PA-EVOH-LDPE laminate pouches (OTR 1.5 mL m<sup>-2</sup> day<sup>-1</sup> but temperature and RH unspecified) with air or N<sub>2</sub> and stored at 2°C or 23°C for 1 year. Optimum shelf life from a flavor standpoint was about 3 months, and N<sub>2</sub> flushing greatly enhanced the storage stability of WMP by preventing the development of "painty" flavor. Packaging headspace O<sub>2</sub> levels should be as low as possible to prevent lipid oxidation and off-flavors. Storage at 2°C enhanced storage stability of WMP, but to a lesser extent than N<sub>2</sub> flushing.

# 19.6.5.2 Vacuum Packaging

An alternative method of reducing the  $O_2$  content of packaged WMP is by compression of the powder. However, although such a procedure reduces the air content, it results in a significant increase in bulk density and (usually) a decrease in performance as measured by ease of solubility. Vacuum packaging is used to reduce the  $O_2$  content and it achieves this by evacuation of the interstitial air and some compression as a consequence of the vacuumization. The main problem associated with vacuum packaging of milk powder is that of removing air from the package without removing powder fines, which could damage the vacuum pump and contaminate the sealing area of the laminate bag. This problem can be avoided by applying the vacuum at a slow rate so that powder particles are not disturbed.

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