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Review

# Challenges in dried whey powder production: Quality problems

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ABSTRACT

Whey is a high nutritional value by-product of the dairy industry. It is generally produced in large quantities and its disposal as wastewater poses environmental risks. For this reason, whey streams are used for the production of value-added products such as dried whey powders. However, there are several challenges related to whey processing that lead to low powder yield and quality, especially caking. These challenges can be addressed by optimization of product formulation and processing parameters. In this review, we discuss the effects of dried whey protein powder production stages and process parameters on the quality of the final powder product. The initial composition of whey used for dried whey powder production affects the final quality of the product. Generally, a high mineral and/or lactic acid content is not desirable since these constituents cause lactose-containing whey particles to adhere to the drying equipment surfaces, thereby reducing the powder yield. An effective lactose pre-crystallization is essential since high amorphous lactose content increases the stickiness of the dried-whey powder particles and induces caking during storage. Therefore, whey should undergo filtration and lactose pre-crystallization before spray drying. Studies show that it is possible to retard caking and improve the quality attributes of dried whey powders by optimizing the product formulation and processing operations.

## 1. Introduction

The dairy industry produces large quantities of whey as a by-product. The yearly production rate of whey is around 121 million tons per year (Petruccioli, Raviv, Di Silvestro, & Dinelli, 2019). In milk, 90% (v/v) of the total volume is composed of whey. Moreover, whey contains more than 50% (w/w) of the total nutrients in milk (Zandona, Blažić, & Režek Jambrak, 2021). Whey has a high organic load and it could become an environmental pollutant if disposed directly as wastewater. The chemical oxygen demand (COD) and biological oxygen demand (BOD) of cheese whey have been reported as 50–80 g/L and 40–60 g/L, respectively (Chatzipaschali & Stamatis, 2012). Protein, fat and lactose are the main components contributing to the high organic load of whey. To reduce the environmental hazards associated with the disposal of whey, as well as to increase the economic value of milk products, the dairy industry has converted whey into a range of value-added ingredients, which are used in the food and other industries for their nutritional and functional attributes (Alsaed et al., 2013). For this purpose, food and chemical industries recycle nearly 50% of residual whey for the production of value-added products (Panghal et al., 2018). For instance, dried whey powder (WP) is commonly produced from whey streams, which can then be used as a food ingredient, *e.g.,* as a flavor enhancer, texture modifier, or nutritional enhancer (Ostojić et al., 2005). However, WP is not the only value-added product of whey stream. In recent years, various technologies have been applied by dairy industry to process whey and produce a great variety of products such as sweetened condensed whey, whey protein concentrate, whey protein isolate, whey lactose, fermented or unfermented whey-based beverages and bio-ethanol (Panghal et al., 2018; Zandona et al., 2021). Especially, whey-based beverages have attracted a great attention in the recent years due to their genuine and refreshing taste. Commercially produced whey-based beverages include plain, carbonated, alcoholic, fruit-flavored as well as fermented beverages (Panghal et al., 2018). Moreover, whey permeate has also some potential applications in various fields (Mehra et al., 2021). Due to the high lactose content of whey permeate, it is generally further processed to obtain lactose in purified form (Das, Sarkar, Sarkar, Bhattacharjee, & Bhattacharjee, 2016). Utilization as wall material for the microencapsulation of probiotic microorganisms (Eckert et al., 2017), use as a source for the production of whey-based sport drinks (Abella et al., 2016) and incorporation in liquid fertilizer

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production (Akib & Setiawati, 2017) are some of the latest applications of whey permeate. Additionally, utilization of whey permeate as feedstock for biomass production of microalgae (Espinosa-Gonzalez, Parashar, & Bressler, 2014) and ethanol production by *Saccharomyces cerevisiae* (Parashar, Jin, Mason, Chae, & Bressler, 2016) has also been reported. It is obvious that there are many whey-based functional products produced by dairy industry. However, the main focus of this review will be on the challenges in dried whey powder (WP) production which is still a relevant issue for the dairy industry.

Other than their nutritional and functional attributes, whey dairy products have a big impact on the worldwide market, in correlation with their wide application potential. The global whey protein market value was around 9.19 billion U.S. dollars in 2020. Additionally, it is estimated to reach 18.56 billion U.S. dollars by 2028 (Wunsch, 2021). Besides the increasing market size of whey protein, the volume of whey powder produced also shows an increasing trend. For instance, the total volume of whey powder produced in the European Union (EU) was approximately 1.89 million tons in 2019. It is estimated that this number will reach 2.44 million tons by the end of 2030 (OECD-FAO Agricultural Outlook 2021 - 2030, 2021). Consumption rate of whey powder in EU is also in alignment with the production rate. The total whey powder consumption volume of 1.3 million tons in 2019 is estimated to increase gradually to 1.4 million tons by 2028 (OECD-FAO Agricultural Outlook 2019 - 2028, 2019).

Although the unit operations used for whey concentration and WP production are well established, there are still some challenges related to the overall production process. The most challenging part during the drying and storage of powders is to prevent the sticking of the particles as this causes caking of the powder (Carpin et al., 2016). The presence of lactose has been identified as the main reason for sticking and caking in dairy powders, which is related to glass transition phenomenon within the solid matrix (Chen et al., 2019). If the amorphous form of lactose dominants in the whey, then the high temperatures used during the drying process induce a phase change from a glassy to a rubbery state, which causes a large increase in the stickiness of the particle surfaces. As a result, inter-particle interactions, such as the formation of liquid bridges, promotes cohesion of the particles leading to caking and inferior powder quality (Chuy & Labuza, 1994). In addition, the particles can also adhere to the surfaces of drying equipment, which significantly reduces powder yield (Özkan, Walisinghe, & Chen, 2002).

Caking can also be observed during storage of WPs. In this case, the relative humidity (RH) and temperature experienced by the powders during storage have a major impact on the caking process. Typically, the glass transition temperature ($T_g$) of the lactose-rich solid matrix in WPs decreases as the RH increases. Consequently, high storage temperatures or RHs can lead to a glass-rubbery transition that promotes particle agglomeration and caking (Langrish, 2008). Therefore, the residual amorphous lactose content in final WPs should be kept at a minimum.

In this article, we begin by reviewing the processing operations used to prepare WPs and highlight some of the major problems associated with producing them, including powder caking and low yields. We then discuss the impact of whey composition and processing operations on powder quality and yield, with an emphasis on optimizing these parameters to improve WP production.

## 2. Whey powder production

In this section, we provide a *concise* overview of the entire WP production process. An overall representative flow chart of WP production is given in Fig. 1. Details about the processes and their effects on the quality will be referred in more detail in latter sections. The process starts with coagulation of the casein fraction from milk, *e.g.,* using acid or enzyme methods. The whey obtained is then separated from the casein fines and whey cream (fat) by centrifugation. Then, the whey is subjected to pasteurization for microbial inactivation. After pasteurization, the whey is passed through a membrane filtration unit (usually nanofiltration) for partial ion removal, deacidification, and concentration of the whey stream. After filtration, the whey has a total solids (TS) content of around 15 to 20% (w/v). The whey is then evaporated to achieve further concentration. Mild evaporation conditions increase the TS and temperature of whey to 55–60% (w/v) and around 70 °C, respectively. Subsequently, the whey concentrate is fed to a crystallization tank for controlled lactose pre-crystallization. After slow crystallization (12–48 h), the whey concentrate leaves the crystallization tank at around 15 °C or lower. Finally, spray drying is performed using inlet air temperatures of around 165 to 180 °C and outlet air temperatures of around 85 to 90 °C. The fresh powder leaves the spray dryer at around 85 to 90 °C, is cooled down to around 25 to 28 °C, and is then packaged (Smithers & Augustin, 2013).

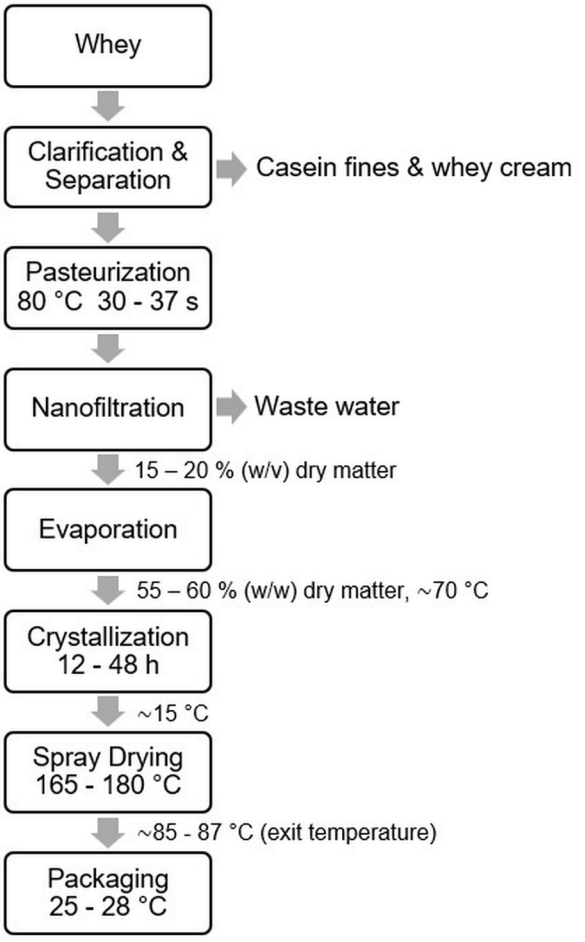


**Fig. 1.** A representative flow chart of whey powder production process.

## 3. Quality problems encountered in WP production

Each processing step may influence the final powder product yield and quality. However, the most encountered problem in WP production is caking, which results in a sticky product. Research on dairy powders has shown that lactose pre-crystallization after concentration but before drying is crucial for reducing caking and improving powder quality (Aguilera, del Valle, & Karel, 1995). During pre-crystallization, the amorphous form of lactose in whey concentrates is converted into a crystalline form using a crystallization tank. The crystalline form of lactose is less susceptible to phase changes when exposed to elevated temperatures during drying, so particle sticking is less of a problem (Fitzpatrick, O'Connor, Cudmore, & Dos Santos, 2017). However, the efficiency of lactose pre-crystallization is greatly influenced by mineral type and whey composition. Typically, high mineral contents inhibit

lactose crystallization. Consequently, membrane filtration is often used to reduce the mineral content of whey before evaporation (Kumar et al., 2013). Membrane filtration also partially concentrates the whey, thereby reducing the temperature/time required in the subsequent evaporation process (Suárez, Lobo, Alvarez-Blanco, Riera, & Álvarez, 2006). Another advantage of whey filtration is the partial removal of lactic acid since it may also retard lactose crystallization (Jelen & Coulter, 1973).

Caking may be induced by different physicochemical processes. The two most common of these processes are known as amorphous and mechanical caking.

### 3.1. Amorphous caking

Amorphous caking is the most common caking mechanism encountered in WPs (Listiohadi, Hourigan, Sleigh, & Steele, 2005). When particles are heated above the glass transition temperature ($T_g$) they enter the rubbery state, which causes their molecular mobility and surface stickiness to increase. As a result, the particles may partially fuse together and form clumps held together by liquid bridges (Palzer, 2005). If the temperature is increased further, the amorphous particles may become more crystalline, which leads to the formation of solid bridges between them, a process known as sintering (Haider et al., 2014). However, sintering is not the only caking mechanism that is triggered by amorphous lactose. The hygroscopic character of amorphous lactose is capable of inducing caking even in mostly crystalline WP. This type of caking occurs during storage due to moisture-induced crystallization of amorphous lactose (Carpin et al., 2016). In Fig. 2, we provide scanning electron microscope (SEM) images of fresh and caked WP samples at two different magnification levels. The fresh sample was deliberately subjected to storing conditions that would induce caking in WP. Distinctions between the fresh and caked samples can easily be observed at both magnification levels. Fresh WP samples have smooth well separated – individual spherical particles (Fig. 2a, c), whereas caked WP samples have large particulate clusters with indefinite shapes (Fig. 2b, d). Bridging between the particles in caked samples (Fig. 2b, d) is also apparent. This is an indication of amorphous caking triggered by the presence of amorphous lactose.

Moisture-induced caking of WP occurs when water absorption by amorphous lactose particles exceeds 5% (w/w) of its dry mass at 25 °C. Absorption of water continues until a moisture content of 10% (w/w) is reached and $T_g$ also continuously declines as more water is absorbed into the bulk of WP (Roos & Karel, 1992). $T_g$ eventually drops below the storage temperature of the WP. Since the molecular mobility of lactose is increased under these conditions, crystal nucleation mostly leads to the formation of α-lactose monohydrate (Lai & Schmidt, 1990). During crystal nucleation, the absorbed water is released and then facilitates the transportation of dissolved lactose molecules to new nucleation sites (Burnett, Thielmann, Sokoloski, & Brum, 2006). The α-anomer of lactose crystals then induces caking due to capillary condensation (Paterson & Bronlund, 2009). However, this type of caking is usually only observed at high moisture levels (25 °C, 75% RH), which are not usually encountered under normal storage conditions (Listiohadi et al., 2005). Nevertheless, crystallization of amorphous lactose can also take place at lower RH levels, but in one of the anhydrous forms (Berggren & Alderborn, 2004). These anhydrous crystals generally consist of fine needle-like crystals that can fuse together. Therefore, WP that experiences moisture-induced crystallization at low to medium RH conditions,

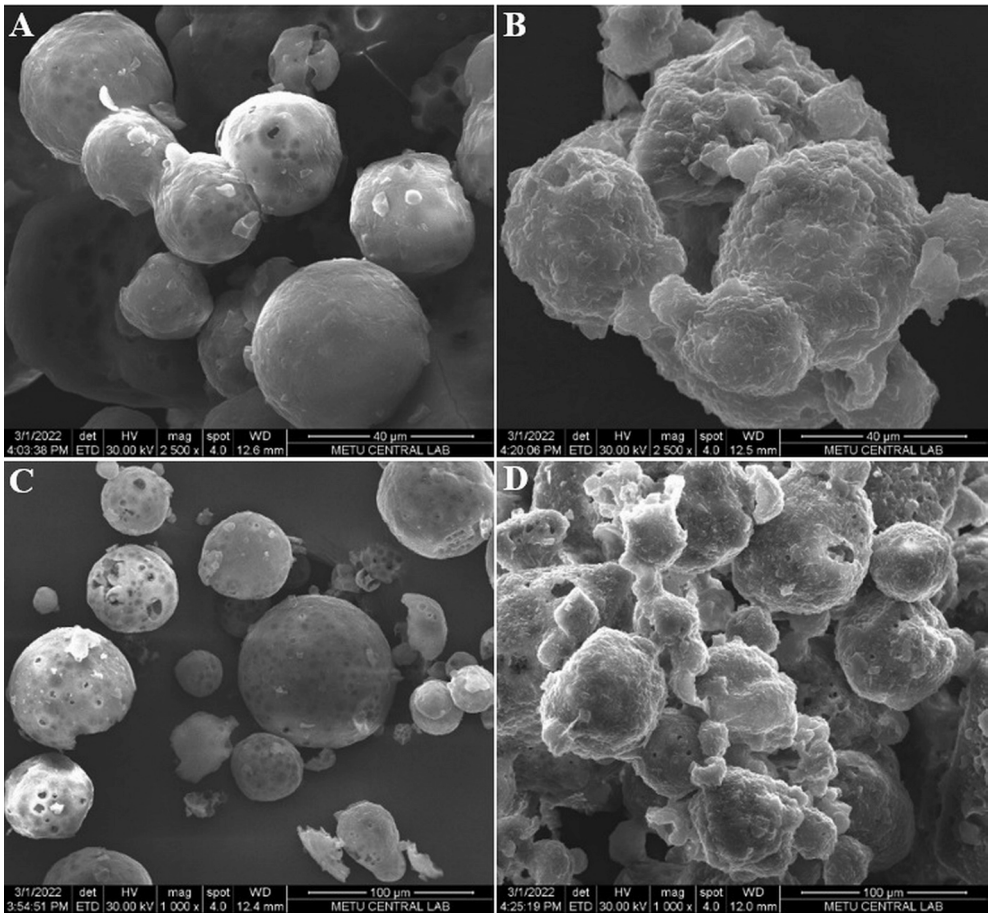


**Fig. 2.** SEM images of a) fresh whey powder at 2500× magnification, b) caked whey powder at 2500× magnification, c) fresh whey powder at 1000× magnification, d) caked whey powder at 1000× magnification.

shows caking into hard crystalline agglomerates, which significantly reduces powder quality (Briggner, Buckton, Bystrom, & Darcy, 1994).

### 3.2. Mechanical caking

In dry powders, particles are attracted to each other through van der Waals interactions (Schulze, 2008). If a mechanical pressure is applied to a powder, then the particles are forced closer together, which can cause them to partially fuse with each other, leading to mechanical caking (Hartmann & Palzer, 2011). As a result, the cohesion of the particles in the powder is increased. The size and shape of the particles in a powder, as well as the magnitude of the pressure applied, are the main factors determining the extent of mechanical caking in WP (Carpin et al., 2016). The depth of the powder bed is also important as the highest pressure is applied to the powder particles at the bottom. As the total height of a powder bed increases, the probability of mechanical caking occurring increases (especially at the bottom). Thus, the risk of mechanical caking occurring increases when WP is stored in large sacks, containers, or piles (Schulze, 2008). In general, a threshold pressure must be exceeded before mechanical caking occurs, which depends on particle properties and ambient storage conditions (Röck & Schwedes, 2005). Fig. 3 provides a schematic representation of amorphous and mechanical caking mechanisms.

## 4. Effects of whey composition and processing parameters on powder quality

### 4.1. Whey composition

The typical chemical composition of whey includes lactose (44–52 g/L), protein (3–10 g/L), calcium (0.4–1.6 g/L), phosphate (1–4.5 g/L), lactate (2–6.4 g/L), and chloride (1–1.2 g/L) with a total solids concentration of 63–70 g/L (Jelen, 2009). Variations in these concentrations originate from differences in the type of milk and isolation methods used to produce the whey. There are two main types of whey, acid and sweet whey, which differ in the way they are produced and that have different compositions (Fischer & Kleinschmidt, 2015). Table 1 summarizes the variations in the acid and sweet whey compositions. Acid whey is produced by acid coagulation of dairy products such as Greek yogurt and soft cheese. The casein fraction tends to coagulate and form a curd when the pH of milk is reduced to around 4.0–4.5, which can then be separated to obtain the acid whey (Stănciuc, Der Van Plancken,

**Table 1**
Variations in the compositions of acid and sweet wheys.

| | Acid Whey Composition | Sweet Whey Composition |
|---|---|---|
| **Total protein** | 0.8–1.0% (m/v) (Chen et al., 2016) | 0.6–1.0% (m/v) (Chen et al., 2016) |
| | 0.5% (w/w) (Chandrapala & Vasiljevic, 2018) | – |
| | 3 g/L (Fischer & Kleinschmidt, 2015) | 5.6 g/L (Fischer & Kleinschmidt, 2015) |
| **Lactose** | 38–49 g/L (Chen et al., 2016) | 46–52 g/L (Chen et al., 2016) |
| | 4.6% (w/w) (Chandrapala & Vasiljevic, 2018) | – |
| | 38 g/L (Fischer & Kleinschmidt, 2015) | 38 g/L (Fischer & Kleinschmidt, 2015) |
| **Lactic acid** | 0.55% (w/w) (Chandrapala & Vasiljevic, 2018) | – |
| | 8.1 g/L (Fischer & Kleinschmidt, 2015) | 0.58 g/L (Fischer & Kleinschmidt, 2015) |
| **pH** | 4.0–4.6 (Chen et al., 2016) | 5.9–6.3 (Chen et al., 2016) |
| **Ca** | 43–160 mg/100 mL (Chen et al., 2016) | 40–60 mg/100 mL (Chen et al., 2016) |
| | 0.1% (w/w) (Chandrapala & Vasiljevic, 2018) | – |
| | 25.5 mM (Fischer & Kleinschmidt, 2015) | 8.5 mM (Fischer & Kleinschmidt, 2015) |
| **Na** | 40–61 mg/100 mL (Chen et al., 2016) | 50 mg/100 mL (Chen et al., 2016) |
| | 18.6 mM (Fischer & Kleinschmidt, 2015) | 16.5 mM (Fischer & Kleinschmidt, 2015) |
| **K** | 143–182 mg/100 mL (Chen et al., 2016) | 160 mg/100 mL (Chen et al., 2016) |
| | 45.2 mM (Fischer & Kleinschmidt, 2015) | 29.6 mM (Fischer & Kleinschmidt, 2015) |
| **Mg** | 9 mg/100 mL (Chen et al., 2016) | – |
| | 4.9 mM (Fischer & Kleinschmidt, 2015) | 2.8 mM (Fischer & Kleinschmidt, 2015) |
| **Cl** | 91–110 mg/100 mL (Chen et al., 2016) | 110 mg/100 mL (Chen et al., 2016) |
| | 42.6 mM (Fischer & Kleinschmidt, 2015) | 21.5 mM (Fischer & Kleinschmidt, 2015) |
| **P** | 44–90 mg/100 mL (Chen et al., 2016) | 32–96 mg/100 mL (Chen et al., 2016) |
| **Citrate** | 100 mg/100 mL (Chen et al., 2016) | – |
| **Phosphate** | 17.6 mM (Fischer & Kleinschmidt, 2015) | 8.4 mM (Fischer & Kleinschmidt, 2015) |

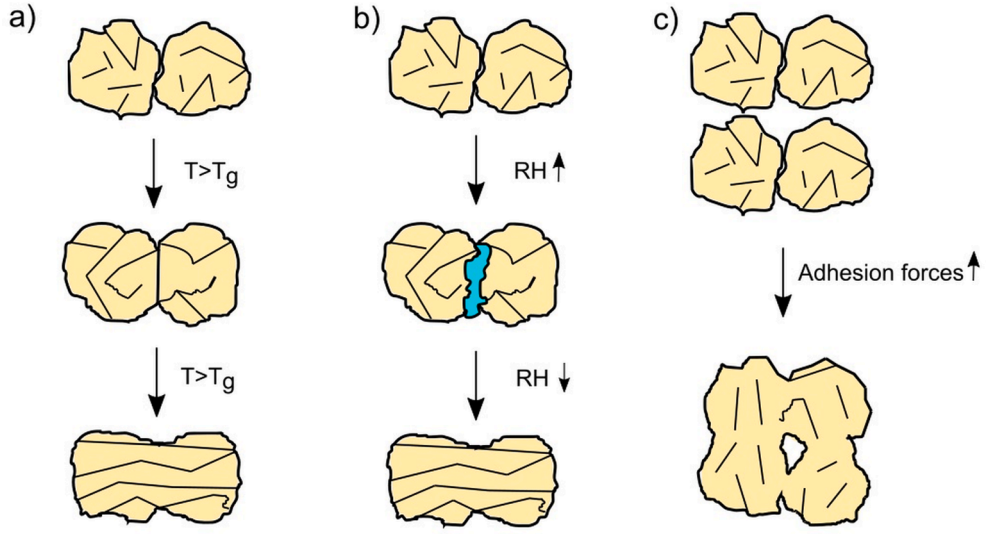


**Fig. 3.** Representative caking mechanisms of whey powder particles; a) temperature induced amorphous caking, b) moisture induced amorphous caking and c) mechanical caking.

Rotaru, & Hendrickx, 2008). Acid whey contains more minerals and lactic acid but less lactose than sweet whey. Sweet whey is produced by coagulation of casein using an enzyme (rennet) during cheese production. This enzyme cleaves a hydrophilic macropeptide from the κ-casein molecules that reside at the exterior of casein micelles, thereby reducing the steric repulsion between them, which leads to their aggregation. The pH of sweet whey is higher (6–6.5) than acid whey (Fischer & Kleinschmidt, 2015). Sweet whey has a relatively low mineral content (especially calcium) because rennet induces the formation of calcium *para*-caseinate, which retains the calcium within the coagulated casein fraction. In contrast, acid whey has a relatively high mineral content because less of the calcium is trapped within the curd. The higher calcium content in acid whey results in calcium lactate formation, which has negative impacts on the WP production process. Acid whey also contains more phosphate and citrate ions than sweet whey (Chen, Eschbach, Weeks, Gras, & Kentish, 2016). These differences in whey composition impact the production and properties of WP.

Since the acidity and mineral content of acid whey are higher than sweet whey, an extensive demineralization and deacidification is required during the production of WP from acid whey (Chen et al., 2016). Otherwise, proper lactose crystallization may not occur, which would lead to caking in the final powder (Chandrapala, Gauthier, & Vasiljevic, 2017). This problem arises because of the slower crystallization kinetics of acid whey samples caused by their higher mineral contents, which leads to the presence of more amorphous lactose that can promote particle aggregation (Chandrapala & Vasiljevic, 2018). As well as retarding lactose crystallization and reducing spray drying efficiency, the high mineral content of acid whey also impairs the evaporation process. Mineral fouling occurs at evaporator surfaces and overall heat transfer resistance increases (Modler & Lefkovitch, 1986). The lactic acid content of acid whey can be decreased by neutralization, but this process may impart a bitter and astringent flavor to the final powder product (Shrestha, Adhikari, Howes, & Bhandari, 2006).

### 4.2. Acidity and minerals

The presence of minerals and acids within whey affects the water-solubility of lactose (Bhargava & Jelen, 1996). In particular, the acids influence the pH, which regulates the behavior of lactose (Holsinger, 1997). Changes in lactose solubility influence the pre-crystallization operation efficiency. If lactose molecules are not supersaturated sufficiently, crystallization of lactose is inhibited and so spray drying causes caking in WP (McLeod, Paterson, Jones, & Bronlund, 2011). Therefore, the effects of minerals and acids on the behavior of whey during processing should be understood and controlled. The presence of lactic acid and calcium in whey is generally associated with poor WP quality due to caking (Nickerson & Moore, 1974). Lactic acid exhibits hygroscopic behavior, which causes it to strongly interact with the water molecules in its environment. As a result, a strong hydration layer containing lactic acid and $H_3O^+$ ions is formed around the lactose molecules, which increases their solubility and inhibits their crystallization (Wijayasinghe, Vasiljevic, & Chandrapala, 2015). Minerals also influence the solubility and crystallization behavior of lactose molecules. If the minerals in whey have strong electric fields, like divalent calcium cations, then they can act as water structure promoters (von Hippel & Schleich, 1969). The structural changes in water caused by calcium ions affect the solubility of lactose (Mullin, 1979). The strong ion–dipole interactions between calcium cations and water restrict the mobility of the water molecules, thereby inducing a hydration layer around the lactose molecules similar to the action of lactic acid (Reid & Fennema, 2008). Hence, lactose crystallization is inhibited. On the other hand, ions with a low charge density, such as chlorine, do not strongly structure the water molecules, which also affects lactose solubility and crystallization (Bhargava & Jelen, 1996). Despite some contradictory reports, it has been found that potassium, sodium, calcium, and phosphate ions inhibit lactose crystal nucleation and promote caking in WP (Guu & Zall, 1991). In contrast, lactates and citrates accelerate the rate of lactose crystal nucleation (Gernigon et al., 2013), which would be expected to inhibit caking. Mineral ions can also be present within the salt forms of whey and their effects on lactose solubility and nucleation and crystal growth rates are quite complex. According to Bhargava and Jelen (1996), low concentrations of calcium chloride and potassium chloride promote lactose crystallization, whereas high concentrations inhibit it. The authors also reported that dipotassium phosphate had a strong inhibitory effect on lactose crystal growth (Bhargava & Jelen, 1996). Mineral ions may also be incorporated into pre-existing crystal lattices of lactose, which affects the crystallization rate. For instance, formation of dilactose phosphate complexes inhibits crystal growth by adsorbing onto specific crystal surfaces (Hartel & Shastry, 1991). Thus, reducing the phosphate ion content of whey would reduce the risk of caking of WP.

The pH of the system also plays a crucial role in determining the crystallization behavior of lactose molecules (Holsinger, 1997). Firstly, the pH of whey determines the solubility of acids and minerals. At low pH values, lactic acid exhibits stronger hygroscopic behavior because it is largely in the undissociated form (Chandrapala et al., 2015). Calcium is also more soluble at low pH (Smithers & Augustin, 2013). Chandrapala et al. (2017) observed a low lactose crystal yield when the whey was adjusted to pH 3.0 where lactic acid ($pK_a$ 3.8) was in its undissociated form. In contrast, a significant increase in lactose crystal yield has been reported when the pH was increased to 6.5 at the same calcium and lactic acid concentrations (Chandrapala et al., 2017). Under these high pH conditions, lactic acid is transformed into its dissociated form (lactate), which has a lower water holding capacity. The form of lactate is also important in determining stickiness during drying. For instance, sodium lactate is extremely sticky as compared to potassium lactate (Smart, 1988). Calcium also loses its hygroscopic character at higher pH values. Therefore, calcium and lactic acid are less effective at structuring water molecules at higher pH values than at lower ones. Consequently, lactose molecules become more mobile and can interact with each other more quickly, which increases the crystallization rate (Chandrapala et al., 2017). Secondly, pH affects the mutarotation rate of lactose molecules. There is an anomeric equilibrium between the α- and β-anomers of lactose molecules (Fox, 2009). Mutarotation of lactose molecules enables the interchange between α- and β-anomers. This interchange and the resulting alteration in the ratio of α-to-β anomers depends on many factors, such as lactose concentration, temperature, presence of impurities, and pH (Hartel & Shastry, 1991). High alkaline conditions have been reported to accelerate mutarotation of lactose but generally extreme pH values are required for a significant change in the lactose mutarotation rate (Herrington, 1934). Nevertheless, even small changes in the anomeric equilibrium of lactose may influence the interactions between water and lactose molecules leading to alterations in the crystallization behavior (Holsinger, 1997).

Lactic acid and most minerals retard lactose crystallization, especially when present at high concentrations, which promotes powder caking, thereby reducing the quality and yield of WP. Consequently, it is important to reduce the concentrations of these components in whey. In the following section, processing operations that can be used to reduce the mineral and lactic acid load of whey are discussed.

### 4.3. Filtration

Nanofiltration is currently the most common filtration technique used in the dairy industry. However, electrodialysis may also be used for this purpose, either alone or in combination with nanofiltration. Electrodialysis is particularly suitable for the removal of ions from acid whey, because it contains substantial levels of these ions (Burling, 2003). Ion exchange methods, such as anion exchange resins or diafiltration, may also be used in conjunction with nanofiltration to improve separation efficiency (Gernigon, Schuck, Jeantet, & Burling, 2011). Nanofiltration and electrodialysis can also be used to reduce the lactic acid concentration in whey (Kumar et al., 2013).

#### 4.3.1. Nanofiltration

Nanofiltration is an energy-efficient membrane filtration technique used for partial ion removal and concentration of whey in the dairy industry. It has a relatively small system size and requires little system maintenance (Okawa et al., 2015). Nanofiltration can reduce the concentrations of both monovalent and divalent ions in whey, however, a greater concentration reduction is generally observed for monovalent ions (like sodium and potassium) than divalent ones (like calcium) (Chandrapala & Vasiljevic, 2017). Nanofiltration can also be used to reduce the lactic acid content of whey. For instance, research has shown that nanofiltration can reduce the lactic acid content by around 30% and the total monovalent ion (sodium, potassium, and chloride) concentration by around 45 to 60% (Bédas et al., 2017). The same study also reported a better drying efficiency and lower overall energy cost when nanofiltration was used. In another study, nanofiltration was reported to increase the powder yield by reducing the lactic acid and calcium concentrations by around 30% and 40%, respectively (Chandrapala & Vasiljevic, 2018).

Processing conditions such as temperature, pH, and transmembrane pressure also affect the effectiveness of nanofiltration (Kelly & Kelly, 1995). Electrostatic interactions, solute size, and particle surface characteristics have been shown to impact nanofiltration efficiency (González, Alvarez, Riera, & Álvarez, 2008). These parameters depend on pH, ionic composition, and temperature. Consequently, monitoring and understanding the physicochemical properties of solutes in whey under these different conditions is crucial for optimizing nanofiltration processes. Protein aggregation during nanofiltration causes membrane fouling, which decreases filter performance by reducing the transmembrane pressure drop and whey flux (Chandrapala et al., 2015). The aggregation of proteins is promoted by calcium phosphate and calcium lactate complexes in whey, especially as the pH and temperature increase. Since complexes formed by calcium induce protein aggregation and precipitation, filtration at low pH levels can be used to decrease membrane fouling. However, processing at low pH may contribute significantly to Maillard browning which in turn induces caking due to the release of water during browning (Chandrapala et al., 2015).

The dry matter content of whey also affects nanofiltration and product quality. When nanofiltration is performed at cold temperatures (~10 °C), proteins can preserve their native state after filtration (Gernigon et al., 2011). However, as the dry matter content of the whey passing through a nanofiltration device increases, the degree of whey protein denaturation during the evaporation and spray drying operations performed after nanofiltration increases (Marx & Kulozik, 2018). This increase in protein denaturation may be due to the reduced efficiency of nanofiltration at high dry matter contents. Nanofiltration leads to a relatively low ionic strength in the whey stream, which influences the electrostatic interactions and thermal stability of proteins. However, the mineral load of whey may not be reduced sufficiently at high dry matter contents. Hence, high concentrations of cations like calcium after nanofiltration can accelerate the thermal denaturation of β-lactoglobulin during the heat treatments performed in the later stages of WP production (Petit, Herbig, Moreau, & Delaplace, 2011). Excessive thermal denaturation of whey proteins during heat treatments is not desirable in WP production, due to an increased risk of caking (Modler & Lefkovitch, 1986). Therefore, the dry matter content of whey should be carefully monitored before nanofiltration.

#### 4.3.2. Electrodialysis

Electrodialysis is a membrane filtration technique that is based on the separation of charged species by a cascade of anion – cation selective membranes operating at constant electrical potential. Filtration of whey using electrodialysis has been used for many years to produce WP (Greiter, Novalin, Wendland, Kulbe, & Fischer, 2002). Electrodialysis can also decrease the lactic acid content to some extent. Lactic acid is a weak acid that dissociates into its conjugate base (lactate anion) and $H^+$ ions at pH values around and above its $pK_a$ value, which makes it possible for electrodialysis to remove these ions from the whey stream (Wee, Yun, Lee, Zeng, & Ryu, 2005). Researchers have shown that increasing the pH from 4.6 to 6.0 enhanced the removal of lactate ions from whey using a continuous electrodialysis operation (Chen et al., 2016). This separation was carried out at a relatively high temperature (45 °C) to reduce the solution viscosity and membrane resistance. Electrodialysis does not separate uncharged lactose molecules which causes problems in the later unit operations (Chen et al., 2016). Consequently, it is important to carefully control the pH during electrodialysis to effectively remove the lactic acid (Galama et al., 2014).

The effectiveness of filtration of whey by electrodialysis depends on ion type. For instance, smaller ions are preferentially removed from the whey stream by electrodialysis. Therefore, lactate and phosphate anions (larger ions) are removed after the removal of the majority of chloride anions (smaller ions) (Bouchoux, Roux-de Balmann, & Lutin, 2006). Furthermore, electrodialysis removes monovalent ions (sodium, potassium) faster than divalent ions (calcium, magnesium). Monovalent ions typically have a lower ionic radius and greater mobility than divalent ions (Van Der Bruggen, Koninckx, & Vandecasteele, 2004), which leads to their faster removal (Bazinet et al., 2000).

#### 4.3.3. Combinations of filtration techniques

Filtration and ion-removal techniques can be used in combination to improve the efficiency of filtration process during WP production. For instance, nanofiltration and anion exchange treatment have been combined for this purpose (Kumar et al., 2013). Nanofiltration maintains the electroneutrality of the solution by passing cations (sodium, potassium) in pairs with anions (chloride) through the membranes (van der Horst, Timmer, Robbertsen, & Leenders, 1995). However, since the total sodium and potassium content in whey is generally higher than the chloride content, the ion removal efficiency of nanofiltration is reduced. To increase the chloride content, a chloride-form anion exchange resin can be used prior to nanofiltration. Utilization of this approach has been shown to increase cation removal and overall ion removal rates by around 90% and 70%, respectively (Okawa et al., 2015). In addition, the concentrations of phosphate and citrate ions in the whey were also reduced by the chloride resin.

Nanofiltration and electrodialysis can also be used together to improve the effectiveness of the separation process. Nanofiltration can reduce the energy consumption of electrodialysis by decreasing the monovalent ion load in the whey (Greiter et al., 2002). Additionally, electrodialysis has been used to remove lactate from whey after nanofiltration (Chen et al., 2016). The resulting WP has better quality due to a substantial reduction in both the ion and lactic acid contents.

### 4.4. Evaporation

The filtrated whey stream is concentrated before lactose precrystallization and spray drying operations by an evaporation process with multiple effect falling film vacuum evaporators. The total solids content of whey is typically around 15 to 20% (w/v) after membrane filtration and around 55 to 60% (w/w) after evaporation (Jelen, 2009). The performance and energy consumption of evaporation depend on the efficiency of the filtration step. If the mineral content of whey fed into the evaporator is too high, then fouling of the evaporator surfaces can occur (Bédas et al., 2017). Fouling decreases the efficiency of heat transfer from the evaporator to the whey, which may lead to prolonged heating times and poor process economics (Modler & Lefkovitch, 1986). Moreover, prolonged heating at high temperatures increases the probability of whey protein denaturation. One of the most common mineral complexes that causes fouling in evaporators is calcium phosphate (Chandrapala et al., 2015). Casein has a stabilizing effect on calcium phosphate, but it is removed from whey stream in the beginning of WP production. Therefore, the tendency for calcium phosphate to promote precipitation in whey increases. Precipitation of calcium phosphate is also promoted at high concentrations, high temperatures, and under

alkaline conditions (Paterson, 2017). Researchers have therefore examined potential strategies for reducing calcium phosphate in whey, *e.g.,* by promoting its precipitation using controlled pH and temperature conditions prior to filtration and evaporation (Rice et al., 2006). However, this procedure should be performed carefully to avoid losing or damaging other valuable components in whey (Smithers & Augustin, 2013).

The presence of lactic acid is another problem during the evaporation and concentration of whey. Some of the lactic acid is removed during membrane filtration, but there may still be a considerable amount remaining in the whey that is fed into the evaporator. Since lactic acid is a relatively nonvolatile substance, drying cannot easily remove it from the whey concentrate (Jelen, 2009). Normally, evaporation increases the degree of lactose supersaturation, which is an important factor required for proper lactose crystallization (Pereira et al., 2020). However, if the lactic acid content of the whey is too high, then the concentrates produced during evaporation become too viscous and the subsequent process operations (pre-crystallization and spray drying) are negatively affected (Jelen, 2009). Due to their higher lactic acid contents, acid whey samples are usually more difficult to evaporate than sweet wheys. In particular, the evaporation concentrates produced from acid whey have slower lactose crystallization kinetics than those produced from sweet whey concentrates. Thus, acid whey has a lower crystallization rate and higher crystal size dispersion during pre-crystallization (Modler & Lefkovitch, 1986). After pre-crystallization, acid whey samples tend to exhibit more hygroscopic behavior than sweet whey samples during spray drying, which results in stickier WPs that are more prone to caking (Bédas et al., 2017). Lastly, the monosaccharide content of whey may also affect the evaporation and subsequent unit operations. Galactose has been reported to hinder the concentration of whey by evaporation (Modler & Lefkovitch, 1986). The presence of high galactose concentrations would therefore slow down the crystallization rate due to the lower degree of lactose supersaturation, thereby leading to products that were more prone to caking.

### 4.5. Lactose pre-crystallization

Lactose pre-crystallization is performed to decrease the amorphous lactose content of whey before spray drying. If this process is not carried out, the final WP would have inferior quality. For instance, the resulting powder would be highly hygroscopic and contain a high amorphous-to-crystalline lactose ratio, which would lead to caking of WP during storage (Aguilera et al., 1995). Industrially, lactose pre-crystallization is usually achieved by subjecting the whey concentrate to a controlled cooling regime using a cooling tank (Carpin et al., 2016). This cooling regime involves an initial rapid cooling period to around 30 °C and then a slow cooling period to around 15 °C. Fast cooling increases the driving force for lactose crystal nucleation, while slow cooling promotes crystal growth (Jelen, 2009). The particle size of the crystals produced can be manipulated by making small changes in the cooling regime. For instance, smaller crystals are obtained if rapid cooling is continued to lower temperatures. However, it is also possible to produce bigger lactose crystals when the slow cooling regime is extended (McLeod et al., 2011). Typically, a small lactose crystal size is preferred during the pre-crystallization stage of WP production (Jelen, 2009).

Various other factors also influence the crystallization behavior of lactose, including the viscosity, temperature, pH, and type of whey used, the presence of minerals, acids and other impurities, the type of nucleation (primary or secondary), the final cooling temperature, the degree of lactose supersaturation, and the design of the crystallizer as summarized in Fig. 4 (Hourigan, Lifran, Vu, Listiohadi, & Sleigh, 2013). In the remainder of this section, an overview of these different factors is given.

#### 4.5.1. Lactose supersaturation

Supersaturation of a solution is required to start crystal nucleation (Parimaladevi & Srinivasan, 2015). However, the degree of supersaturation required depends on the metastable zone for the material being crystallized. The metastable zone is the region between the saturation and super-supersaturation of a material and depends on solute type and temperature (Zhang, Sun, Zhu, & Cheng, 2015). Since lactose has a wide metastable zone, a high supersaturation level is needed to induce crystal nucleation. If the driving force for nucleation is low, the crystal induction would take a long time and the crystallization process would be slow which leads to inefficient process economy (Zamanipoor & Mancera, 2014). Despite the requirement of high supersaturation for lactose crystallization, excessive supersaturation is not desired since the mobility of lactose molecules may be hindered. At excessive supersaturation levels, solution viscosity increases so much that the diffusion of lactose molecules is hindered and crystal growth is inhibited (Y. Shi, Hartel, & Liang, 1989). A reduction in the cooling temperature below around 20 °C also inhibits crystal growth. Lower temperatures increase the degree of supersaturation, but they also increase solution viscosity, which reduces the molecular mobility and therefore the rate of crystal growth (Whittier & Gould, 1930). Therefore, the degree of supersaturation should be carefully controlled after the nucleation step to ensure proper crystal growth (Wong & Hartel, 2014).

#### 4.5.2. Crystal nucleation

After lactose supersaturation has been achieved in whey, its crystallization can be spontaneously initiated by decreasing the temperature, leading to homogeneous nucleation (Martini, Herrera, & Hartel,

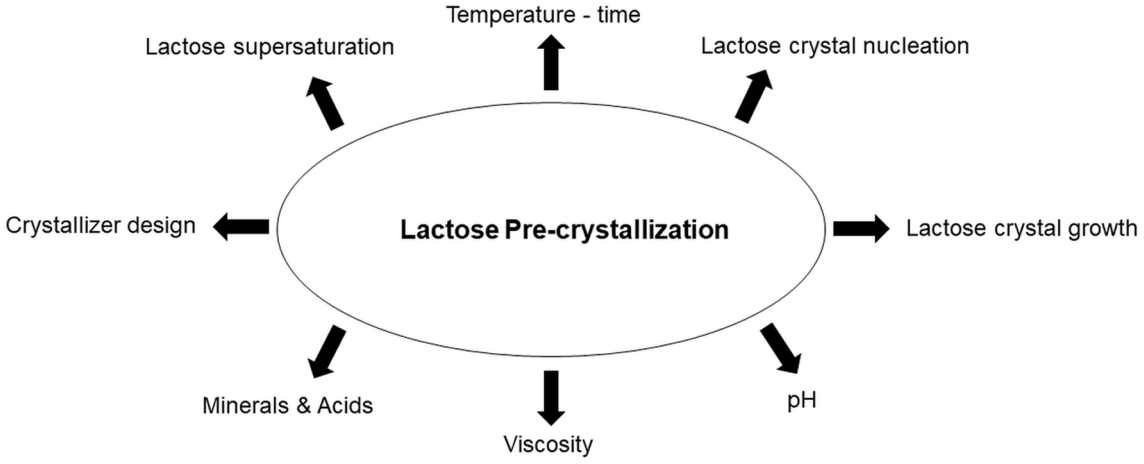


**Fig. 4.** Summary of factors affecting the lactose pre-crystallization process.

2001). In the presence of impurities, lactose molecules in a saturated solution may attach to the surfaces of the impurities, which promotes primary heterogeneous nucleation because the free energy required to form a new nucleus is diminished (McLeod et al., 2011). In addition to non-crystalline impurities, preexisting crystals can also act as heterogeneous nucleation sites (seeds) and promote secondary heterogeneous nucleation. Thus, small amounts of lactose crystals can be added to whey concentrates to initiate this kind of nucleation, but this is a rarely used crystallization technique in the dairy industry (Jones, 2002). Besides seeding, broken crystals formed by agitation of the crystallizer can also act as nucleation sites and promote secondary heterogeneous nucleation (Myerson & Ginde, 2002). Heterogeneous nucleation mechanisms require lower levels of supersaturation than homogeneous nucleation ones.

Cooling parameters and the nucleation mechanism should be carefully manipulated during lactose pre-crystallization to control the number and size distribution of the lactose crystals produced. It has been reported that secondary heterogeneous nucleation results in a broader crystal size distribution than primary heterogeneous nucleation promoted by whey proteins (Zamanipoor & Mancera, 2014). In another study, it was reported that in the absence of secondary nuclei, the crystallization of whey concentrate mainly occurred due to homogeneous nucleation (Perrone et al., 2017).

#### 4.5.3. Lactose anomers and mutarotation

Lactose has a lower solubility than most other sugars including glucose, fructose, and sucrose (Brito & Giulietti, 2007). Lactose exists in two anomeric forms (Fig. 5), α- and β-lactose, which have different solubility characteristics. The solubility of these two anomers is temperature dependent, with α-lactose being more soluble than β-lactose at temperatures exceeding 93.5 °C, and the opposite being true at lower temperature. For instance, the water-solubilities are 70 and 500 g/L at 20 °C for α-and β-lactose, respectively. As a result, α-lactose tends to convert to β-lactose when aqueous lactose solutions are cooled due to mutarotation, until an appropriate equilibrium between the two anomeric forms is reached at the holding temperature (Fox, 2009). Consequently, cooling of whey in crystallization tanks mainly produces α-lactose crystals in a monohydrate form. The formation of β-lactose crystals is only possible at elevated temperatures (>93.5 °C) and they tend to crystallize in the anhydrate form (Walstra, Wouters, & Geurts, 2006).

The crystal structure of α-lactose monohydrate includes one water molecule per lactose molecule, with the water molecule linking the oxygens on the four lactose molecules. These interactions lead to a high stability in the α-lactose monohydrate crystals (Clydesdale, Roberts, Telfer, & Grant, 1997). The presence of the water molecule also imparts a non-hygroscopic character to α-lactose monohydrates. Thus, α-lactose monohydrate is stable below 95% RH at 25 °C (Salameh, Mauer, & Taylor, 2006). Accordingly, α-lactose monohydrate is the most stable anomeric form of lactose under cooling conditions of industrial lactose pre-crystallization process (Walstra & Jenness, 1984). Due to their high stability, the dairy industry is working to identify pre-crystallization conditions that produce a higher percentage of α-lactose monohydrate crystals (Hourigan et al., 2013). A higher ratio of α-lactose monohydrate crystals after pre-crystallization is typically associated with better powder quality, including less caking and improved storage stability (Gänzle, Haase, & Jelen, 2008).

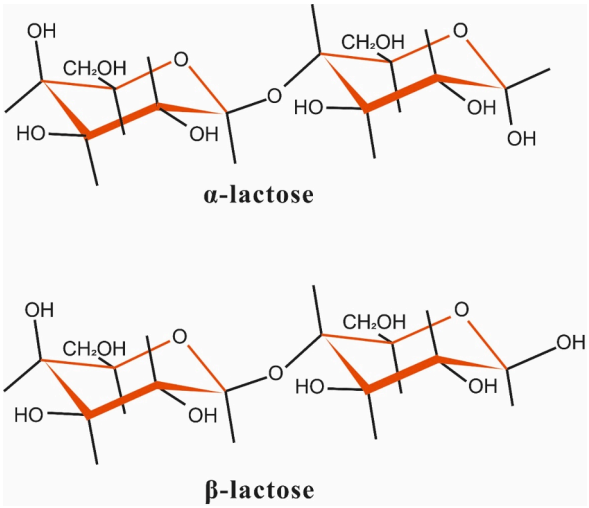


**Fig. 5.** Molecular structures of α- and β-lactose anomers.

After formation, lactose crystals may undergo transformation between the α- and β-anomers due to mutarotation. The rate of mutarotation and the types and concentrations of lactose anomers formed depend on pH, temperature, whey composition, and processing conditions (Holsinger, 1997). When α-lactose monohydrate loses the water of crystallization from its crystal lattice, one of the anhydrous forms of α-lactose (stable or unstable anhydrous α-lactose) are formed. The unstable anhydrous α-lactose is highly hygroscopic even at low RH and temperature conditions *e.g.,* 10% RH and 20 °C. The porous structure of this anomeric form could be the origin of its undesired hygroscopic character (Listiohadi et al., 2005). However, the stable form of anhydrous α-lactose shows an intermediate stability (stable below 50% RH at 20–22 °C) between the unstable anhydrous and the monohydrate forms of α-lactose. Anhydrous forms can absorb water and reform α-lactose monohydrate under suitable conditions (Figura & Epple, 1995). On the other hand, the only form of β-lactose anomer is anhydrous β-lactose, which has a high stability (stable below 95% RH at 25 °C) like α-lactose monohydrate. However, preferential crystallization of lactose solutions in this form is only possible above 93.5 °C which is not commercially viable for the lactose pre-crystallization process (Gänzle et al., 2008).

The controlled gradual cooling used in the lactose pre-crystallization step is designed to provide a sufficient degree of lactose crystallization (>70%) to ensure the production of high-quality WP (Simeão et al., 2018). Application of just flash cooling or slow cooling during the whole crystallization period may result in poor process economics due to the prolonged process time and/or insufficient degree of lactose crystallization (Jelen, 2009). The presence of β-lactose molecules in concentrated whey is also undesirable since the crystal morphology of α-lactose crystals is affected by β-lactose (Dincer, Parkinson, Rohl, & Ogden, 1999). β-lactose molecules act as an impurity and incorporate non-uniformly into the growing interfaces of α-lactose monohydrate crystals leading to crystals with a tomahawk shape. This inhibits the growth of the α-lactose monohydrate crystals and the morphology of the crystals produced becomes asymmetric (Michaels & van Kreveld, 1966). The resulting strain in the crystals created by the inclusion of β-lactose reduces the growth rate of the crystals and leads to a broader crystal size distribution (Raghavan et al., 2000). In addition to their growth limiting effect, the presence of large quantities of β-lactose in whey concentrate may also inhibit the initial crystal nucleation step. Under these conditions, an excessively high degree of lactose supersaturation is required to initiate crystal nucleation. The presence of β-lactose is unavoidable in whey but its effects should be kept to a minimum by adjusting the operation conditions before the pre-crystallization process so as to reduce its nucleation and growth inhibition effects (Raghavan, Ristic, Sheen, & Sherwood, 2001).

#### 4.5.4. Presence of acids, minerals, and other impurities

Acid whey generally have a high acidity (higher lactic acid content) and higher mineral content than sweet whey leading to poorer lactose crystallization and stickier powders. One of the main components retarding lactose crystallization is lactic acid, which can be found in both acid and sweet wheys at different levels (Jelen & Coulter, 1973). Excessive lactic acid concentrations create a hydration layer around the lactose molecules, which increases their solubility and inhibits their

crystallization (Wijayasinghe et al., 2015). A lower pH also promotes interactions between the hydration layer and lactose molecules, which further reduces the tendency for crystallization to occur. A lactic acid concentration of 1% (w/w) in a model lactose system was reported to have negative impacts on lactose crystallization by decreasing the lactose crystal yield and increasing the crystal size (Chandrapala, Wijayasinghe, & Vasiljevic, 2016).

Some salts speed up lactose crystallization when present at low concentrations (such as calcium chloride and magnesium sulfate), but most salts retard lactose crystallization (such as calcium and dipotassium phosphate) (Bhargava & Jelen, 1996). Other substances in whey can also affect the crystallization behavior of lactose, including potassium chloride, calcium phosphate, lactose phosphate, and riboflavin. While riboflavin may deposit on lactose crystals, impurities like calcium phosphate and lactose phosphate may be embedded into the crystal lattice. These embedded impurities inhibit both crystal nucleation and growth (Pandalaneni & Amamcharla, 2018). Calcium phosphate deposition onto lactose crystal surfaces has been observed during alkalized acid whey processing, which was attributed to the reduction in calcium solubility at higher pH values (pH 6.5 to 7.0) (Drapala et al., 2018). The rate of cooling during crystallization impacts the number of impurities incorporated into the lactose crystals. Prolonged slow cooling restricts the diffusion of lactose molecules to crystal surfaces in the solution, which leads to a higher degree of impurities being embedded in the crystals (Lifran, Vu, Durham, Hourigan, & Sleigh, 2007). Calcium cations can also be incorporated into lactose crystals and inhibit their growth (Elmonsef Omar & Roos, 2007).

The presence of both lactic acid and minerals in whey influences lactose crystallization by an amount that depends on their concentrations. For instance, high concentrations of both lactic acid and calcium have been reported to retard lactose crystallization and lead to a bimodal crystal size distribution, whereas a moderate calcium concentration and high lactic acid concentration was reported to promote lactose crystallization (Chandrapala et al., 2016). The reason for this phenomenon could be the water structuring effects of the calcium ions. Calcium may have changed the hydrogen bonding patterns of the lactose, thereby changing its crystallization behavior (Mähler & Persson, 2012). The hydrogen bonding patterns in lactose solutions, as detected by Fourier transform infrared (FTIR) spectroscopy, have been reported to depend on the level of calcium ions present (Chandrapala et al., 2016). The results of this study demonstrate that the crystallization behavior of lactose can be manipulated by changing the concentrations of lactic acid and calcium.

Despite the lactose crystallization retarding effect of many salts, sodium citrate has recently been reported to improve the lactose crystallization of concentrated whey (Pereira et al., 2020). It has also been reported to increase the rate of lactose crystallization in model aqueous solutions (Gernigon et al., 2013). This effect may be due to the catalytic effects of sodium citrate on the mutarotation of lactose molecules, which improved the lactose crystallization behavior (Patel & Nickerson, 1970). The addition of sodium citrate to concentrated whey was reported not to significantly alter the pH of the solution, and so its ability to promote lactose crystallization does not appear to be due to its impact on pH (Pereira et al., 2020). The same authors also showed that sodium citrate addition decreased the average size of the crystals formed.

The protein concentration of whey may also affect lactose crystallization. Due to their predominantly hydrophilic properties, whey proteins generally increase the lactose crystal nucleation rate by creating local supersaturation spots (Mimouni, Schuck, & Bouhallab, 2005; Pandalaneni & Amamcharla, 2018). Whey proteins have also been reported to act as nuclei centers for the heterogeneous nucleation of lactose crystals (Sánchez-García, García-Vega, Leal-Ramos, Salmeron, & Gutiérrez-Méndez, 2018). The impact of whey proteins on lactose crystallization (such as crystal number, size, and polydispersity) also depends on pH. The electrical charge, quaternary structure, solubility, and hydration of whey proteins depend on pH (Sawyer, 2013). Researchers have reported that a higher inclusion of whey proteins into the lactose crystal lattice and a greater number of lactose crystals were formed under neutral conditions than under acidic ones (Sánchez-García, Gutiérrez-Méndez, Orozco-Mena, Ramos-Sánchez, & Leal-Ramos, 2019). The main reason that whey proteins can promote more lactose crystallization at higher pH values was attributed to their higher solubility under these conditions. The charge, conformation, and aggregation state of β-Lg, the major protein in whey, depend on pH, thereby influencing its solubility characteristics (O'Mahony & Fox, 2013). β-Lg tends to form neutral octamers around its isoelectric point (pH 5.2–5.4), which reduces its solubility. In contrast, it forms negatively charged dimers under neutral conditions, which increases its solubility (Sawyer, 2013). Consequently, whey proteins can interact with water under neutral conditions, which create local supersaturation spots for the lactose molecules (Bhargava & Jelen, 1996). Furthermore, whey proteins can migrate more freely due to their smaller molecular dimensions at pH 7, and act as nucleation centers for lactose crystallization, also increasing the amount of protein incorporated into the crystals (Das & Langrish, 2012). Generally, the rate and extent of lactose crystallization increases with increasing soluble whey solids concentration (Perrone et al., 2017). Although whey proteins are associated with increased crystal nucleation rates, some authors claim that whey proteins retard the growth of lactose crystals (Huppertz & Gazi, 2016). However, slow growth of lactose molecules could be an advantage since smaller crystals are preferred in spray drying. In this way, whey proteins also contribute to the narrow crystal size distribution after crystallization especially at higher pH values (Zamanipoor & Mancera, 2014).

#### 4.5.5. Crystallizer design

The design of the crystallization device may also impact the nature of the lactose crystals formed. For instance, the stirring position within the crystallization vat has been shown to alter the crystallization behavior of lactose in whey concentrates. It has been reported that the rate of lactose crystallization was higher for central stirring than for lateral stirring at the same stirring rate, soluble solids, and cooling rate (Simeão et al., 2018). These authors also reported that the dissolved sugar concentration of the whey concentrates influenced the crystallization efficiency. The promotion of lactose crystallization by central stirring was more pronounced at low and moderate dissolved sugar concentrations (50 and 55 °Brix), with smaller and more numerous crystals being generated. However, the number and size of the crystals produced by both central and lateral stirring were similar at higher sugar concentrations (60 °Brix) with an average size of around 65$\mu$ m. When central stirring was carried out at low and moderate sugar concentrations (50 and 55 °Brix), fewer deposits were formed within the crystallization vat, which improved the drying ability of the crystallized whey (Carpin et al., 2016).

### 4.6. Spray drying

The final step in producing WP is spray drying, which also has an important effect on WP yield and quality. Consequently, it is important to understand and control the product formulation and processing parameters throughout the spray drying process. Table 2 summarizes some recent whey-based formulations – applications and their respective spray drying process parameters. A major challenge in spray drying is the loss of powder yield (Jelen, 2009). If the lactic acid and galactose contents in the whey fed into the spray dryer is too high, then the particles will stick to the equipment surfaces, thereby decreasing the powder yield. Moreover, if the amorphous lactose content of the whey that is spray dried is high, particles in the final powder will tend to form clumps during storage, leading to caking. All of these effects have detrimental effects on the overall powder quality and shelf life (Aguilera et al., 1995). However, powder quality can be improved by carrying out an appropriate pre-crystallization step before spray drying. Thickening of the whey concentrate due to thermal protein denaturation is another

**Table 2**
Drying conditions of some whey-based applications.

| Whey Sample | Added Materials – Drying Parameters | | Reference |
|---|---|---|---|
| | Ingredients | Spray Drying | |
| **Acid Whey** | Millet powders | 120 °C (air inlet), 160 °C (air outlet) | (Malik, Krishnaswamy, & Mustapha, 2021) |
| | – | 140 °C (air inlet), 70 °C (air outlet) | (Bédas et al., 2017) |
| **Whey Powder** | Chitosan | 170 °C (air inlet), 90 °C (air outlet), 17 rpm (feed flow rate) | (Lekshmi et al., 2019) |
| | *L. acidophilus*, gum arabic | 140 °C (air inlet), 0.485 L/h (feed flow rate) | (Leylak, Özdemir, Gurakan, & Ogel, 2021) |
| **Whey Conc.** | Spent coffee ground | 170 °C (air inlet), 90 °C (air outlet), 2 L/h (feed flow rate), 540 L/h (air flow rate) | (Osorio-Arias et al., 2020) |
| **WPC** | Maltodextrin, grape juice | 140 °C (air inlet), 2 mL/min (feed flow rate), 500 L/h (air flow rate) | (Moser, De Souza, & Nicoletti Telis, 2017) |
| | Grape-skin pulp | 190 °C (air inlet), 10 mL/min (feed flow rate), 245 kPa (atomization pressure) | (Oliveira et al., 2018) |
| | Ghee flavor extract, guar gum | 160 °C (air inlet), 45 – 55 °C (air outlet), 5 mL/min (feed flow rate), 70 $Nm^3$/h (air flow rate), 1.55 $kg/cm^2$ (atomization pressure) | (Duhan, Sahu, Mohapatra, & Naik, 2021) |
| | Phenolic extracts, maltodextrin, gum arabic | 180 °C (air inlet), 78.5 °C (air outlet), 1.12 L/h (feed flow rate), 1.7 $m^3$/min (air flow rate), | (de Rocha, 2019) |
| **WPI** | Maltodextrin, eugenol | 180 °C (air inlet), 80 °C (air outlet), 20 mL/min (feed flow rate), 1.9 bar (atomization pressure) | (Talón et al., 2019) |
| | Inulin, beetroot juice | 130, 150, 170 °C (air inlet), 85, 95, 109 °C (air outlet), 0.8 L/h (feed flow rate), 35 L/min (air flow rate) | (do Carmo et al., 2019) |
| | Maltodextrin, soybean oil | 135 °C (air inlet), 70 °C (air outlet), 10.5 mL/min (feed flow rate), 0.667 $m^3$/h (air flow rate) | (Li, Pan, Ma, Miao, & Ji, 2020) |
| | Curcumin | 150 – 110 °C (air inlet) | (Liu, Chen, Cheng, & Selomulya, 2016) |
| | Tributyrin | 140 °C (air inlet), 5 mL/min (feed flow rate) | (Shi & Lee, 2020) |
| | β-cyclodextrin, vanillin | 110 °C (air inlet), 60 °C (air outlet), 20 mL/min (feed flow rate), 24 psi (atomization pressure) | (Hundre, Karthik, & Anandharamakrishnan, 2015) |
| | Peppermint oil, carboxymethyl cellulose | 170 °C (air inlet), 70 °C (air outlet), 15 – 20 mL/min (feed flow rate), 300 NL/min (air flow rate) | (Bakry et al., 2016) |

* Whey conc.: Whey concentrated, WPC: Whey protein concentrate, WPI: Whey protein isolate.

common difficulty encountered during spray drying (Jelen, 2009). These negative effects can be reduced by controlling the lactic acid concentration and thermal history of the whey.

Amorphous lactose materials undergo a phase transition from a glassy to a rubbery state at the $T_g$. Exceeding $T_g$ increases the molecular mobility and lowers the surface viscosity of lactose particles, thereby increasing their stickiness (Foster, Bronlund, & Paterson, 2006). The $T_g$ decreases with increasing RH due to the plasticizing effects of water, thereby increasing the tendency for lactose particles to become sticky (Kamrul Haque & Roos, 2004). In addition to the $T_g$ value, the sticking point temperature ($T_s$) of WP should also be considered (Jouppila & Roos, 1994). $T_s$ represents the temperature at which sticking of particles begins due to the reduction in their surface viscosity (Roos & Karel, 1991). The temperature difference between $T_s$ and $T_g$ ($T_s - T_g$) depends on many factors such as drying time, powder composition, and particle size (Hogan, O'Callaghan, & Bloore, 2009; Karel et al., 1994). For instance, it has been reported that $T_s - T_g$ increased at higher whey protein concentrations, which reduced particle stickiness (Hogan & O'Callaghan, 2010). The whey proteins probably preferentially absorbed some of the water molecules, thereby reducing the number available for plasticization of the amorphous lactose particles (Shrestha, Howes, Adhikari, Wood, & Bhandari, 2007). Other researchers have claimed that reaching temperatures exceeding $T_g$ by 25 °C during spray drying of amorphous lactose induces instantaneous sticking, thereby decreasing powder yield significantly. Even temperatures just a few degrees above $T_g$ have been reported to cause caking of whey powders during storage (Paterson, Brooks, Bronlund, & Foster, 2005).

The pH of whey concentrates also affects the spray drying process. Since lactic acid is mostly in its undissociated (neutral) form at low pH values (<4.0), it acts as a plasticizer that promotes sticking and agglomeration of the lactose particles during spray drying (Shrestha et al., 2006). Therefore, the lactic acid concentration should be reduced as much as possible before spray drying to prevent the $T_g$ lowering effect of lactic acid (Maltini, Anese, & Shtylla, 1997). Furthermore, the increased inter-particle adhesion caused by the thermoplastic effects of undissociated lactic acid molecules has been reported to promote the formation of a bimodal crystal size distribution containing a high proportion of large particles (Chandrapala & Vasiljevic, 2017). Higher pH conditions (pH > 7.0) are also undesirable for spray drying. For instance, calcium can form complexes with phosphates and lactates under these pH conditions and induce whey protein complexation, leading to particle agglomeration (Chandrapala et al., 2015). Consequently, removal of water from agglomerates is hindered due to the limitations in the heat – mass transfer and an extensive amount of particles adhere to the dryer surfaces, thereby decreasing the powder yield substantially (Chandrapala & Vasiljevic, 2018). Thus, mineral load should be reduced by the membrane filtration process prior to spray drying (Bédas et al., 2017).

The design of a spray drying unit also influences the efficiency of the drying operation and the quality of the powder produced. The atomization of the concentrate and the distribution of the hot air within the spray drying unit are important factors affecting WP production (Smithers & Augustin, 2013). It is important the atomized droplets rapidly come into direct contact with the drying air. To achieve this goal, most spray drying systems have the air disperser on the top of the dryer and the atomizer is placed in the middle of this air disperser (Westergaard, 2003). Atomizing the concentrate increases its specific surface area, thereby increasing the drying rate. Atomization also influences the final size, shape, and density of the powder particles produced. Typically, the particles produced by spray drying whey concentrates have diameters around 10 to 250$\mu$ m (Písecký, 2005). The viscosity and homogeneity of the whey concentrate, as well as the atomizing pressure, determine the particle size formed after atomization. A low concentrate viscosity and high atomizing pressure tend to decrease the particle size, while a high concentrate viscosity and low atomizing pressure tend to increase it. Moreover, inhomogeneous whey concentrates tend to lead to powders with broad particle size distributions (Schuck et al., 2005). The relatively short drying times associated with spray drying tend to preserve the native state of the whey proteins by minimizing thermal denaturation (Refstrup, 2003).

## 5. Conclusions and future directions

WP production is a complex process in which many factors need to be controlled to obtain a final product with the desired quality and shelf life. The most challenging problem encountered during WP production and storage is caking. The presence of amorphous lactose in whey concentrates is mainly responsible for caking. Amorphous lactose undergoes a transition from a brittle glassy state to a rubbery state above the $T_g$, which increases the stickiness of the powder particles, thereby leading to adhesion to the surfaces of processing equipment and caking in the powder. The $T_g$ decreases with increasing RH, and so it is important to control both the temperature and RH of samples during processing and storage. The sticking of powder particles to the surfaces of drying equipment reduces the product yield, leading to lost revenue and increased waste. The sticking of powder particles during storage leads to caking, which reduces the quality and shelf life of the product. In this article, it has been shown how various kinds of processing operations can be used to reduce the level of amorphous lactose in WP, thereby reducing their tendency to undergo caking. For instance, minerals and lactic acid can be reduced using various kinds of filtration and electrodialysis methods. In addition, pre-crystallization can be carried out before spray drying to generate crystalline (rather than amorphous) lactose. However, the increase in whey viscosity during lactose pre-crystallization may decrease the efficiency of the process. In order to overcome this problem, the initial composition and the efficiency of filtration process should be carefully controlled. Additionally, improvement of the design of lactose crystallization equipments will be of great interest in the future. There are already some studies showing the positive effects of changes in lactose crystallization equipment design on the final WP quality. Further investigation on this matter is required. Overall, it is important to optimize the initial composition of the whey used, as well as the various processing operations employed, to obtain high quality whey powders. This article has focused on the production of dried powders from whey but much of the knowledge gained in this area over the past few decades may also be useful to produce powders from plant proteins, which is a rapidly growing field of study. In this way, a variety of value-added foods meeting consumer demands for nutritious products could be produced in a sustainable manner.

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## Credit authorship contribution statement

**Baris Ozel:** Conceptualization, Writing – original draft. **David Julian McClements:** Writing – review & editing. **Cagatay Arikan:** Resources. **Ozlem Kaner:** Resources. **Mecit Halil Oztop:** Conceptualization, Writing – review & editing, Supervision.

## Declaration of Competing Interest

The authors declare that they have no known competing financial interests or personal relationships that could have appeared to influence the work reported in this paper.

## References

Abella, M., Leano, M. L., Malig, J., Martin, G., Dela Cruz, C., & De Leon, A. (2016). Formulation of a sports drink from fermented whey. *CLSU International Journal of Science & Technology, 1*(1), 1–10. https://doi.org/10.22137/ijst.2016.v1n1.01

Aguilera, J. M., del Valle, J. M., & Karel, M. (1995). Caking phenomena in amorphous food powders. *Trends in Food Science & Technology, 6*(5), 149–155.

Akib, M. A., & Setiawati, H. (2017). Fermentation of whey waste as organic liquid fertilizer "Pucafu". *Agrotech Journal, 2*(2), 7–13. https://doi.org/10.31327/atj.v2i2.277

Alsaed, A. K., Ahmad, R., Aldoomy, H., El-Qader, S. A., Saleh, D., Sakejha, H., & Mustafa, L. (2013). Characterization, concentration and utilization of sweet and acid whey. *Pakistan Journal of Nutrition, 12*(2), 172–177. https://doi.org/10.3923/pjn.2013.172.177

Bakry, A. M., Fang, Z., Ni, Y., Cheng, H., Chen, Y. Q., & Liang, L. (2016). Stability of tuna oil and tuna oil/peppermint oil blend microencapsulated using whey protein isolate in combination with carboxymethyl cellulose or pullulan. *Food Hydrocolloids, 60*, 559–571. https://doi.org/10.1016/j.foodhyd.2016.04.026

Bazinet, L., Ippersiel, D., Gendron, C., Beaudry, J., Mahdavi, B., Amiot, J., et al. (2000). Cationic balance in skim milk during bipolar membrane electroacidification. *Journal of Membrane Science, 173*(2), 201–209. https://doi.org/10.1016/S0376-7388(00)00373-2

Bédas, M., Tanguy, G., Dolivet, A., Méjean, S., Gaucheron, F., Garric, G., et al. (2017). Nanofiltration of lactic acid whey prior to spray drying: Scaling up to a semi-industrial scale. *LWT - Food Science and Technology, 79*, 355–360. https://doi.org/10.1016/j.lwt.2017.01.061

Berggren, J., & Alderborn, G. (2004). Long-term stabilisation potential of poly (vinylpyrrolidone) for amorphous lactose in spray-dried composites. *European Journal of Pharmaceutical Sciences, 21*(2–3), 209–215. https://doi.org/10.1016/j.ejps.2003.10.005

Bhargava, A., & Jelen, P. (1996). Lactose solubility and crystal growth as affected by mineral impurities. *Journal of Food Science, 61*(1), 180–184. https://doi.org/10.1111/j.1365-2621.1996.tb14754.x

Bouchoux, A., Roux-de Balmann, H., & Lutin, F. (2006). Investigation of nanofiltration as a purification step for lactic acid production processes based on conventional and bipolar electrodialysis operations. *Separation and Purification Technology, 52*(2), 266–273. https://doi.org/10.1016/j.seppur.2006.05.011

Briggner, L. E., Buckton, G., Bystrom, K., & Darcy, P. (1994). The use of isothermal microcalorimetry in the study of changes in crystallinity induced during the processing of powders. *International Journal of Pharmaceutics, 105*(2), 125–135. https://doi.org/10.1016/0378-5173(94)90458-8

Brito, A. B. N., & Giulietti, M. (2007). Study of lactose crystallization in water-acetone solutions. *Crystal Research and Technology, 42*(6), 583–588. https://doi.org/10.1002/crat.200610867

Burling, H. (2003). Demineralization. In H. Roginski, J. W. Fuquay, & P. F. Fox (Eds.), *Encyclopedia of dairy sciences* (pp. 2745–2751). Academic Press.

Burnett, D. J., Thielmann, F., Sokoloski, T., & Brum, J. (2006). Investigating the moisture-induced crystallization kinetics of spray-dried lactose. *International Journal of Pharmaceutics, 313*(1–2), 23–28. https://doi.org/10.1016/j.ijpharm.2006.01.012

Carpin, M., Bertelsen, H., Bech, J. K., Jeantet, R., Risbo, J., & Schuck, P. (2016). Caking of lactose: A critical review. *Trends in Food Science and Technology, 53*(2016), 1–12. https://doi.org/10.1016/j.tifs.2016.04.002

Chandrapala, J., Duke, M. C., Gray, S. R., Zisu, B., Weeks, M., Palmer, M., & Vasiljevic, T. (2015). Properties of acid whey as a function of pH and temperature. *Journal of Dairy Science, 98*(7), 4352–4363. https://doi.org/10.3168/jds.2015-9435

Chandrapala, J., Gauthier, C., & Vasiljevic, T. (2017). Behaviour of lactose with the presence of lactic acid and Ca as affected by pH. *Journal of Dairy Research, 84*(4), 484–487. https://doi.org/10.1017/S0022029917000553

Chandrapala, J., & Vasiljevic, T. (2017). Properties of spray dried lactose powders influenced by presence of lactic acid and calcium. *Journal of Food Engineering, 198*, 63–71. https://doi.org/10.1016/j.jfoodeng.2016.11.017

Chandrapala, J., & Vasiljevic, T. (2018). Feasibility of spray drying concentrated acid whey after nanofiltration. *Food and Bioprocess Technology, 11*(8), 1505–1515. https://doi.org/10.1007/s11947-018-2118-1

Chandrapala, J., Wijayasinghe, R., & Vasiljevic, T. (2016). Lactose crystallization as affected by presence of lactic acid and calcium in model lactose systems. *Journal of Food Engineering, 178*, 181–189. https://doi.org/10.1016/j.jfoodeng.2016.01.019

Chatzipaschali, A. A., & Stamatis, A. G. (2012). Biotechnological utilization with a focus on anaerobic treatment of cheese whey: Current status and prospects. *Energies, 5*(9), 3492–3525. https://doi.org/10.3390/en5093492

Chen, G. Q., Eschbach, F. I. I., Weeks, M., Gras, S. L., & Kentish, S. E. (2016). Removal of lactic acid from acid whey using electrodialysis. *Separation and Purification Technology, 158*, 230–237. https://doi.org/10.1016/j.seppur.2015.12.016

Chen, M., Zhang, D., Dong, W., Luo, Z., Kang, C., Li, H., et al. (2019). Amorphous and humidity caking: A review. *Chinese Journal of Chemical Engineering, 27*(6), 1429–1438. https://doi.org/10.1016/j.cjche.2019.02.004

Chuy, L. E., & Labuza, T. P. (1994). Caking and Stickiness of Dairy-Based Food Powders as Related to Glass Transition. *Journal of Food Science, 59*(1), 43–46. https://doi.org/10.1111/j.1365-2621.1994.tb06893.x

Clydesdale, G., Roberts, K. J., Telfer, G. B., & Grant, D. J. W. (1997). Modeling the crystal morphology of α-lactose monohydrate. *Journal of Pharmaceutical Sciences, 86*(1), 135–141. https://doi.org/10.1021/js950496w

Das, D., & Langrish, T. A. G. (2012). Activated-rate theory: Effect of protein inhibition and the temperature dependence of crystallization kinetics for lactose-protein mixtures. *Food Research International, 48*(2), 367–373. https://doi.org/10.1016/j.foodres.2012.04.019

Das, B., Sarkar, S., Sarkar, A., Bhattacharjee, S., & Bhattacharjee, C. (2016). Recovery of whey proteins and lactose from dairy waste: A step towards green waste management. *Process Safety and Environmental Protection, 101*, 27–33. https://doi.org/10.1016/j.psep.2015.05.006

Dincer, T. D., Parkinson, G. M., Rohl, A. L., & Ogden, M. I. (1999). Crystallisation of α-lactose monohydrate from dimethyl sulfoxide (DMSO) solutions: Influence of β-lactose. *Journal of Crystal Growth, 205*(3), 368–374. https://doi.org/10.1016/S0022-0248(99)00238-9

do Carmo, E. L., Teodoro, R. A. R., Campelo, P. H., Figueiredo, J. de A., Botrel, D. A., Fernandes, R. V. de B., et al. (2019). The use of different temperatures and inulin: Whey protein isolate ratios in the spray drying of beetroot juice. *Journal of Food Processing and Preservation*, *43*(10), 1–9. doi: 10.1111/jfpp.14113.

Drapala, K. P., Murphy, K. M., Ho, Q. T., Crowley, S. V., Mulcahy, S., McCarthy, N. A., et al. (2018). Short communication: Multi-component interactions causing solidification during industrial-scale manufacture of pre-crystallized acid whey powders. *Journal of Dairy Science, 101*(12), 10743–10749. https://doi.org/10.3168/jds.2018-14836

Duhan, N., Sahu, J. K., Mohapatra, A., & Naik, S. N. (2021). Microencapsulation of ghee flavorants with whey protein concentrate and guar gum using spray drying. *Journal of Food Processing and Preservation, 45*(6), 1–13. https://doi.org/10.1111/jfpp.15537

Eckert, C., Serpa, V. G., Felipe dos Santos, A. C., Marinês da Costa, S., Dalpubel, V., Lehn, D. N., et al. (2017). Microencapsulation of Lactobacillus plantarum ATCC 8014 through spray drying and using dairy whey as wall materials. *LWT - Food Science and Technology, 82*, 176–183. https://doi.org/10.1016/j.lwt.2017.04.045

Elmonsef Omar, A. M., & Roos, Y. H. (2007). Glass transition and crystallization behaviour of freeze-dried lactose-salt mixtures. *LWT - Food Science and Technology, 40*(3), 536–543. https://doi.org/10.1016/j.lwt.2005.12.007

Espinosa-Gonzalez, I., Parashar, A., & Bressler, D. C. (2014). Heterotrophic growth and lipid accumulation of Chlorella protothecoides in whey permeate, a dairy by-product stream, for biofuel production. *Bioresource Technology, 155*, 170–176. https://doi.org/10.1016/j.biortech.2013.12.028

Figura, L. O., & Epple, M. (1995). Anhydrous α-lactose A study with DSC and TXRD. *Journal of Thermal Analysis, 44*(1), 45–53. https://doi.org/10.1007/BF02547132

Fischer, C., & Kleinschmidt, T. (2015). Synthesis of galactooligosaccharides using sweet and acid whey as a substrate. *International Dairy Journal, 48*, 15–22. https://doi.org/10.1016/j.idairyj.2015.01.003

Fitzpatrick, J. J., O'Connor, J., Cudmore, M., & Dos Santos, D. (2017). Caking behaviour of food powder binary mixes containing sticky and non-sticky powders. *Journal of Food Engineering, 204*, 73–79. https://doi.org/10.1016/j.jfoodeng.2017.02.021

Foster, K. D., Bronlund, J. E., & Paterson, A. H. J. (Tony. (2006). Glass transition related cohesion of amorphous sugar powders. *Journal of Food Engineering*, *77*(4), 997–1006. doi: 10.1016/j.jfoodeng.2005.08.028.

Fox, P. F. (2009). Lactose: Chemistry and properties. In: P. L. H. McSweeney & P. F. Fox (Eds.), *Advanced dairy chemistry volume 3: Lactose, water, salts and minor constituents* (3rd ed., pp. 1–15). Springer Science.

Galama, A. H., Vermaas, D. A., Veerman, J., Saakes, M., Rijnaarts, H. H. M., Post, J. W., et al. (2014). Membrane resistance: The effect of salinity gradients over a cation exchange membrane. *Journal of Membrane Science, 467*, 279–291. https://doi.org/10.1016/j.memsci.2014.05.046

Gänzle, M. G., Haase, G., & Jelen, P. (2008). Lactose: Crystallization, hydrolysis and value-added derivatives. *International Dairy Journal, 18*(7), 685–694. https://doi.org/10.1016/j.idairyj.2008.03.003

Gernigon, G., Baillon, F., Espitalier, F., Le Floch-Fouéré, C., Schuck, P., & Jeantet, R. (2013). Effects of the addition of various minerals, proteins and salts of organic acids on the principal steps of α-lactose monohydrate crystallisation. *International Dairy Journal, 30*(2), 88–95. https://doi.org/10.1016/j.idairyj.2012.12.005

Gernigon, G, Schuck, P., Jeantet, R., & Burling, H. (2011). Whey processing| Demineralization. In: J. W. Fuquay, P. F. Fox, & P. L. H. McSweeney (Eds.), *Encyclopedia of Dairy Sciences Vol. 3* (2nd ed., pp. 738–743). Academic Press.

González, M. I., Alvarez, S., Riera, F. A., & Álvarez, R. (2008). Lactic acid recovery from whey ultrafiltrate fermentation broths and artificial solutions by nanofiltration. *Desalination, 228*(1–3), 84–96. https://doi.org/10.1016/j.desal.2007.08.009

Greiter, M., Novalin, S., Wendland, M., Kulbe, K. D., & Fischer, J. (2002). Desalination of whey by electrodialysis and ion exchange resins: Analysis of both processes with regard to sustainability by calculating their cumulative energy demand. *Journal of Membrane Science, 210*(1), 91–102. https://doi.org/10.1016/S0376-7388(02)00378-2

Guu, M. Y. K., & Zall, R. R. (1991). Lactose crystallization: Effects of minerals and seeding. *Process Biochemistry, 26*(3), 167–172. https://doi.org/10.1016/0032-9592(91)80013-F

Haider, C. I., Hounslow, M. J., Salman, A. D., Althaus, T. O., Niederreiter, G., & Palzer, S. (2014). Influence of environmental conditions on caking mechanisms in individual amorphous food particle contacts. *AIChE Journal, 60*(8), 2774–2787. https://doi.org/10.1002/aic

Hartel, R. W., & Shastry, A. V. (1991). Sugar crystallization in food products. *Critical Reviews in Food Science and Nutrition, 1*(1), 49–112.

Hartmann, M., & Palzer, S. (2011). Caking of amorphous powders - Material aspects, modelling and applications. *Powder Technology, 206*(1–2), 112–121. https://doi.org/10.1016/j.powtec.2010.04.014

Herrington, B. L. (1934). Some physico-chemical properties of lactose: IV. The influence of salts and acids upon the mutarotation velocity of lactose. *Journal of Dairy Science, 17*(10), 659–670. https://doi.org/10.3168/jds.S0022-0302(34)93285-9

Hogan, S. A., & O'Callaghan, D. J. (2010). Influence of milk proteins on the development of lactose-induced stickiness in dairy powders. *International Dairy Journal, 20*(3), 212–221. https://doi.org/10.1016/j.idairyj.2009.11.002

Hogan, S. A., O'Callaghan, D. J., & Bloore, C. G. (2009). Application of fluidised bed stickiness apparatus to dairy powder production. *Milchwissenschaft, 64*(3), 308–312.

Holsinger, V. H. (1997). Physical and chemical properties of lactose. In: P. F. Fox (Ed.), *Advanced dairy chemistry volume 3* (2nd ed., pp. 1–38). Springer US.

Hourigan, J., Lifran, E., Vu, L., Listiohadi, Y., & Sleigh, R. (2013). Lactose: Chemistry, processing and utilization. In G. W. Smithers, & M. A. Augustin (Eds.), *Advances in dairy ingredients* (1st ed., pp. 31–69). John Wiley & Sons.

Hundre, S. Y., Karthik, P., & Anandharamakrishnan, C. (2015). Effect of whey protein isolate and β-cyclodextrin wall systems on stability of microencapsulated vanillin by spray-freeze drying method. *Food Chemistry, 174*, 16–24. https://doi.org/10.1016/j.foodchem.2014.11.016

Huppertz, T., & Gazi, I. (2016). Lactose in dairy ingredients: Effect on processing and storage stability. *Journal of Dairy Science, 99*(8), 6842–6851. https://doi.org/10.3168/jds.2015-10033

Jelen, P. (2009). Dried whey, whey proteins, lactose and lactose derivative products. In A. Y. Tamime (Ed.), *Dairy powders and concentrated products* (1st ed., pp. 255–267). Blackwell Publishing Ltd.. https://doi.org/10.1002/9781444322729.ch7

Jelen, P., & Coulter, S. T. (1973). Effects of certain salts and other whey substances on the growth of lactose crystals. *Journal of Food Science, 38*(7), 1186–1189. https://doi.org/10.1111/j.1365-2621.1973.tb07234.x

Jones, A. G. (2002). *Crystallization Process Systems* (1st ed.). Butterworth-Heinemann.

Jouppila, K., & Roos, Y. H. (1994). Glass transitions and crystallization in milk powders. *Journal of Dairy Science, 77*(10), 2907–2915. https://doi.org/10.3168/jds.S0022-0302(94)77231-3

Kamrul Haque, M., & Roos, Y. H. (2004). Water plasticization and crystallization of lactose in spray-dried lactose/protein mixtures. *Journal of Food Science, 69*(1). https://doi.org/10.1111/j.1365-2621.2004.tb17863.x

Karel, M., Anglea, S., Buera, P., Karmas, R., Levi, G., & Roos, Y. (1994). Stability-related transitions of amorphous foods. *Thermochimica Acta, 246*(2), 249–269. https://doi.org/10.1016/0040-6031(94)80094-4

Kelly, J., & Kelly, P. (1995). Desalination of acid casein whey by nanofiltration. *International Dairy Journal, 5*(3), 291–303. https://doi.org/10.1016/0958-6946(94)00007-C

Kumar, P., Sharma, N., Ranjan, R., Kumar, S., Bhat, Z. F., & Jeong, D. K. (2013). Perspective of membrane technology in dairy industry: A review. *Asian-Australasian Journal of Animal Sciences, 26*(9), 1347–1358. https://doi.org/10.5713/ajas.2013.13082

Lai, H.-M., & Schmidt, S. J. (1990). Lactose crystallization in skim milk powder observed by hydrodynamic equilibria, scanning electron microscopy and 2H nuclear magnetic resonance. *Journal of Food Science, 55*(4), 994–999. https://doi.org/10.1111/j.1365-2621.1990.tb01582.x

Langrish, T. A. G. (2008). Assessing the rate of solid-phase crystallization for lactose: The effect of the difference between material and glass-transition temperatures. *Food Research International, 41*(6), 630–636. https://doi.org/10.1016/j.foodres.2008.04.010

Lekshmi, R. G. K., Rahima, M., Chatterjee, N. S., Tejpal, C. S., Anas, K. K., Vishnu, K. V., et al. (2019). Chitosan – Whey protein as efficient delivery system for squalene: Characterization and functional food application. *International Journal of Biological Macromolecules, 135*, 855–863. https://doi.org/10.1016/j.ijbiomac.2019.05.153

Leylak, C., Özdemir, K. S., Gurakan, G. C., & Ogel, Z. B. (2021). Optimisation of spray drying parameters for Lactobacillus acidophilus encapsulation in whey and gum Arabic: Its application in yoghurt. *International Dairy Journal, 112*. https://doi.org/10.1016/j.idairyj.2020.104865

Li, K., Pan, B., Ma, L., Miao, S., & Ji, J. (2020). Effect of dextrose equivalent on maltodextrin/whey protein spray-dried powder microcapsules and dynamic release of loaded flavor during storage and powder rehydration. *Foods, 9*(12). https://doi.org/10.3390/foods9121878

Lifran, E. V., Vu, T. T. L., Durham, R. J., Hourigan, J. A., & Sleigh, R. W. (2007). Crystallisation kinetics of lactose in the presence of lactose phosphate. *Powder Technology, 179*(1–2), 43–54. https://doi.org/10.1016/j.powtec.2006.11.010

Listiohadi, Y., Hourigan, J., Sleigh, R., & Steele, R. (2005). Role of amorphous lactose in the caking of alpha-lactose monohydrate powders. *Australian Journal of Dairy Technology, 60*(1), 19–32.

Liu, W., Chen, X. D., Cheng, Z., & Selomulya, C. (2016). On enhancing the solubility of curcumin by microencapsulation in whey protein isolate via spray drying. *Journal of Food Engineering, 169*, 189–195. https://doi.org/10.1016/j.jfoodeng.2015.08.034

Mähler, J., & Persson, I. (2012). A study of the hydration of the alkali metal ions in aqueous solution. *Inorganic Chemistry, 51*(1), 425–438. https://doi.org/10.1021/ic2018693

Malik, S., Krishnaswamy, K., & Mustapha, A. (2021). Physical properties of complementary food powder obtained from upcycling of Greek yogurt acid whey with kodo and proso millets. *Journal of Food Process Engineering, 44*(11). https://doi.org/10.1111/jfpe.13878

Maltini, E., Anese, M., & Shtylla, I. (1997). State diagrams of some organic acid-water systems of interest in food. *Cryo-Letters, 18*(5), 263–265.

Martini, S., Herrera, M. L., & Hartel, R. W. (2001). Effect of cooling rate on nucleation behavior of milk fat - Sunflower oil blends. *Journal of Agricultural and Food Chemistry, 49*(7), 3223–3229. https://doi.org/10.1021/jf001101j

Marx, M., & Kulozik, U. (2018). Thermal denaturation kinetics of whey proteins in reverse osmosis and nanofiltration sweet whey concentrates. *International Dairy Journal, 85*, 270–279. https://doi.org/10.1016/j.idairyj.2018.04.009

McLeod, J., Paterson, A. H. J., Jones, J. R., & Bronlund, J. E. (2011). Primary nucleation of alpha-lactose monohydrate: The effect of supersaturation and temperature. *International Dairy Journal, 21*(7), 455–461. https://doi.org/10.1016/j.idairyj.2011.01.006

Mehra, R., Kumar, H., Kumar, N., Ranvir, S., Jana, A., Buttar, H. S., et al. (2021). Whey proteins processing and emergent derivatives: An insight perspective from constituents, bioactivities, functionalities to therapeutic applications. *Journal of Functional Foods, 87*. https://doi.org/10.1016/j.jff.2021.104760

Michaels, A. S., & van Kreveld, A. (1966). Influences of additives on growth rates in lactose crystals. *Netherlands Milk and Dairy Journal, 20*(3), 163–181.
Mimouni, A., Schuck, P., & Bouhallab, S. (2005). Kinetics of lactose crystallization and crystal size as monitored by refractometry and laser light scattering: Effect of proteins. *Le Lait, 85*(4–5), 253–260. https://doi.org/10.1051/lait
Modler, H. W., & Lefkovitch, L. P. (1986). Influence of pH, casein, and whey protein denaturation on the composition, crystal size, and yield of lactose from condensed whey. *Journal of Dairy Science, 69*(3), 684–697. https://doi.org/10.3168/jds.S0022-0302(86)80457-X
Moser, P., De Souza, R. T., & Nicoletti Telis, V. R. (2017). Spray drying of grape juice from hybrid CV. BRS violeta: Microencapsulation of anthocyanins using protein/maltodextrin blends as drying aids. *Journal of Food Processing and Preservation, 41*(1). https://doi.org/10.1111/jfpp.12852
Mullin, J. W. (1979). Crystal growth in pure and impure systems. In S. J. Jancic, & E. J. de Jong (Eds.), *Industrial crystallization* (pp. 93–103). North-Holland Publishing Co.
Myerson, A. S., & Ginde, R. (2002). Crystals, crystal growth, and nucleation. In A. S. Myerson (Ed.), *Handbook of industrial crystallization* (2nd ed., pp. 33–65). Butterworth-Heinemann.
Nickerson, T. A., & Moore, E. E. (1974). Factors influencing lactose crystallization. *Journal of Dairy Science, 57*(11), 1315–1319. https://doi.org/10.3168/jds.S0022-0302(74)85061-7
O'Mahony, J. A., & Fox, P. F. (2013). Milk proteins: Introduction and historical aspects. In P. L. H. McSweeney, & P. F. Fox (Eds.), *Advanced dairy chemistry: Volume 1A: Proteins: Basic aspects* (4th ed., pp. 43–85). US: Springer.
*OECD-FAO Agricultural Outlook 2019-2028*. (2019). Available from https://www.statista.com/statistics/547609/whey-powder-consumption-volume-european-union-28/.
*OECD-FAO Agricultural Outlook 2021-2030*. (2021). Available from https://www.oecd.org/publications/oecd-fao-agricultural-outlook-19991142.htm.
Okawa, T., Shimada, M., Ushida, Y., Seki, N., Watai, N., Ohnishi, M., et al. (2015). Demineralisation of whey by a combination of nanofiltration and anion-exchange treatment: A preliminary study. *International Journal of Dairy Technology, 68*(4), 478–485. https://doi.org/10.1111/1471-0307.12283
Oliveira, B. E., Junior, P. C. G., Cilli, L. P., Contini, L. R. F., Venturini, A. C., Yoshida, C. M. P., et al. (2018). Spray-drying of grape skin-whey protein concentrate mixture. *Journal of Food Science and Technology, 55*(9), 3693–3702. https://doi.org/10.1007/s13197-018-3299-3
Osorio-Arias, J., Delgado-Arias, S., Duarte-Correa, Y., Largo-Ávila, E., Montaño, D., Simpson, R., et al. (2020). New powder material obtained from spent coffee ground and whey protein; Thermal and morphological analysis. *Materials Chemistry and Physics, 240*(52). https://doi.org/10.1016/j.matchemphys.2019.122171
Ostojić, S., Pavlović, M., Živić, M., Filipović, Z., Gorjanović, S., Hranisavljević, S., et al. (2005). Processing of whey from dairy industry waste. *Environmental Chemistry Letters, 3*(1), 29–32. https://doi.org/10.1007/s10311-005-0108-9
Özkan, N., Walisinghe, N., & Chen, X. D. (2002). Characterization of stickiness and cake formation in whole and skim milk powders. *Journal of Food Engineering, 55*(4), 293–303. https://doi.org/10.1016/S0260-8774(02)00104-8
Palzer, S. (2005). The effect of glass transition on the desired and undesired agglomeration of amorphous food powders. *Chemical Engineering Science, 60*(14), 3959–3968. https://doi.org/10.1016/j.ces.2005.02.015
Pandalaneni, K., & Amamcharla, J. K. (2018). Evaluating the crystallization of lactose at different cooling rates from milk and whey permeates in terms of crystal yield and purity. *Journal of Dairy Science, 101*(10), 8805–8821. https://doi.org/10.3168/jds.2018-14846
Panghal, A., Patidar, R., Jaglan, S., Chhikara, N., Khatkar, S. K., Gat, Y., et al. (2018). Whey valorization: Current options and future scenario – A critical review. *Nutrition and Food Science, 48*(3), 520–535. https://doi.org/10.1108/NFS-01-2018-0017
Parashar, A., Jin, Y., Mason, B., Chae, M., & Bressler, D. C. (2016). Incorporation of whey permeate, a dairy effluent, in ethanol fermentation to provide a zero waste solution for the dairy industry. *Journal of Dairy Science, 99*(3), 1859–1867. https://doi.org/10.3168/jds.2015-10059
Parimaladevi, P., & Srinivasan, K. (2015). Achievement of favorable uniform crystal size distribution of alpha-lactose monohydrate (α-LM) through swift cooling process. *Journal of Food Engineering, 151*, 1–6. https://doi.org/10.1016/j.jfoodeng.2014.11.019
Patel, K. N., & Nickerson, T. A. (1970). Influence of sucrose on the mutarotation velocity of lactose. *Journal of Dairy Science, 53*(12), 1654–1658. https://doi.org/10.3168/jds.S0022-0302(70)86458-X
Paterson, A. H. J. (2017). Lactose processing: From fundamental understanding to industrial application. *International Dairy Journal, 67*, 80–90. https://doi.org/10.1016/j.idairyj.2016.07.018
Paterson, A. H. J., & Bronlund, J. E. (2009). The practical implications of temperature induced moisture migration in bulk lactose. *Journal of Food Engineering, 91*(1), 85–90. https://doi.org/10.1016/j.jfoodeng.2008.08.021
Paterson, A. H. J., Brooks, G. F., Bronlund, J. E., & Foster, K. D. (2005). Development of stickiness in amorphous lactose at constant T-Tg levels. *International Dairy Journal, 15*(5), 513–519. https://doi.org/10.1016/j.idairyj.2004.08.012
Pereira, J. P. F., Melquíades, L. L., Stephani, R., de Oliveira, L. F. C., Perrone, Í. T., & de Carvalho, A. F. (2020). Effect of sodium citrate on lactose crystallization in concentrated whey. *International Journal of Dairy Technology, 73*(4), 757–764. https://doi.org/10.1111/1471-0307.12700
Perrone, Í. T., Pereira, J. P. F., Renhe, I. R. T., Francisquini, J. D., Stephani, R., & De Carvalho, A. F. (2017). Study of lactose crystallization in concentrated whey. *Revista Do Instituto de Laticínios Cândido Tostes, 72*(4), 215–226. https://doi.org/10.14295/2238-6416.v72i4.647
Petit, J., Herbig, A. L., Moreau, A., & Delaplace, G. (2011). Influence of calcium on β-lactoglobulin denaturation kinetics: Implications in unfolding and aggregation mechanisms. *Journal of Dairy Science, 94*(12), 5794–5810. https://doi.org/10.3168/jds.2011-4470
Petruccioli, M., Raviv, M., Di Silvestro, R., & Dinelli, G. (2019). Agriculture and agro industrial wastes, by-products, and wastewaters: Origin, characteristics, and potential in bio-based compounds production. In: M. Moo-Young (Ed.), *Comprehensive Biotechnology* (3rd ed., pp. 477–490). Elsevier.
Písecký, J. (2005). Spray drying in the cheese industry. *International Dairy Journal, 15*(6–9), 531–536. https://doi.org/10.1016/j.idairyj.2004.11.010
Raghavan, S. L., Ristic, R. I., Sheen, D. B., & Sherwood, J. N. (2001). The bulk crystallization of α-lactose monohydrate from aqueous solution. *Journal of Pharmaceutical Sciences, 90*(7), 823–832. https://doi.org/10.1002/jps.1036
Raghavan, S. L., Ristic, R. I., Sheen, D. B., Sherwood, J. N., Trowbridge, L., & York, P. (2000). Morphology of crystals of α-lactose hydrate grown from aqueous solution. *Journal of Physical Chemistry B, 104*(51), 12256–12262. https://doi.org/10.1021/jp002051o
Refstrup, E. (2003). Drying of milk. In H. Roginski (Ed.), *Encyclopedia of dairy sciences* (1st ed., pp. 860–871). Academic Press.
Reid, D. S., & Fennema, O. R. (2008). Water and ice. In S. Damodaran, K. L. Parkin, & O. R. Fennema (Eds.), *Fennema's food chemistry* (4th ed., pp. 17–83). CRC Press.
Rice, G., Kentish, S., O'Connor, A., Stevens, G., Lawrence, N., & Barber, A. (2006). Fouling behaviour during the nanofiltration of dairy ultrafiltration permeate. *Desalination, 199*(1–3), 239–241. https://doi.org/10.1016/j.desal.2006.03.058
Rocha, J. de C. G., de Barros, F. A. R., Perrone, Í. T., Viana, K. W. C., Tavares, G. M., Stephani, R., et al. (2019). Microencapsulation by atomization of the mixture of phenolic extracts. *Powder Technology*, *343*, 317–325. doi: 10.1016/j.powtec.2018.11.040.
Röck, M., & Schwedes, J. (2005). Investigations on the caking behaviour of bulk solids - Macroscale experiments. *Powder Technology, 157*(1–3), 121–127. https://doi.org/10.1016/j.powtec.2005.05.018
Roos, Y., & Karel, M. (1991). Plasticizing effect of water on thermal behavior and crystallization of amorphous food models. *Journal of Food Science, 56*(1), 38–43. https://doi.org/10.1111/j.1365-2621.1991.tb07970.x
Roos, Y., & Karel, M. (1992). Crystallization of amorphous lactose. *Journal of Food Science, 57*(3), 775–777. https://doi.org/10.1111/j.1365-2621.1992.tb08095.x
Salameh, A. K., Mauer, L. J., & Taylor, L. S. (2006). Deliquescence lowering in food ingredient mixtures. *Journal of Food Science, 71*(1), E10–E16. https://doi.org/10.1016/j.foodchem.2006.11.029
Sánchez-García, Y. I., García-Vega, K. S., Leal-Ramos, M. Y., Salmeron, I., & Gutiérrez-Méndez, N. (2018). Ultrasound-assisted crystallization of lactose in the presence of whey proteins and κ-carrageenan. *Ultrasonics Sonochemistry*, *42*(November 2017), 714–722. doi: 10.1016/j.ultsonch.2017.12.020.
Sánchez-García, Y. I., Gutiérrez-Méndez, N., Orozco-Mena, R. E., Ramos-Sánchez, V. H., & Leal-Ramos, M. Y. (2019). Individual and combined effect of pH and whey proteins on lactose crystallization. *Food Research International*, *116*(January 2018), 455–461. doi: 10.1016/j.foodres.2018.08.061.
Sawyer, L. (2013). β-Lactoglobulin. In P. L. H. McSweeney, & P. F. Fox (Eds.), *Advanced dairy chemistry: Volume 1A: Proteins: Basic aspects* (4th ed., pp. 211–259). US: Springer.
Schuck, P., Blanchard, E., Dolivet, A., Mejean, S., Onillon, E., & Jeantet, R. (2005). Water activity and glass transition in dairy ingredients. *Le Lait, 85*, 295–304. https://doi.org/10.1051/lait:2005020
Schulze, D. (2008). *Powders and bulk solids: Behavior, characterization, storage and flow*. Berlin Heidelberg: Springer.
Shi, Y., Hartel, R. W., & Liang, B. (1989). Formation and growth phenomena of lactose nuclei under contact nucleation conditions. *Journal of Dairy Science, 72*(11), 2906–2915. https://doi.org/10.3168/jds.S0022-0302(89)79441-8
Shi, X., & Lee, Y. (2020). Encapsulation of tributyrin with whey protein isolate (WPI) by spray-drying with a three-fluid nozzle. *Journal of Food Engineering, 281*(February), Article 109992. https://doi.org/10.1016/j.jfoodeng.2020.109992
Shrestha, A. K., Adhikari, B. P., Howes, T. E., & Bhandari, B. R. (2006). Effect of lactic acid on spray drying behaviour of acid-whey and study of their glass transition temperature. *Journal of Food Science and Technology Nepal, 2*, 57–62.
Shrestha, A. K., Howes, T., Adhikari, B. P., Wood, B. J., & Bhandari, B. R. (2007). Effect of protein concentration on the surface composition, water sorption and glass transition temperature of spray-dried skim milk powders. *Food Chemistry, 104*(4), 1436–1444. https://doi.org/10.1016/j.foodchem.2007.02.015
Simeão, M., Da Silva, C. R., Stephani, R., de Oliveira, L. F. C., Schuck, P., de Carvalho, A. F., et al. (2018). Lactose crystallisation in concentrated whey: The influence of vat type. *International Journal of Dairy Technology, 71*(2), 478–483. https://doi.org/10.1111/1471-0307.12455
Smart, J. B. (1988). Effect of whey components on the rate of crystallization and solubility of alfa-lactose monohydrate. *New Zealand Journal of Dairy Science and Technology, 23*, 275–289.
Smithers, G. W., & Augustin, M. A. (2013). *Advances in dairy ingredients* (1st ed.). John Wiley & Sons.
Stănciuc, N., Der Van Plancken, I., Rotaru, G., & Hendrickx, M. (2008). Denaturation impact in susceptibility of beta-lactoglobulin to enzymatic hydrolysis: A kinetic study. *Revue Roumaine de Chimie, 53*(10), 921–929.
Suárez, E., Lobo, A., Alvarez-Blanco, S., Riera, F. A., & Álvarez, R. (2006). Utilization of nanofiltration membranes for whey and milk ultrafiltration permeate demineralization. *Desalination, 199*(1–3), 345–347. https://doi.org/10.1016/j.desal.2006.03.081
Talón, E., Lampi, A. M., Vargas, M., Chiralt, A., Jouppila, K., & González-Martínez, C. (2019). Encapsulation of eugenol by spray-drying using whey protein isolate or

lecithin: Release kinetics, antioxidant and antimicrobial properties. *Food Chemistry, 295*(May), 588–598. https://doi.org/10.1016/j.foodchem.2019.05.115

Van Der Bruggen, B., Koninckx, A., & Vandecasteele, C. (2004). Separation of monovalent and divalent ions from aqueous solution by electrodialysis and nanofiltration. *Water Research, 38*(5), 1347–1353. https://doi.org/10.1016/j.watres.2003.11.008

van der Horst, H. C., Timmer, J. M. K., Robbertsen, T., & Leenders, J. (1995). Use of nanofiltration for concentration and demineralization in the dairy industry: Model for mass transport. *Journal of Membrane Science, 104*(3), 205–218. https://doi.org/10.3923/pjbs.2006.991.994

von Hippel, P. H., & Schleich, T. (1969). The effects of neutral salts on the structure and conformational stability of macromolecules in solution. In S. N. Timasheff, & G. D. Fasman (Eds.), *Structure and stability of biological macromolecules* (2nd ed.,, pp. 417–574). Marcel Dekker.

Walstra, P., & Jenness, R. (1984). *Dairy chemistry and physics* (1st ed.). John Wiley & Sons.

Walstra, P., Wouters, J. T. M., & Geurts, T. J. (2006). *Dairy science and technology* (2nd ed.). CRC Press.

Wee, Y. J., Yun, J. S., Lee, Y. Y., Zeng, A. P., & Ryu, H. W. (2005). Recovery of lactic acid by repeated batch electrodialysis and lactic acid production using electrodialysis wastewater. *Journal of Bioscience and Bioengineering, 99*(2), 104–108. https://doi.org/10.1263/jbb.99.104

Westergaard, V. (2003). Dryer design. In H. Roginski (Ed.), *Encyclopedia of dairy sciences* (1st ed., pp. 871–889). Academic Press.

Whittier, E. O., & Gould, S. P. (1930). Vapor pressures of saturated equilibrated solutions of lactose, sucrose, glucose and galactose. *Industrial and Engineering Chemistry, 22*(1), 77–78. https://doi.org/10.1021/ie50241a022

Wijayasinghe, R., Vasiljevic, T., & Chandrapala, J. (2015). Water-lactose behavior as a function of concentration and presence of lactic acid in lactose model systems. *Journal of Dairy Science, 98*(12), 8505–8514. https://doi.org/10.3168/jds.2015-9959

Wong, S. Y., & Hartel, R. W. (2014). Crystallization in lactose refining-A review. *Journal of Food Science, 79*(3). https://doi.org/10.1111/1750-3841.12349

Wunsch, N.-G. (2021). Size of the whey protein market worldwide 2020 & 2028. *Statista/Polaris Market Research.* https://www.statista.com/statistics/728005/global-whey-protein-market-size/.

Zamanipoor, M. H., & Mancera, R. L. (2014). The emerging application of ultrasound in lactose crystallisation. *Trends in Food Science and Technology, 38*(1), 47–59. https://doi.org/10.1016/j.tifs.2014.04.005

Zandona, E., Blažić, M., & Režek Jambrak, A. (2021). Whey utilisation: Sustainable uses and environmental approach. *Food Technology and Biotechnology, 59*(2), 147–161. https://doi.org/10.17113/ftb.59.02.21.6968

Zhang, Z., Sun, D. W., Zhu, Z., & Cheng, L. (2015). Enhancement of crystallization processes by power ultrasound: Current state-of-the-art and research advances. *Comprehensive Reviews in Food Science and Food Safety, 14*(4), 303–316. https://doi.org/10.1111/1541-4337.12132