Advances in Dairy Ingredients

1 Dairy Protein Powders

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1.1 INTRODUCTION

The purpose of the dehydration of milk and whey is to stabilize these products for their storage and later use. Dehydration by spray drying is a valuable technique for water evaporation. Milk and whey powders are used mostly in animal feeds. With changes in agricultural policies (such as the implementation of the quota system and the dissolution of the price support system in the European Union), the dairy industry has been forced to look for better uses for the dairy surplus and for the by-products of cheese (whey) produced from milk and buttermilk produced from cream. Studies on the use of protein fractions with nutritional qualities and functionality led us to believe that they could have several applications (Corredig, 2009; Thompson et al., 2009).

In the past 30 years, the dairy industry has developed new technological processes for extracting and purifying proteins (e.g., casein, caseinates, and whey proteins) (Kjaergaard et al., 1987; Maubois, 1991), such as milk protein concentrate (MPC), milk protein isolate (MPI), whey protein concentrate (WPC), whey protein isolate (WPI) (Goudédranche et al., 1980; Madsen and Bjerre, 1981; Maubois et al., 1987; Caron et al., 1997), micellar casein concentrates (MCC) and isolates (MCI) (Fauquant et al., 1988; Pierre et al., 1992; Schuck et al., 1994a,b) whey concentrates, and selectively demineralized whey concentrates (Jeantet et al., 1996), mainly because of the emergence of filtration technology (e.g., microfiltration [MF], ultrafiltration, nanofiltration, and reverse osmosis). This recent emergence of new membrane separation techniques and improvements in chromatographic resins now provide the dairy technologist with several types of techniques for the extraction and purification of almost all of the main milk proteins.

The most frequently used technique for the dehydration of dairy products is spray drying. It became popular in the dairy industry in the 1970s, but at that time, there were few scientific or technical studies on spray drying, and, in particular, none on the effects of spray drying parameters or on the effects of the physicochemical composition and microbiology of the concentrates on powder quality. Manufacturers acquired expertise in milk drying and eventually in whey drying processes through trial and error. Because of the variety and complexity of the mixes to be dried, more rigorous method based on physicochemical and thermodynamic properties have become necessary. Greater understanding

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of the biochemical properties of milk products before drying, water transfer during spray drying, the properties of powders, and influencing factors is now essential for the production of milk powder. The lack of technical and economic information and understanding of scientific methods prevents the manufacturer from optimizing his plant in terms of energy costs and powder quality. In view of the increasing development of filtration processes, the dairy industry requires greater understanding of the effects of spray drying on the quality of dairy protein powders.

A dairy powder is characterized not only by its composition (proteins, carbohydrates, fats, minerals, and water), but also by its microbiological and physical properties (bulk and particle density, instant characteristics, flowability, floodability, hygroscopicity, degree of caking, whey protein nitrogen index (WPNI), thermostability, insolubility index (ISI), dispersibility index, wettability index, sinkability index, "free fat," occluded air, interstitial air, and particle size), which form the basic elements of quality specifications. There are well-defined test methods for the determination of powder characteristics according to international standards (Pisecky, 1986, 1990, 1997; American Dairy Products Institute, 1990; Master, 2002). These characteristics depend on drying parameters (e.g., type of tower spray drier, nozzles/wheels, pressure, agglomeration and thermodynamic conditions of the air, such as temperature, relative humidity, and velocity), the composition and physicochemical characteristics of the concentrate before spraying (e.g., viscosity, thermo-sensitivity and availability of water), and storage conditions. Several scientific papers have been published on the effects of technological parameters on these properties (Baldwin et al., 1980; Pisecky, 1980, 1981, 1986; De Vilder, 1986; Tuohy, 1989; Jeantet et al., 2008a; Master, 2002) (see Figure 1.1). Water content, water dynamics and water availability are among the most important properties for all these powder properties and powder characteristics.

The nutritional quality of dairy powders depends on the intensity of the thermal processing during the technological process. Thermal processing induces physicochemical changes that tend to decrease the availability of nutrients (e.g., loss of vitamins, reduction of available lysine content, and whey protein denaturation) or to produce nutritional compounds, such as lactulose (Straatsma et al., 1999a,b).

The aim of this chapter is to provide information on the extraction of milk proteins; the principles of spray drying, including equipment and energy consumption; the drying of high protein products, including the relationships between process and product; and the physical, functional, and biochemical properties of the powders. Following an in-depth introduction on dairy protein products, this chapter covers four major areas: the extraction of milk proteins, the principles of spray drying (equipment and energy consumption), the drying of dairy protein products and the properties of these powders.

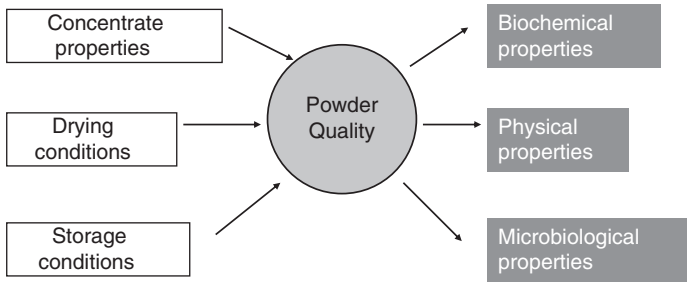

Figure 1.1 Properties and qualities of powders.

1.2 EXTRACTION OF MILK PROTEINS

1.2.1 Milk proteins

It would be impossible to develop the extraction of milk proteins without prior thorough knowledge of their biochemical and physicochemical properties. A brief description of the milk protein system is therefore important for understanding the principles used before discussing the recent developments in extraction procedures (Maubois and Ollivier, 1997).

Bovine milk contains several that are classically divided into two major groups, that is, caseins, proteins which are insoluble at pH 4.6 and 20°C, and whey proteins which remain in solution at pH 4.6. The protein content of normal milk is expressed as $N \times 6.38$. Milk contains 30–35 g protein/L. About 78% of these proteins are caseins, which consist of four principal components, αs_1, αs_2, β, and κ, in approximate ratios of 40 : 10 : 35 : 12. In milk, these caseins are organized in the form of micelles, which are large spherical complexes (diameters varying between 50 and 600 nm, average 120 nm) containing 92% proteins and 8% inorganic salts, principally calcium phosphate (Rollema, 1992; Swaisgood, 1992). The structure of the micelles has not yet been fully established, and there is still controversy between the supporters of the submicellar model and those of the coat-core model (Schmidt, 1982; Holt, 1992; Farrell et al., 2006; Horne, 2006). The casein micelles dissociate on removing colloidal calcium phosphate either by the addition of Ca chelating agents (e.g., phosphate, citrate, and EDTA) or by acidification. The casein micelles are partly responsible for the white color of milk. Their stability results from their zeta potential (approximately −20 mV) and from steric hindrance caused by the protruding ("hairy") C-terminal segments of glycosylated κ-casein, which prevent the close approach of micelles. Removal of these protruding segments by chymosin, the main enzyme present in the neonate calf stomach, results in coagulation of the damaged casein micelles. The integrity of casein micelles is also affected by cooling. At temperatures lower than 4°C, β-casein and Ca phosphate are released into the serum phase of milk.

The whey protein fraction contains several proteins. The main components in bovine milk are β-lactoglobulin (β-Lg), α-lactalbumin (α-La), bovine serum albumin (BSA), and immunoglobulin (Ig), representing approximately 2.7, 1.2, 0.25, and 0.65 g/L, respectively (Alais, 1984). There are many other minor proteins, including lactoferrin (Lf), enzymes (lipoprotein lipase, acid and alkaline phosphatases, lysozyme, xanthine oxidase, lactoperoxidase, catalase, superoxide dismutase, α-amylase etc), growth factors, and hormones (Alais, 1984). The whey protein fraction of human milk is very different from that of bovine milk in that it contains no β-Lg and is very rich in α-La, Lf, lysozyme, and stimulatory factors (bifidus growth factor, epidermal growth factor, bombesin, insulin-like growth factors, etc.) (Fox and Flynn, 1992; Maubois and Ollivier, 1997).

1.2.2 Separation of proteins

Most of the dairy proteins, used as either nutritional or functional ingredients, are marketed in a dehydrated form (see Figure 1.2). The application of different processing steps allows the production of a wide range of different dried and stable intermediate dairy products. Many new uses for these constituents have emerged with the manufacture of formula products, substitutes, and adapted raw materials.

Figure 1.2 summarizes the procedures available for the separation of milk proteins. Before discussing the details of the processes shown in this diagram, it is necessary to set

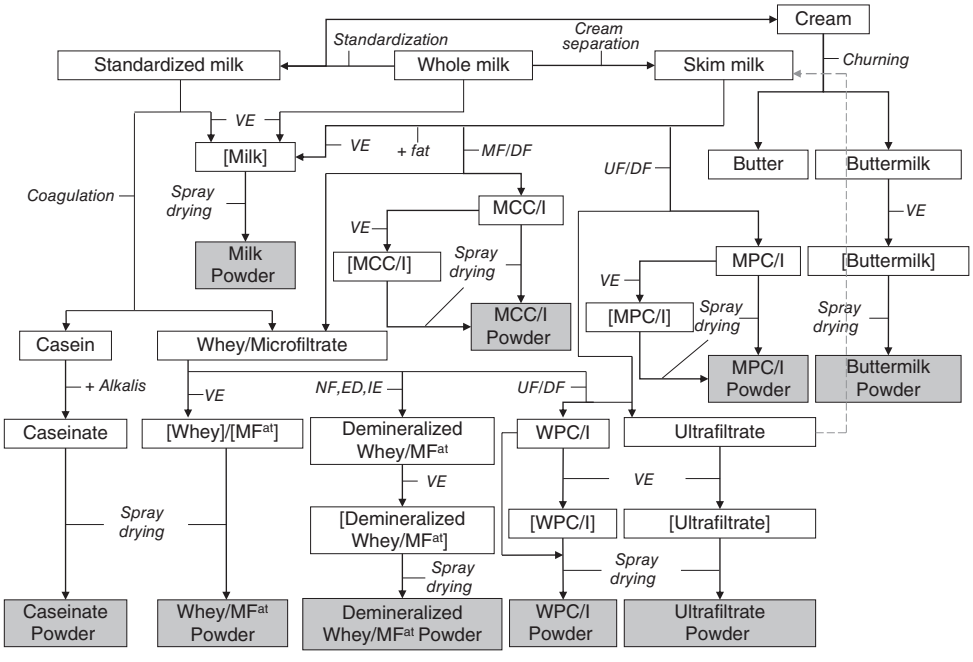

Figure 1.2 Fractionation of milk. [], Concentrate by vacuum evaporation; MCC/I, micellar casein concentrate/isolate; WPC/I, whey protein concentrate/isolate; MPC/I, milk protein concentrate/isolate; ED, electrodialysis; IE, ion exchange; VE, vacuum evaporation; MF, microfiltration; MFat, microfiltrate; NF, nanofiltration; UF, ultrafiltration; DF, diafiltration.

out some general guidelines to food technologists who wish to develop strategies for extraction of a dairy protein or a group of dairy proteins.

At each stage of the extraction process, the preservation of desirable qualities (i.e., functional, biological, and nutritional) must be taken into account when choosing technologies and the physicochemical parameters to be used. Particular attention must be paid to successive heat treatments, the effects of which are cumulative. Like most liquid foods, milk and its derivatives are very favorable media for spoilage microorganisms. Consequently, pretreatments and temperature–time parameters and residence time temperature (Jeantet et al., 2008b) must be chosen in order to control microbial growth.

Each separation and fractionation step generates at least one coproduct; for environmental reasons, and because of their potential value, the coproducts must be considered not only as by-products, but also as value-added components of milk. Thus, all extraction procedures must be envisaged in an integrated technological concept that utilizes all of the products and effluents generated.

1.2.3 Pretreatment of milk

Milk collected by the dairy industry contains lipids (≈40 g/L) organized in fat globules. Most extraction procedures for milk proteins use skim milk as the starting material. Whole milk is therefore centrifuged in a separator comprising conical discs and running at 5000 rpm at around 50°C. The separated cream generally represents 10% of the volume of the entering whole milk. Fat separation is never perfect, and the skim milk thus obtained still

contains around 0.5 g/L fat, which can considerably influence the effectiveness of downstream processes and the resulting protein products.

In developed countries, milk is normally contaminated by common mesophilic and psychrotrophic microflora and rarely by pathogenic microorganisms. To minimize possible health hazards and to control bacterial growth during milk processing, moderate heat treatments (at 63°C for 30 minutes or pasteurization at 72°C for 15 seconds) are applied to skim milk before further processing. The consequences of this heat treatment are numerous and significant: it decreases the pH, shifts the delicate protein–calcium phosphate equilibrium, causes changes in the micellar structure of casein, which in turn affects its hydration and zeta potential (Fox, 1982), and initiates the Maillard reaction, which permanently modifies the functional and nutritional properties of the whey proteins (Maubois et al., 1995). An interesting recent development in the removal of microorganisms from skim milk is the use of membrane MF technology. Following the recommendations of Sandblom (1974) and Meersohn (1989), skim milk is microfiltered through a ceramic membrane (average pore size of 1.4 μm) at a temperature between 20 and 55°C. The average decimal reduction observed between the inlet milk and microfiltered milk is 2.6, corresponding to a reduction of the contaminating microorganisms of 99.5%.

1.2.3.1 *Isolation of whole proteins*

There are two major methods to produce dairy proteins: coprecipitation techniques and ultrafiltration technology. Coprecipitation involves the application of high heat during the precipitation process to cross-link the two major proteins (caseins and whey proteins), but this also results in denaturation of whey proteins. Calcium chloride or acid is also injected through a spray that is countercurrent to the direction of milk flow to provide full mixing. The mixture is transformed into curd in a holding tube (20–25 seconds). The curd is separated from the whey, and the coprecipitate is washed and pressed. At optimal process conditions, it is possible to recover 95–97% of the milk proteins.

There are three basic varieties of coprecipitates, each having different amounts of calcium: low calcium coprecipitate (0.1–0.5% Ca), medium calcium coprecipitate (1–1.5% Ca) and high calcium coprecipitate (2.5–3.5% Ca). These coprecipitates can then be dried and used as ingredients. The nutritional quality of coprecipitates is better than that of caseinates, but functionality of coprecipitates is limited to applications where denatured whey proteins are needed, such as in bakery products. To address this limitation, New Zealand Milk Products Inc. patented a process for the production of total MPIs in 1983 that has since been improved (Mistry, 2002). The process involves the coprecipitation of caseins and whey proteins using a series of pH adjustments to prevent the heat denaturation of whey proteins. A modification of this process that involves ultrafiltration and diafiltration has also been patented. An ultrafiltration process was developed for the production of high milk protein powders (MPC/MPI) for applications in cheese and yogurt making (Fox, 1982; Mistry, 2002). This process was developed using ultrafiltration and diafiltration of skim milk without pH adjustment to concentrate the proteins, followed by spray drying.

1.2.3.2 *Isolation of whole casein*

There are three principal ways available for the production of whole casein on an industrial scale, that is, isoelectric precipitation, rennet coagulation, and MF. Acid casein is produced

by the use of hydrochloric or sulfuric acid or by lactic acid produced by bacteria. Sufficient acid is added to reduce the pH of the milk to 4.6 and the mix is diluted with four parts water with thorough mixing. After a short holding period in a vat, the whey is drained off. The curd is washed twice with cold water, pressed and milled. Acid casein may also be produced by inoculating skim milk with acid-producing bacteria and incubating it at 20–30°C until the acidity reaches 0.64%. The curd is stirred and heated to 50–65°C. The whey is then drained and the curd washed twice with cold water and pressed for 10–15 hours. It is then milled and ready for drying. Rennet casein is made by adding sufficient rennet and calcium chloride to skim milk to cause it to clot in 20–30 minutes. Stirring is commenced 2–5 minutes after coagulation has started. The temperature is raised to 55–70°C, and the curd is cooked for 30 minutes. The whey is then drained off. Acid or rennet whey may be dried in a cabinet, tunnel, or spray dryer to a moisture content of 4%. The dried casein is milled and screened to an appropriate particle size. Casein is used in the manufacture of paint and plastics and for paper coating. It is also used in coffee/tea whiteners, whipping powders, and imitation milks (Maubois and Ollivier, 1997).

The most promising technology for the selective separation of casein micelles is undoubtedly membrane MF. When whole or skim milk is circulated through a MF membrane with a pore size diameter of 0.1–0.2 μm, a microfiltrate is obtained with a composition close to that of sweet whey. Moreover, it is crystal clear and can be sterile if the downstream equipment prevents recontamination. The retentate is an enriched solution of native and micellar casein, that is, MCC (Fauquant et al., 1988). Diafiltration against water allows its purification into micellar casein isolate (MCI), and it is easily concentrated again by MF and then spray-dried (Pierre et al., 1992; Schuck et al., 1994b). MCI has excellent rennet-coagulating abilities. The coagulation time of a 3% MCI solution is reduced by 53% compared with that of raw milk, and gel firmness at 30 minutes is increased by more than 50% (Pierre et al., 1992). MCI and its coproduct, the WPI obtained by submitting the microfiltrate to a subsequent ultrafiltration step, are excellent starting substrates for further fractionation and isolation of milk proteins.

1.2.3.3 *Fractionation of whole casein*

There is considerable interest in developing technologies for the fractionation of whole casein into individual components (α_{s1}, α_{s2}, β, and κ) on an industrial scale. These fractions can be potentially used in bovine milk-based infant formulas and for the preparation of biologically active peptides and specific additives. Most published studies have focused on isolation of β-casein, the main component of human casein, which contains numerous peptide sequences with physiological activities, such as the well-known β-casomorphin. The growing commercial interest in the production of functional peptide fragments of α_s- and κ-caseins will probably lead to future developments in the fractionation of these proteins, either from the coproduct resulting from fractionation of β-casein or from the native casein micelles dissociated by the combined action of pH, NaCl, and sodium citrate, as proposed by Pouliot et al. (1994).

1.2.3.4 *Whey protein separation*

There are several industrial methods suitable for the production of various WPCs and WPIs. The interest in whey processing is a result of two factors. One is a worldwide shortage of high-quality animal proteins that whey proteins may alleviate, and the other is the problem

with the disposal of whey. The high biological oxygen demand of whey makes this cheese by-product a pollutant so that it is more desirable to process it than to dump it.

In addition to traditional methods, such as evaporation and drying, modern methods used in industrial whey processing include ultrafiltration, MF, reverse osmosis (hyperfiltration) and demineralization (electrodialysis and ion exchange). The most commonly used membrane method in dairying is ultrafiltration. Its industrial application was aided by the introduction of cross-flow instead of dead-end filtration and the invention of asymmetric membranes (Carić, 1993). During the ultrafiltration of whey, low molecular weight compounds, such as lactose, minerals, nonprotein nitrogen, and vitamins, are separated in the permeate, whereas proteins are concentrated in the retentate. This permits a WPC to be obtained with 20–60% protein in total solids and low quantities of lactose and mineral matter. Permeate, a by-product of this processing, is used for producing lactose, alcohol, single-cell protein, yeast, galactose, glucose, cattle feed, and various pharmaceuticals.

Further increases in protein content (up to 98%) may be achieved by adding water to the feed. This procedure is called diafiltration. The best moment to start diafiltration is when the optimal total solids content has been reached and at a point where the ultrafiltration flux is still relatively high.

Sweet whey is first subjected to clarification (removal of casein fine particles, fat separation, and pasteurization). After pasteurization, the whey is cooled to 60–65°C and held at this temperature for 30–60 minutes before cooling to 50°C for ultrafiltration. This heat-and-hold treatment has the function of stabilizing the calcium phosphate complex, thus reducing the fouling of the membranes during ultrafiltration. Further reduction of other minerals in WPC is achieved by adjusting the pH of the whey to pH 5.7–6.0 with HCl. The solubility of calcium is increased with decreasing pH, thus resulting in a greater proportion of calcium in the permeate. After ultrafiltration, the retentate is pasteurized, evaporated (or not, depending on the viscosity and protein content), and dried.

1.2.3.5 *Fractionation of whey proteins*

The main proteins found in WPCs and isolates are: β-Lg, α-La, glycomacropeptides, BSA, Igs, Lf, and lactoperoxidase. Each of these proteins or groups of proteins has been proven or believed to have unique functional, nutritional, or nutraceutical properties. Some putative nutraceutical activities include digestive function (β-Lg and glycomacropeptide), anticarcinogenic properties (α-La), antimicrobial activity (Lf and lactoperoxidase) and passive immunity (Igs). There are also nutritional considerations. It is known that α-La binds minerals, specifically calcium, magnesium, zinc, and cobalt. By being bound to a protein, these minerals are more readily delivered for absorption in the human body. The lack of β-Lg in human milk suggests that bovine whey protein products rich in α-La and low in β-Lg would be more appropriate for infant formulae.

There is a considerable interest in developing technologies for the fractionation of whey proteins into α-La and β-Lg on an industrial scale (Maubois and Ollivier, 1997). A number of methods have been developed (Slack et al., 1986; Maubois et al., 1987; Pearce, 1987) with commercial scale potential to fractionate the major whey protein components, β-Lg and α-La, and to produce WPCs enriched in these fractions. These methods depend on either mild heat treatments of a whey concentrate or a clarified whey under controlled pH and ionic conditions, or on demineralization of whey concentrate under controlled pH conditions. These treatments are used to achieve selective reversible precipitation of α-La- or β-Lg-enriched fractions and the separation of the precipitate from β-Lg- or

α-La-enriched solutions. The precipitate is resolubilized by the addition of water and pH adjustment and then dried, while the soluble protein is further concentrated by ultrafiltration/diafiltration prior to drying.

1.3 DRYING PRINCIPLES

Drying is defined as the removal of a liquid, usually water, from a product by evaporation, leaving the solids in an essentially dry state. A number of different drying processes are in use in the dairy, food, chemical, and pharmaceutical industries, such as:

- spray drying;
- fluid bed drying;
- roller drying;
- freeze-drying;
- microwave drying; and
- superheated steam drying.

Due to considerations of drying economics and final product quality, the only processes of significance in milk protein powder manufacture are spray drying, fluid bed drying (the two most often in combination), and roller drying, although the latter is in only limited use nowadays. Only these three drying processes will be discussed here.

1.3.1 Roller drying

In roller drying, a preconcentrated product is applied as a thin film on the outer surface of an internally heated rotating metal drum (Refstrup, 2003). A vapor hood and exhaust system are placed above the drum. The milk film is scraped off the drum surface as a sheet of dry product by stationary knives located opposite the point of milk concentrate application. The product sheet or flakes fall into an auger trough, which partly disintegrates it and conveys it to a pneumatic cooling and conveying system, often with integrated milling, and thence to storage and packaging.

1.3.1.1 *Types of drum driers*

Several types of drum dryer exist. They can be characterized by the combination of the number of drums (single or double) or the method of product application (sump between two closely positioned drums, spray with nozzles, or immersed applicator roll system).

The main process parameters affecting the plant capacity and product properties are:

- Drum surface temperature: Saturated steam (at up to 0.5-MPa pressure, corresponding to about 150°C) is used as heating medium.
- Feed temperature: This may vary from about 10–80°C depending on the type of product. The higher the feed temperature, the greater is the plant capacity.
- Feed solids content: A total solids content of up to 45% is usually used. The higher the solids content, the faster is the product drying rate.
- Drum rotation speed: The time of exposure to the hot drum surface, and hence the final moisture content, is controlled by the rate of rotation of the drum. This is because, for

a given drum temperature, feed solids content, and viscosity, the drum rate of rotation also affects the thickness of product film.
- Distance between drums and/or applicator rollers: The gap between drums, which is usually <100 μm, also controls the thickness of product film.
- Area of heat transfer surface: The plant capacity is proportional to the effective area of heat transfer.

Generally, drum drying has a number of serious disadvantages compared with spray drying. These include:

- Severe heat damage and protein denaturation during the slow drying and relatively long residence time on the hot drum (3–6 seconds), resulting in poor solubility and cooked or burned flavor.
- Relatively low evaporative capacity, with the greatest capacity of a single drum drying unit being about 1000 kg/h water evaporation.
- Inflexibility in relation to control of powder properties as there is no possibility of producing agglomerated or instant products with drum drying.

Despite these disadvantages, drum dryers are still in use in niche production where the special functional properties of drum-dried powders are desirable. For instance, the high "free fat" content of drum-dried whole milk is advantageous in the chocolate industry, and the high water-binding capacity of drum-dried skim milk is desirable in the meat-processing industry. Drum dryers are also used to dry off highly viscous cereal- or starch-based product blends that cannot easily be atomized (Refstrup, 2003).

1.3.2 Spray drying and fluid bed drying/cooling

The basic principle of spray drying is the exposure of a fine dispersion of droplets, created by means of atomization of preconcentrated milk products on a hot air stream. Spray drying is an industrial process for the dehydration of a liquid by transforming the liquid into a spray of small droplets and exposing these droplets to a flow of hot air (Pisecky, 1997). The very large surface area of the spray droplets causes evaporation of the water to take place very quickly, converting the droplets into dry powder particles. The small droplet size created, and hence large total surface area, results in very rapid evaporation of water at a relatively low temperature, whereby heat damage to the product is minimized (Refstrup, 2003).

In fact, when a wet droplet is exposed to hot dry gas, variations in the temperature and the partial pressure of water vapor are spontaneously established between the droplet and the air. This results in heat transfer from the air to the droplet, which occurs under the temperature variation between the air and the droplet. Water transfer occurs in the opposite direction, and this is explained by variation in the partial pressure of water vapor between the air and the droplet surface.

Air is thus used both for fluid heating and as a carrier gas for the removal of water. The air enters the spray drier hot and dry and leaves wet and cool. Spray drying is a phenomenon of surface water evaporation maintained by the movement of capillary water from the interior to the surface of the droplet. As long as the average moisture is sufficient to feed the surface regularly, the evaporation rate is constant. If not, it decreases.

The drying kinetics is related to three factors:

- The evaporation surface created by the diameter of the particles. Spraying increases the exchange surface. For example, 1 L of liquid sprayed in particles of 100-µm diameter develops a surface area of 60 m^2, whereas the surface area for one sphere of the same volume is only approximately 5 dm^2.
- The difference in the partial pressure of water vapor between the particle and the drying air. A decrease in the absolute humidity of the air and/or an increase in the air temperature tend to increase the difference in the partial pressure of water vapor between the particle and the drying air.
- The rate of water migration from the center of the particle towards its surface. This parameter is essential for the quality of dairy powders. Indeed, it is important that there is always water on the surface of the product so that the powder surface remains at the wet bulb temperature for as long as possible. The rate of water migration depends on the water diffusion coefficient, which varies according to the biochemical composition, water content, and droplet temperature. This is why the calculation of this coefficient is complex, and the mathematical models suggested are not easily exploitable by the dairy industry.

1.3.2.1 *Components of spray drying installations*

To define the components of a spray drying installation, according to Masters (1991), Pisecky (1997) and Westergaard (2003), the main components of the spray drier shown in Figure 1.3 are as follows.

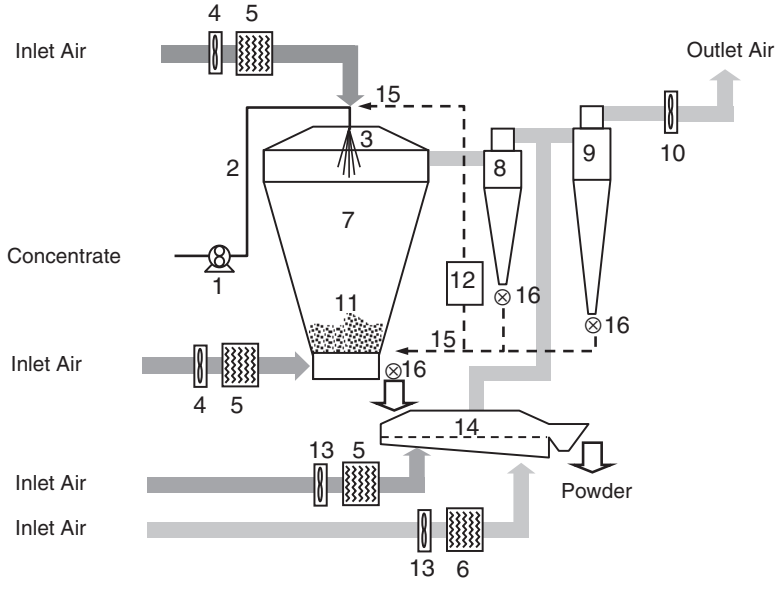

Figure 1.3 Multiple effect spray dryer. 1, Feed pump; 2, feed flow; 3, sprayer / air disperser; 4, inlet air fan; 5, air heater; 6, air cooler; 7, drying chamber; 8, primary cyclone; 9, secondary cyclone; 10, outlet air fan; 11, integrated fluid bed; 12, pressure conveyer system; 13, vibrofluidizer air fan; 14, vibrofluidizer; 15, reincorporating fines; 16, rotary valve.

Drying chamber (Figure 1.3)
Various designs of drying chambers are available on the market. The chamber can be horizontal (box drier), although in the dairy industry, the chamber design is generally vertical, with a conical or flat base. The most common is the cylindrical chamber with a cone of 40–60°, so that the powder can leave the chamber by gravity. This chamber may also have a flat bottom, in which case a scraper or suction device is needed to remove the powder from the chamber.

Air filtration
Until a few years ago, no special requirements were stipulated for the filtration of the process air for the spray drying process. Today, however, there are very strict requirements made by local authorities in order to ensure a cleaner operation. Common requirements for the different standards are:

- The air should be prefiltered and supplied by a separate fan to the fan/filter/heater room, and should be under pressure to avoid the entry of unfiltered air.
- The degree of filtration and filter position must depend on the final temperature of the process air as follows: for air to be heated above 120°C, only coarse filtration up to 90% is needed, and the filter should be placed on the pressure side of the fan; for air to be heated below 120°C or not heated at ail, filtration must be 95% or above, and the filter must be placed after the heater/cooler. Some countries have even stricter requirements, demanding filtration of up to 99.995% (Westergaard, 2003).

Air heating system
The drying air can be heated in different ways: either indirectly by steam, oil, gas, or hot oil, or directly by gas or electricity. A steam heater is a simple radiator. The temperature to be obtained depends on the steam pressure available. The air heater consists of rows of finned tubes housed in an insulated metal case. In indirect oil and gas heaters, drying air and combustion gases have separate flow passages. The combustion gases pass through galvanized tubes that act as a heat transfer surface for the drying air. The combustion chamber is made of heat-resistant steel. Heaters of this type have an efficiency of about 85% in the range of 175–250°C. Direct gas heaters are used only when the combustion gas can be allowed to come into contact with the product. They are, therefore, uncommon in the dairy industry. The direct gas heater is cheap, it is highly efficient, and the temperature obtainable can be as high as 2000°C. When a plant is designed with an air heater with direct combustion, it is necessary to calculate the amount of vapor resulting from the combustion (44 mg/kg dry air/°C), as this will increase the humidity of the drying air. The outlet temperature must therefore be increased in order to compensate for this increase in humidity and to maintain the relative humidity. Electric air heaters are common in laboratory and pilot plant spray dryers. The heater has low investment costs, but is expensive to operate and therefore is not used in industrial-sized plants (Westergaard, 2003).

Air distribution
The system of air distribution is one of the most vital points in a spray dryer. There are various systems, depending on the plant design and the type of product to be produced. The most common system is where the air disperser is situated on top of the dryer ceiling

and the atomizing device is placed in the middle of the air disperser, thus ensuring optimal mixing of the air and the atomized droplets. The air flow chamber can be in cocurrent, counter-current, or mixed mode.

Atomization

An atomizing device with a feed supply system, such as feed tank, feed pump, water tank, concentrate heater, and atomizing device, is required. The aim of atomizing the concentrate is to provide a very large surface area from which the evaporation of water can take place. The smaller the droplets, the greater the surface area, and easier evaporation and better thermal efficiency of the dryer are obtained. The ideal situation from a drying point of view is a spray of droplets of the same size, where the drying time for all particles would be the same to obtain equal moisture content. As mentioned previously, air distribution and atomization are the key factors in successful utilization of the spray dryer. Atomization is directly responsible for many distinctive advantages offered by spray drying: first, the very short drying time of the particles; second, very short particle retention time in the hot atmosphere and low particle temperature (wet bulb temperature), and finally, transformation of the liquid feed into a powder with long storage stability ready for packing and transport.

In summary, the prime functions of atomization are:

- to produce a high surface to mass ratio, resulting in a high evaporation rate; and
- to produce particles of the desired shape, size, and density.

There are three types of atomizing devices: rotary atomizer (wheel or disc), nozzle atomizer (pressure, pneumatic, or sonic), and combined atomizer (rotary and pneumatic) (see Figure 1.3, no. 3).

The basic function of pressure nozzles is to convert the pressure energy supplied by the high-pressure pump into kinetic energy in the form of a thin film, the stability of which is determined by the properties of the liquid, such as viscosity, surface tension, density, and quantity per unit of time, and by the medium into which the liquid is sprayed.

The energy available for atomization in two-fluid atomizers is independent of liquid flow and pressure. The necessary energy (kinetic) is supplied by compressed air. Two-fluid atomization is the only successful nozzle method for producing very small particles, especially from highly viscous liquids. It is not normally used in the drying of milk products.

In rotary atomizers, the liquid is accelerated continuously to the wheel edge by centrifugal forces produced by the rotation of the wheel. The liquid is distributed centrally and then extends over the wheel surface in a thin sheet and discharged at high speed at the periphery of the wheel. The degree of atomization depends on peripheral speed, the properties of the liquid, and feed rate. According to Westergaard (2003), to select an optimal atomizer wheel, the liquid feed rate, peripheral speed, and viscosity of the liquid should be taken into consideration.

Powder recovery system

Separation of the dried product can be achieved by a primary discharge from the drying chamber followed by a secondary discharge from a particulate collector (using a cyclone,

bag filter, or electrostatic precipitation), followed by total discharge from the particulate collector, and finishing with final exhaust air cleaning in a wet scrubber and dry filter.

- *Cyclone.* The operating principle of the cyclone is based on a vortex motion in which the centrifugal force acts on each particle and causes the particle to move away from the cyclone axis towards the inner cyclone wall (Figure 2.3, nos. 8 and 9).
- *Wet scrubber.* The operating principle is based on the venturi scrubber principle. The droplet separator is designed according to cyclone principles, with a modified outlet, resulting in a minimum liquid level, thereby minimizing bacterial growth, and a design ensuring deaeration, thus avoiding the buildup of foam.
- *Bag filter.* This consists of numerous bags, installed so that each receives approximately equal quantities of air. The collection of bags is termed the baghouse. The simple principle of passing powder-laden air through a close woven fabric is one of the oldest methods of air cleaning, but it remains an effective means of powder separation, being able to remove particles below 10 μm. With the correct weave, 1-μm particle sizes can be collected. The performance of bag filters depends upon (1) the type of product handled, (2) the powder loading in air, (3) bag fabric, and (4) bag cleaning procedure. Modern bag filter designs have proven operational reliability, but do require regular maintenance and inspection to ensure no bag leakage. The slightest leak will quickly diminish the very high collection efficiency levels this equipment is capable of achieving (Master, 2002).

Authorities normally conclude that powder loss of 250 mg/m^3 (obtained classically by using only cyclones) is too high, and set a standard of 50 mg/m^3 (obtained by using cyclones plus bag filters or cyclones plus wet scrubber that requires a final cleaning of the air). Authorities demand reduced powder emission while powder producers demand lower energy consumption and reduced space requirements. For these reasons, a new powder recovery system has been developed, the cleanable-in-place (CIP-able) bag filter, which replaces the cyclones/bag filter (Westergaard, 2003).

1.3.2.2 *Types of drying installations*

According to Sougnez (1983), Masters (1991), and Pisecky (1997), the simplest types of installation are single-stage systems with a very short residence time (20–60 seconds). Thus, there is no real balance between the relative humidity of the air and the moisture content of the powder. The outlet temperature of the air must therefore be higher, and the thermal efficiency of the single-stage spray drier is then reduced. This type of drying chamber was the standard equipment for drying milk in the 1960s. Space requirements were small and building costs were low. Generally, installations without any posttreatment system are suitable only for nonagglomerated powders not requiring cooling. If necessary, a pneumatic conveying system could be added to cool the powder while transporting the chamber fraction and the cyclone fraction to a single discharge point.

The two-stage drying system consists of limiting the spray drying process to a process with a longer residence time (several minutes) to provide a better thermodynamic balance. This involves a considerable reduction in the outlet air temperature, and also an increase in the inlet air temperature. A second final drying stage is necessary to optimize the moisture content by using an integrated fluid bed (static) or an external fluid bed (vibrating), the air temperatures of which are 15–25°C lower than with a single-stage system to improve

and/or preserve the quality of the dairy powder (Figure 1.3, nos. 11 and 14). Consequently, the surrounding air temperature at the critical drying stage, and the particle temperature are also correspondingly lower, thus contributing to further improvement in economics. The integrated fluid bed can be either circular (e.g., multistage drier [MSD™] chamber, GEA-Process Engineering, Soeborg, Denmark) or annular (e.g., compact drier [CD] chamber). Two-stage drying has its limitations, but it can be applied to products such as skim milk, whole milk, precrystallized whey, caseinates, whey proteins, and derivatives. The moisture content of the powder leaving the first stage is limited by the thermoplasticity of the wet powder, that is, by its stickiness in relation to the water activity and the glass transition temperature (Roos, 2002). The moisture content leaving the first stage must be close to 7–8, 9–10, and 2–3% for skim/whole milk, caseinate/whey protein, and precrystallized whey powders, respectively. The two-stage drying techniques can be applied to the production of both nonagglomerated and agglomerated powders. However, this technique is very suitable for the production of agglomerated powders by separating the nonagglomerated particles from the agglomerates (i.e., collecting the cyclone fractions and reintroducing these fine fractions [called fines] into the wet zone around the atomizer of the chamber) (Figure 1.3, no. 15).

The three-stage drying systems, with an internal fluid bed as a second stage in combination with an external vibrating fluid bed as a third-stage drier, first appeared at the beginning of the 1980s, and were called compact drier instantization (CDI) or MSD. Today, they dominate the dairy powder industry (see Figure 1.3). Three-stage systems combine all the advantages of extended two-stage drying, using spray drying as the primary stage, fluid bed drying of a static fluid as the second drying stage and drying on an external vibrating fluid bed as the third drying stage. The final drying stage terminates with cooling to under the glass transition temperature. Evaporation performed at each stage can be optimized to achieve both gentle drying conditions and good thermal economy.

Compact drying is suitable for producing both nonagglomerated and agglomerated powders of practically any kind of dried dairy product. It can also cope successfully with whey powders, fat-filled milk, and whey products, as well as caseinates, both nonagglomerated and agglomerated. It has a fat content limit of about 50% fat in total solids. Powder quality and appearance are comparable with those of products from two-stage drying systems, but they have considerably better flowability, and the process is more economical. In comparison with CD, MSD can process an even wider range of products and can handle an even higher fat content. The main characteristic of a MSD powder is due to very good agglomeration and mechanical stability, low particle size fractions (below 125 μm), and very good flowability. Optimization of the process allows considerable improvement in drying efficiency and the quality of the product obtained is generally better. The various advantages are:

- Improved thermal efficiency—significant reduction in outlet air temperature, permitting an increase in inlet air temperature;
- Reduced material obstruction—the capacity in one volume is two or three times greater than for a traditional unit;
- Considerable reduction in powder emission to the atmosphere—reduction of the drying air flow and increase in powder moisture content decrease the loss of fine particles in the outlet air;
- Improved powder quality in relation to agglomeration level, solubility, dispersibility, wettability, particle size, density, and so on.

There are other examples of drying equipment such as the tall form drier, the Filtermat™ drier (GEA-Process Engineering), the Paraflash™ drier (SPX, Soeborg, Denmark), the Tixotherm™ drier (GEA-Process Engineering), and the Integrated Filter Dryer™ (IFD™) (GEA-Process Engineering). All these towers have characteristics related to the specific properties (e.g., high fat content) and type of the product (e.g., starch, maltodextrin, egg, and hygroscopic products) being dried.

In terms of energy balance, Westergaard (2004) showed that energy consumption varies according to the drying processes. Energy consumption is 1595, 1350, 1280, 1038, and 960 Kcal for a one-stage spray dryer, a two-stage spray dryer, a two stage-spray dryer with high inlet air temperature, CDI, and MSD, respectively, to produce 1 kg of powder from skim milk concentrate at 45% of total solids. This is explained by the increase in the number of drying stages simultaneously increasing the residence time, allowing increase in the inlet air temperature, concentrate flow rate, and finally energy yield while preserving/improving powder quality (Bimbenet et al., 2002).

1.4 DRYING OF DAIRY PROTEINS

1.4.1 Heat treatment

The native properties of milk components are mainly unaffected by moderate drying conditions. Depending on the preheating conditions, drier design, and temperature operation, the properties of the spray-dried powder may vary significantly. An evaporating milk droplet in a spray drier in cocurrent air flow initially does not appreciably exceed the unit bulb temperature and can be held effectively at temperatures below 60°C. As the falling temperature period is approached in the course of further evaporation, the temperature rises to a final value determined by the final temperature of the drying gas and the residence time in the drier. Under properly controlled spray drying conditions, the changes in milk protein structure and solubility are minor. Spray drying does not lead to significant denaturation of the whey protein, and the levels of denatured whey protein in dairy powders are more or less equal to those of condensed milk and heated milk fed into the dryer.

The WPNI has traditionally been used to classify milk powders. The WPNI expresses the amount of undenatured whey protein (milligrams of whey protein nitrogen per gram of powder) (Pisecky, 1997). It represents the sum of heat treatments to which the milk has been subjected prior to evaporation and spray drying. The heat treatment of a concentrate, and subsequently of a powder, has only a negligible effect on the WPNI. The main operation to adjust the required value is the pasteurization process, that is, time/temperature combination. However, there are many other factors that influence the WPNI, including the total amount of whey protein and the overall composition of the milk processed that can be influenced by animal breed and seasonal variations. The individual design of the processing equipment, that is, the pasteurizer and holding tubes, also has a significant influence. It is therefore difficult to predict the conditions for achieving the required WPNI on a general basis. Obviously, the primary purpose of heat pretreatment is to ensure the microbiological quality of dairy products. In milk powder production, the influence of the heat treatment on the denaturation of whey proteins to achieve the desired properties of the final products is just as important. Skim milk powder (SMP) for cheese manufacture should have as much undenatured protein as possible, that is, it should be *low heat* powder (WPNI > 6), whereas for bakeries, *high heat* powder with high denaturation is required

(WPNI < 1.5). For ice cream, chocolate, and confectionery, *medium heat* powder is required. According to Schuck et al. (1994a), the use of MF (pore diameter, 1.4 μm), coupled with low heat treatment during vacuum evaporation, allows the production of a "*very low heat*" SMP with a WPNI close to 9 mg of whey protein nitrogen/g of powder, a bacterial count <1000 coliform forming units (CFU)/g powder, a solubility index >99.5%, a dispersibility index >98.5%, and a wettability index <15 seconds. Such a powder has the same renneting time after water rehydration as the original raw milk, and can be used as a reference powder for either industrial or scientific purposes.

The stability of dairy protein powders during storage is critically affected by the moisture content and the storage temperature. More precisely, such stability is governed by the water activity (a_w) and the glass transition (T_g) temperature. The a_w should be close to 0.2 at 25°C for optimal preservation, with an ideal moisture content determined by using the sorption isotherm of some dairy powders. For example, the corresponding moisture content for skim milk, whey, and high protein powders must be close to 4, 2–3, and 6%, respectively. The optimal storage temperature must be below the T_g temperature, which is close to 40–50°C at 0.2 a_w for SMP (Schuck et al., 2007).

1.4.2 Water transfer

In relation to the drying of dairy concentrates of high protein content, reports by Schuck et al. (1994a,b) have shown that the increase in casein content of the concentrate decreases the water diffusion through the dried product, that is, the final residue obtained at the end of drying. Water transport is probably affected by the high micellar casein content of the sprayed droplet in the atomization tower, and, similarly, when the powder granule is dissolved in water.

Studies by Schuck et al. 1998; 1999) showed that sodium caseinate and potassium caseinate dried more easily than calcium caseinate. The limited water diffusion through the calcium caseinate may be explained by the structure of this colloidal dispersion. Whereas the casein subunits are more aggregated in calcium caseinate because of calcium binding, in sodium and potassium caseinates, the caseins are more soluble. The results showed that the water bound in a micellar casein structure was more strongly bound than that bound to the soluble caseins in sodium caseinate. The situation was intermediate (i.e., between that of micellar casein and sodium caseinate) for calcium caseinate. It was assumed that these differences in water transfer during drying could be explained by the casein structure. The decrease in water inside the dairy concentrate led to a decrease in the water concentration on the surface of the concentrate in the water activity meter, or on the surface of the droplet during spray drying, and decreased the drying kinetics. These results were confirmed by the desorption drying of two different types of protein with the same protein content (89% of total solids) and the same water content before desorption drying, but with different drying time. All these results show that the drying rate is dependent on the nature and structure of the casein. Water may be less available during the drying of a protein with a micellar structure than during the drying of a protein with a globular structure.

1.4.3 Energy consumption

The aims of this section are to evaluate water transfer during the spray drying of different dairy concentrates (WPCs, WPC35, WPC50, WPC70, and isolates WPI90), with or without heat denaturation, MCI, sodium caseinate (NaCas), and milk (with and without whey

protein enrichment), using thermodynamic and biochemical approaches. When the concentrate temperature, air flow rate, concentrate flow rate, total solids content of the concentrate, inlet air absolute humidity, inlet air temperature before and after heating, and outlet air temperature after drying are known, it is possible to determine the specific energy consumption (SEC), which is the ratio of the energy consumed in the evaporation of 1 kg of water (measured in kJ/kg water) (Bimbenet et al., 2002). Thus, if you spray dry only free water, the energy spent in terms of the SEC would be close to 2500 kJ/kg water. For example, if the concentrate has increasingly greater amounts of bound water to free water, the SEC increases to 10,000 kJ/kg water. The significance of very high SEC is related to the water, which becomes less and less available, limiting water transfer, and thus increasing the surface temperature of the droplet and consequently increasing the risk of protein denaturation of the powder.

1.4.3.1 *Whey proteins*

The results presented in Table 1.1 show that water transfer during spray drying decreased when the whey proteins were native proteins. For the same moisture, the SEC for drying was higher when (1) the native whey protein content increased in WPC and in milk and (2) the whey proteins were heat denatured in WPC35. However, the SEC was lower when the whey proteins were heat denatured in WPC50, WPC70, or WPI90. These results can be explained by the availability of the water (bound and unbound) in the concentrate in relation to the nature and the content of the whey proteins.

1.4.3.2 *Caseins*

The results presented in Table 1.1 show that water transfer during spray drying decreased when the micellar casein content increased. For the same moisture, the SEC for drying was higher when (1) the micellar casein content increased in MCI compared with skim milk, and (2) casein remained in a micellar state (as in MCI) rather than a soluble state (e.g., in NaCas). These results can be explained by the availability of the water in the concentrate in relation to the content and structure of the caseins. Water is more available when the caseins are soluble than when they are in a micellar state.

All these results also show that water transfer depends on the relationship between the water and the protein components, and that these components should be taken into account when optimizing spray drying parameters for proteins. Proteins have an important role in the mechanisms of water transfer during drying and rehydration. The residence time of the

Table 1.1. Specific energy consumption for the drying of dairy powders at 4% moisture.

	WPC						**WPI**		**MCI**	**NaCas**
Protein content (%)	35		50		70		90		90	90
Heat treatment (72°C/4 min)	N	Y	N	Y	N	Y	N	Y	N	N
SEC (±3%) (kJ/kg water)	5950	7700	6800	6550	7050	6600	7200	6500	6900	5900

Y, heat treatment; N, no heat treatment; WPC, whey protein concentrate; WPI, whey protein isolate; MCI, micellar casein isolate; NaCas, sodium caseinate; SEC, specific energy consumption at 4% moisture content.

droplet and then the powder is so short that it is very difficult to study the mechanism of the structural changes in the protein without fundamental research into relationships between the process/product interactions.

1.5 POWDER PROPERTIES

Milk powders may be categorized by their physical, functional, biochemical, microbiological, and sensory properties (Carić, 2003). There is a significant interrelationship between them, which affects the final quality. The physical and functional properties of milk powders are especially important when the powders are intended for recombining and in the manufacture of various food products. When intended for use as a food ingredient, milk powders should be light in color, free of off-flavors, and easy to hydrate, disperse, and dissolve in water. The basic properties that determine the quality of milk powder, and where defects are most likely, include powder structure, solubility, water content, scorched particles, flowability, oxidative changes, flavor, color, and microorganism contamination.

1.5.1 Powder structure

The physical structure of milk powders can be defined as the way in which its chemical components are distributed and connected.

Powder structure is very strongly affected by the drying technique. Powder produced by roller drying has a compact structure of irregular shape with no occluded air. Roller-dried powder particles have a low bulk density (300–500 kg/m^3) due to their irregular structure. The particles of spray-dried powder are spherical, with diameters in the range 10–250 μm. The particles contain occluded air and either large central vacuoles or smaller vacuoles, which are distributed through the interior of particles. The surface of spray-dried SMP particles is usually wrinkled but is smooth for high protein powders. The high inlet air temperature and large temperature differential between the hot air and the powder particles are the main causes of wrinkle formation, as is also the presence of lactose (Carić and Kaláb, 1987; Mistry et al., 1992; Aguilar and Ziegler, 1993).

1.5.2 Particle size distribution

According to Carić (2003), the particle size of powders, which affects its appearance, reconstitution, and flow characteristics, depends mainly on the atomization conditions and the viscosity of the concentrate. High atomizing pressure and low concentrate viscosity reduce particle size.

1.5.3 Powder density

Densities are classified into three groups: bulk (apparent) density, particle density, and the density of the dry milk solids. All three are very much interrelated.

1.5.3.1 *Bulk density*

Bulk density is regarded as the weight per unit volume and is expressed as kg/m^3. It is a very important property, both from the point of view of cost and market requirements. Bulk

density is currently determined by measuring the volume of 100 g of powder in a 250-mL graduated glass cylinder. The bulk density of milk powders is a very complex property, being the result of many other properties and influenced by a number of factors, such as feed concentration, feed temperature, feed foamability, milk preheating, age thickening, feed composition, type of atomizer, particle temperature history, and particle size distribution (Pisecky, 1997). Bulk density depends also on particle density and occluded and interstitial air.

1.5.3.2 *Particle density*

Particle density corresponds to the mass of particles (in grams) having a total volume of 1 cm^3. Particle density is influenced mainly by the amount of entrapped air. The processing factors that contribute significantly to particle density are viscosity and the incorporation of air into the concentrate prior to drying. The type of spray atomization affects air retention. Certain types of centrifugal spray-dried milk have more entrapped air than pressure spray products (Carić, 2003).

1.5.3.3 *Occluded air*

The occluded air content is defined as the difference between the volume of a given mass of particles and the volume of the same mass of air-free milk solids.

Many factors influence the occluded air content in powder particles, including incorporation of air into the feed, the system chosen for spray drying the concentrate, whipping action before and/or during atomization, properties of the feed, and the ability of the feed to form a stable foam. The content and state of proteins might markedly affect stable foam formation, while fat has the opposite effect. High fat concentrates are much less susceptible to foaming than skim milk. Undenaturated whey proteins in skim milk have a greater tendency to foam, which can be reduced by heat treatment, which causes protein denaturation. Concentrates with a low total solids content foam more than highly concentrated content. A higher temperature reduces the tendency to form foam (Carić, 2003).

1.5.3.4 *Interstitial air*

Interstitial air is defined as the difference between the volume of a given mass of particles and the volume of the same mass of tapped powder. This property depends primarily on the particle size distribution and the degree of agglomeration (Carić, 2003).

1.5.4 Flowability

Flowability is the ability of a powder to flow freely, like sand, without forming lumps, clusters, or aggregates. Flowability can be measured as the time (in seconds) necessary for a given volume of powder to leave a rotary drum through given slits (Haugaard Sorensen et al., 1978) or by the method developed by Carr (1965). Flowability depends also on particle size and shape, density, and electrical charge. Large particles flow more easily than fines (particles with a diameter of <90 μm). Consequently, agglomeration is beneficial, as is uniformity of size. Moreover, according to Carić (2003), a wide variation in particle size permits fines to occupy spaces between the large particles, which results in closer packing.

1.5.5 Rehydration of dairy protein powders

Most food additives are prepared in powder form and need to be dissolved before use. Water interactions in dehydrated products and dissolution are thus important factors in food development and formulation (Hardy et al., 2002). Dissolution is an essential quality attribute of a dairy powder as a food ingredient (King, 1966). Many sensors and analytical methods, such as the ISI (International Dairy Federation, 1988; American Dairy Products Institute, 1990), nuclear magnetic resonance (NMR) spectroscopy (Davenel et al., 1997), turbidity, viscosity and particle size distribution (Gaiani et al., 2006) can now be used to study water transfer in dairy protein concentrates during rehydration. Using combinations of these methods, it is very easy to determine the different stages of the rehydration process (i.e., wettability, swellability, sinkability, dispersibility, and solubility).

1.5.5.1 *Stages of the rehydration process*

Wettability is the ability (expressed as time in seconds) necessary for a given amount of powder to penetrate the still surface of water. In other words, wettability is the ability of a powder to absorb water on the surface and get wet (Haugaard Sorensen et al., 1978). Generally, the wettability of powder particles depends on the surface activity of the particles, surface area, surface charge, particle size, density, porosity, and presence of moisture-absorbing substances.

Sinkability is the ability of powder particles to overcome the surface tension of water and sink into water after passing through the surface. Sinkability is expressed as milligrams of powder that sink per minute per centimeter square of surface area. This property of powder is influenced by the forces that tend to submerge a particle on the surface and depends on the density of the particles, that is, on the mass of the particles and the quantity of occluded air. Higher particle density and lower quantity of occluded air cause particles to sink (Carić, 2003).

Dispersibility reflects the ability of the wetted aggregates of powder particles to become uniformly dispersed when in contact with water. The effects of total heat treatment on casein during processing are of particular importance for good dispersibility. The dispersibility of milk powder can be improved by: (1) keeping the heat treatment on preheating to a minimum and (2) minimizing the holding time and temperature of the concentrate.

The ISI (in %), described by the IDF standard (International Dairy Federation, 1988) for skim milk, is the volume of sediment (for 50 mL) after rehydration (10 g of powder in 100 mL of distilled water, at 25°C), mixing (90 seconds, at 4000 rev/min) and centrifugation (300 seconds, at 160 *g*). With this method, the quantity of insoluble material (true and false not differentiated) can be determined.

1.5.5.2 *Methods for assessing rehydration properties*

NMR spectroscopy is a technique for determining the rate of solution, the time required for complete reconstitution of powders, and the transverse relaxation rate of reconstituted solutions (Davenel et al., 1997). With this method, it is possible to differentiate between the truly insoluble material and the falsely insoluble material. The falsely insoluble material can be explained by low water transfer during rehydration and not by denatured protein, which is truly insoluble (Schuck et al., 1994b).

For viscosity measurement, a rheometer can be used to obtain viscosity profiles. In our study, the blades were placed at right angles to each other to provide good homogenization. Industrial dissolution processes usually include stirring at a constant speed, and the experiments were therefore designed to provide a constant shear rate (100 per second). MCP was added to the rheometer cup manually. The aqueous phase used was distilled water at a volume of 18 mL. The powder was dispersed in the rheometer cup 50 seconds after starting the rheometer. Dissolution is highly dependent on temperature and concentration. The total nitrogen concentration employed to study these effects was about 5% (w/v), and the temperature was about 24°C (Gaiani et al., 2005, 2006).

The experiments to provide the turbidity profiles were carried out in a 2-L vessel equipped with a four-blade 45° impeller rotating at 400 rev/min. A double-walled jacket vessel maintained the temperature at 24°C. The turbidity sensor was placed 3 cm below the surface of the water and was positioned through the vessel wall to avoid disturbance during stirring. Turbidity changes accompanying powder rehydration were followed using a turbidity meter. The apparatus uses light in the near-infrared region (860 nm), the incident beam being reflected back at 180° by any particle in suspension in the fluid to a sensitive electronic receptor (Gaiani et al., 2005).

A laser light diffraction apparatus with a 5-mW He–Ne laser operating at a wavelength of 632.8 nm can be used to record particle size distributions. In the study of Gaiani et al. (2005, 2006), the particle size distribution of dried particles was determined using a dry powder feeder attachment, and the standard optical model presentation for particles dispersed in air was used. To measure the particle size distribution of micellar casein in concentrates, 0.5 mL of suspension was taken from the rheometer cup and introduced into 100 mL of prefiltered distilled water (membrane diameter, 0.22 µm) to reach the correct obscuration. The results obtained corresponded to average diameters calculated according to the Mie theory. The criterion selected was $d(50)$, meaning that 50% of the particles had diameters lower than this criterion (midpoint of cumulative volume distribution) (Gaiani et al., 2005, 2006).

Using this combination of three methods, it was possible to follow water transfer during rehydration and to obtain the wetting time, determined using the first peak of increased viscosity and turbidity, and the swelling time, determined using the second peak of viscosity in relation to the increase in particle size. The rehydration time was then determined according to stabilization of the viscosity, turbidity, and particle size values.

The results in Table 1.2 show that rehydration of MC powder occurs in various stages. First, there is wetting and swelling of the particles, followed by slow dispersion to reach a homogeneous fluid (Gaiani et al., 2005, 2006). Using an NMR method, Davenel et al. (1997) also demonstrated two stages during MC rehydration, attributed to water absorption by powder and solubilization of particles (i.e., swelling and dispersion stages). These authors estimated the water uptake by the powder at around 5 g water/g powder during the first 20 minutes of rehydration, but could not identify a wetting stage with this method.

1.5.5.3 *Rehydration properties of various dairy protein powders*

MC powders with a high ISI (14.5 mL) are generally considered to be poorly soluble powders in which rehydration of the micelle remains incomplete (Jost, 1993). On the other hand, the rehydration of whey powders in our study was totally different (Table 1.2). As the wettability of whey powders is poor, the turbidity instability at the beginning of the profile may be due to lump formation going past the sensor, as reported by Freudig et al.

Table 1.2. Reconstitution period, insolubility index, and rehydration time of dairy protein powders.

Powders	RP using NMR (minutes)	ISI using IDF standard (mL)	WT (minutes)	ST (minutes)	DT + SolT (minutes)	RT (minutes)
MCP (G)	22	14.5	1	2	804	807
MCP (NG)	8	3.5	3	17	551	571
WPP (G)	5	<0.5	4	0	0	4
WPP (NG)	15	<0.5	17	0	0	17

MCP, micellar casein powder; G, granulated; NG, nongranulated; WPP, whey protein powder; RP, reconstitution period; ISI, insolubility index; WT, wetting time; ST, swelling time; DT, dispersibility time; SolT, solubility time; RT, rehydration time = WT + ST + DT + SolT.

(1999). For nongranulated (NG) WPI powder, the very long signal instability may be explained by a tendency for the lumps to stick together in a thick layer of wet particles, due to the small size of the particles. Powder swelling has not been reported for WPI powders, probably because globular protein powders bind less water than intact casein micelle powders. Many authors have also reported that whey powders have a lower water holding capacity than casein powders (De Moor and Huyghebaert, 1983).

As expected, granulation has been a positive effect on wetting. The wetting time has been systematically better for granulated particles. This phenomenon is well known, as fast wetting is enhanced, with large particles forming large pores, high porosity, and small contact angles between the powder surface and the penetrating water (Freudig et al., 1999). A surprising influence of granulation on the rehydration time was observed in our study (Gaiani et al., 2005). Depending on the nature of the protein, the granulation influence had opposite effects. WPI rehydration was enhanced for granulated particles, whereas the rehydration time was shorter for nongranulated particles of MCP. This was unexpected and could be explained by the rate-controlling stage. The controlling stage for whey proteins is wetting. As granulation improves the wetting stage, the rehydration of whey powders is enhanced for granulated particles. In contrast, in our study, the controlling stage for casein proteins was dispersion. In fact, even with a shorter wetting time, a granulated powder is slower to rehydrate than a nongranulated powder (Gaiani et al., 2005).

These results are not compatible with those of other studies, in which it was generally accepted that a single particle size around 200 µm (Neff and Morris, 1968) or 400 µm (Freudig et al., 1999) represented optimal dispersibility and sinkability. In fact, this optimal particle size depends on the composition of the dairy powder. As shown in Table 1.2, if the industry wishes to optimize powder rehydration, it seems to be better to rehydrate granulated powders when the protein is whey and to rehydrate nongranulated powders when the protein is casein.

A modification of the rehydration parameters recommended by the IDF method (1988) induced notable variations in the ISI of MCI powder. This index decreased to 6.2 mL if the casein content of the sample of MCI powder was close to the casein content of milk (25 g/L), if the duration of stirring increased from 90 to 900 seconds (ISI = 1.8 mL), if the rehydration temperature increased from 24 to 30°C (ISI = 7.2 mL), 40°C (ISI = 2.3 mL), 50°C (ISI = 0.9 mL%), or 60°C (ISI = 0.1 mL) or if the stirring velocity increased from 4000 to 10,000 rpm (ISI = 0.8 mL). The substitution of the rehydration water by a saline solution (NaCl at 0.1 mol/L), a microfiltrate, or an ultrafiltrate did not change the ISI for MCI powder (Schuck et al., 1994a,b). These results confirm that the ISI given by the IDF method (1988) results from a decrease in the water transfer toward the center of the particle

Table 1.3. Examples of biochemical and physical properties of some high protein powders.

	Unit	MCI	MPI	NaCas	WPI
a_w	–	0.24	0.22	0.20	0.19
Free moisture	g/kg	944.3	949.5	944.4	931.7
Total moisture	g/kg	932.5	938.2	938.2	924.8
$Ashes_{(550°C)}$	g/kg	88.9	75.8	36.9	44.5
TMN (TN × 6.38)	g/kg	846.4	833.6	905.3	839.8
$NS_{pH4.6}$ × 6.38	g/kg	75.3	215.4	12.1	732.5
NPN	g/kg	5.5	2.5	2.2	29.2
Color L	–	69.6	73.5	73.2	73.1
Color a	–	–5.1	–5.5	–5.8	–6.0
Color b	–	12.0	9.3	10.0	13.1
Dispersibility	%	5.1	25.6	10.0	66.0
Solubility	%	64.6	59.5	99.8	99.8
Wetability	s	>120	>120	>120	>120
d (0.1)	µm	166	20	18	23
d (0.5)	µm	287	65	77	75
d (0.9)	µm	473	135	259	205
T_g onset	°C	70	65	57	82
Flowability	–	71	47	46	56
Floodability	–	52	51	58	62
Bulk density	kg/m^3	242	292	303	318
Packed density	kg/m^3	290	354	570	507
Particle density	kg/m^3	1168	1205	1301	1224
Interstitial air	$cm^3.100g^{-1}$	68	60	155	117
Occluded air	$cm^3.100g^{-1}$	259	199	99	116
H at 39% RH	%	6.9	6.3	5.9	6.2
H at 75% RH	%	11.7	13.5	10.9	15.4

NaCas, sodium caseinate; MCI, micellar casein isolate; MPI, milk protein isolate; WPI, whey protein isolate; a_w, water activity; TNM, total nitrogen matter; TN, total nitrogen; NS, soluble nitrogen at pH 4.6; NPN, non protein nitrogen; *d*, diameter of the particle, (0.*n*), $n \times 100\%$ of particles which have a size below *d*; T_g, glass transition temperature; H, hygroscopicity; RH, relative humidity.

and not from denaturation. Everything occurs as if the setting in contact with water created a high surface viscosity, slowing down the internal hydration of the MCI powder.

To summarize, Table 1.3 gives some examples of findings related to the physical and biochemical properties of various high protein powders.

1.6 CONCLUSION

Figure 1.4 shows that the biochemical, microbiological, and physical properties of a dairy powder and its recombined product depend on many parameters. For example: (1) the pretreatment process parameters, concentration by membrane filtration or by vacuum evaporation, crystallization, homogenization, spray drying, and fluidization, (2) the storage conditions (e.g., relative humidity, temperature, packaging) used to optimize stability over time, and (3) the rehydration conditions (e.g., stirring conditions, temperature, and concentration) used to improve water transfer to obtain the best quality recombined product from the corresponding powder.

Moreover, the biochemical composition (nature and content) and water availability interact in all the stages of production, stability, and rehydration. The quality of a dairy

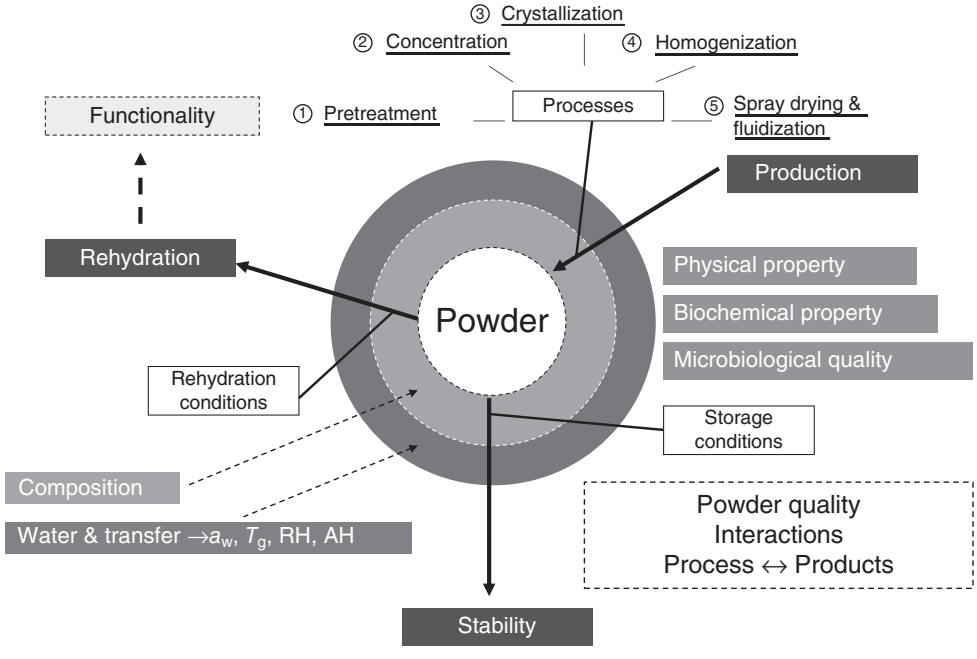

Figure 1.4 Properties of dairy powders in relation to production, storage, and rehydration.

powder can therefore only be improved if research is undertaken on the process–product interactions.

To conclude, this chapter explains certain processes for the extraction of milk protein and dehydration (spray drying) in order to clarify the effects of spray drying on the quality of protein powders during drying and rehydration. In this chapter, it was also demonstrated that the quality of these powders depends on the biochemical environment.

It is thus very important for the dairy industry to understand that enrichment of milk in micellar casein (by ultrafiltration or MF) decreases water transfer during the drying and rehydration processes. Insolubility (International Dairy Federation, 1988) is related to the lower water transfer required for rehydration and not to thermal denaturation, and reduction in water transfer is related to the micellar structure.

Moreover, it is essential for both dairy powder producers and dairy powder users to have a method to evaluate the rehydration behavior of dairy powders. The industry should take into account certain technological factors, such as granulation and the incorporation mode, and also the nature of the protein being rehydrated, in order to optimize the rehydration of dairy powders. In contrast to other studies, we found that improving the wetting stage by using granulated powders did not systematically improve total rehydration. Depending on the nature of the protein, it seems to be better to work with granulated powders for whey and nongranulated powders for micellar casein to obtain more rapid rehydration (Gaiani et al., 2007).

In conclusion, water transfer in dairy protein concentrates both during dehydration and during rehydration depends on the aqueous environment, the nature of the mineral salts, the structure of the dairy proteins, and powder properties (biochemical, microbiological, and physical).

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