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Conventional and alternative concentration processes in milk manufacturing: a comparative study on dairy properties

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Abstract

The concentration of dairy products is widely applied in dairy manufacturing due to obtaining products with the high dry matter, added value, reduced volume, and an increase in shelf-life. Traditional thermal concentration processes are the most applied in dairy industries, however, high temperatures can damage the bioactive compounds in milk, in addition to modifying the physicochemical, sensory, and nutritional characteristics of concentrated products. This review summarizes the importance of replacing traditional concentration methods with unconventional non-thermal processes, which can bring an option to dairy industries due to the concentration enabling the preservation of proteins, enzymes, vitamins, color, and flavor of the product. Alternative methods, such as freeze concentration, membrane separation processes, and freeze-drying, compose recent works about new methodologies to concentrate dairy products without changing specific properties and increase the quality, which is one of the main purposes for the dairy industries. Through a comparative study with recent researches, this overview highlights some alternative concentration processes that can improve the yield and increase the quality of concentrated dairy products. With new environmentally sustainable methods and the possibility of reducing the costs of the concentration process, these emerging concentration methods become attractive for dairy industries from a technological and economic perspective.

Keywords: non-thermal processing; freeze concentration; membrane separation; freeze-drying; dairy processing; thermolabile compounds.

Practical Application: Improving the quality of concentrated dairy products by non-thermal emerging technologies.

1 Introduction

Milk is a highly nutritional valuable food that can be processed, fractionated, and included in dairy products, beverages, or food formulations (Al-Hilphy et al., 2020; Muñoz et al., 2018; Prestes et al., 2021; Vargas et al., 2021). In addition, dairy products are elucidated as being excellent sources of nutritional compounds, bring health benefits if introduced in a well-balanced diet (Feeney et al., 2021; Verruck et al., 2019a).

Thermal and non-thermal processes implemented in dairy manufacturing have the main purpose to increase the shelf-life and produce a safe, stable, nutritional, and product (Al-Hilphy et al., 2020; Musina et al., 2018; Stratakos et al., 2019).

Dairy products contain high water content and, with the purpose to expand the shelf-life, concentration processes are fundamental in dairy industries, since the employed technology can improve the efficiency of milk processing, reducing the volume of production and total costs of shipping and storage (Balde & Aïder, 2017; Liz et al., 2020; Muñoz et al., 2018). In addition, there is an increase in total dry matter which benefits the added value of a product with high fat and protein content (Carter et al., 2021; Rao, 2018; Vargas et al., 2021).

In large-scale production, traditional concentration methods are the most employed in dairy manufacturing, mainly the evaporation and spray drying processes These unit operations reduce the water content by applying high temperatures during the procedure. However, an intense heat treatment may exceed the heat stability of milk and result in undesired sensory and physiochemical changes, such as separation of milk fat, grittiness, phase separation and sediment formation (Dumpler et al., 2020). Besides, the intense thermal processes may decrease original thermolabile bioactive compounds such as enzymes, vitamins, and proteins (Dumpler et al., 2018, 2020; Moejes et al., 2020).

Emerging non-thermal technologies are promising alternatives that have been developed and explored in dairy manufacturing. With a purpose to decrease the negative effects of the conventional concentration processes and contribute with dairy products with high quality, these alternative procedures preserve sensory and flavor properties and maintain food pigments, original volatile compounds, vitamins, enzymes, and proteins (Liz et al., 2020; Faion et al., 2019; Canella et al., 2020; Moejes et al., 2020; Muñoz et al., 2018; Stratakos et al., 2019). In recent research

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about milk concentration processes, technologies such as freeze concentration, membrane separation and freeze-drying are efficient, satisfactory and capable to replace traditional concentration processes and develop concentrated dairy products with high quality (Barros et al., 2022a; Camelo-Silva et al., 2022a; Liz et al., 2020; Deshwal et al., 2020; Faion et al., 2019; Merkel et al., 2021; Muñoz et al., 2018; Zhu et al., 2020). Studies can bring essential and attractive information for dairy industries, which can apply different concentration procedures according to the desirable characteristics of their manufacturing processes.

Alternative milk concentration processes have several techniques and different procedures, bringing unique properties to each process. In dairy science and technology, our research group has been operating in the field for over 15 years, with experience in conventional and alternative technologies in milk processing (Camelo-Silva et al., 2022a; Dantas et al., 2021; Magenis et al., 2006; Muñoz et al., 2018, 2019; Prudêncio et al., 2014). The purpose of the group, through this review, is to present an essential background of the main emerging technologies of milk concentration and the benefits caused in the organoleptic properties of dairy products concerning the traditional concentration methods.

2 Conventional concentration processes in milk manufacturing

To produce concentrated or dried dairy products, there are several food processes to remove the liquid fraction, including traditional thermal processes or innovative technologies. The choice will depend on the desired effect of the process on the product's morphology and the extent of concentration required (Cheng et al., 2018; Morison & Hartel, 2018). Concentration, drying or the combination of these two technologies are the most energy-intensive operations of the dairy industry and, for this reason, the total costs of these concentration processes must also be considered to apply them on a large-scale (Moejes et al., 2020; Ramírez et al., 2006).

2.1 Evaporation process

The evaporation of milk has been employed for several years and the main purpose of this unitary operation is to remove the water from the solution to increase the solutes content such as proteins, fat, sugars, and minerals from milk composition. Using high temperatures, the objective is to concentrate on minimum total cost, which is included the energy and cleaning expenditures, capital and operating costs, and product loss (Dumpler et al., 2020; Morison & Hartel, 2018). Offering the lowest annual capital cost (approximately € 4M) and high concentration levels, evaporation is considered the most practical concentration approach (Table 1) (Ali et al., 2021; Balde & Aïder, 2017; Schuck et al., 2015; Tanguy et al., 2015).

Usually, the development of evaporators permits the liquid to be concentrated to flow through a tube in which the heat is applied outside. The liquid is heated up to the boiling point at ambient pressure (100 °C at sea level and 85 °C at an altitude approximately 5000 m above sea level) and the water is separated from the concentrated fraction. Due to the high latent heat of water evaporation, the energy efficiency is increased with the employment of multiple stages of evaporators or vapor recycling to reuse energy, and decrease this source demand (Morison & Hartel, 2018; Ramírez et al., 2006).

In dairy industries, the evaporation process is usually done in long-tube vertical falling film evaporators. In this process, the milk (near at its boiling point) is uniformly fed at the top of the inner surface of a tube, which is built side by side with other tubes, fixed, and enclosed by a jacket (Figure 1A). After the milk passes down inside of each tube, forming a thin film, it boils due to the heat applied by the steam. The concentrated liquid is separated at the bottom part of the equipment and the remaining part is removed from the steam in a subsequent separator. In evaporators with multiple effects, the concentrated liquid is pumped to the next stage, while the steam is used to heat the next stage (Figure 1B)(Fernández-Seara & Pardiñas, 2014; Guichet & Jouhara, 2020; Morison & Hartel, 2018).

Falling film evaporators offer the advantage of the short residence time of the liquid within the equipment. In addition, to increase energy efficiency, this process is generally operated under vacuum conditions, which is beneficial for milk concentration, since several bioactive compounds can be damaged by extreme heat exposition (Dumpler et al., 2020; Morison & Hartel, 2018). In dairy manufacturing, the evaporation process is used as a first step to concentrate products to drying such as whey protein concentrate, lactose, or powdered milk to increase the stability, reduce the volume and production costs, storage, and transportation. Some products such as condensed milk, *dulce de leche*, and evaporated milk are sold as concentrated liquids (Dumpler et al., 2020; Morison & Hartel, 2018; Vargas et al., 2021).

Effects in the dairy matrix composition through the evaporation process

Processing of concentrated dairy products includes several steps that may affect the stability of dairy matrix compounds. Due to the high sensitivity of milk nutritional compounds to intense thermal and mechanical processes, undesirable changes may occur in the physicochemical, sensory, or microbiological characteristics of the dairy matrix and its concentrated products (Dumpler et al., 2020; Masum et al., 2020; Tari et al., 2021; Verruck et al., 2019b; Wu et al., 2021).

Evaporators must have time and temperature controlled throughout the procedure. Intense heat exposure of the evaporation process affects the natural pH of milk (approximately 6.6-6.8), resulting in changes in the milk salt equilibrium and denaturation proteins during this procedure and a consequently level of coagulation (Dumpler et al., 2020; Lin et al., 2018; Verruck et al., 2019b). A significant decrease in pH during heating is primarily due to acid produced from lactose oxidation at high temperatures, hydrolysis of organic phosphate groups, and precipitation of calcium phosphate (Koutina & Skibsted, 2015; Wu et al., 2021).

Milk proteins directly and indirectly interact with lactose. During any heat treatment, an intense and prolonged heat exposure (above 100 °C) results in the formation of early and, in some dairy products, undesired Maillard products with changes in color, texture and flavor aspects (Dumpler et al., 2018, 2020;

Table 1. Comparation between the traditional and alternative milk concentration processes.

	Process	Advantages	Disadvantages	Studies about energy expenditures
conventional concentration processes	Evaporation	-The most practical milk concentration approach;	- High temperatures may decrease milk bioactive compounds (vitamins, enzymes, proteins);	3200 kJ.kg^{-1} water removed (single- effect evaporator under partial vacuum and under atmospheric pressure at boiling temperature);
		- Low energy costs using multi-stages evaporators;	- Denaturation of milk proteins may result in heat-induced fouling inside of evaporators;	900 kJ.kg^{-1} (pilot-scale roller dryers in a partial vacuum chamber with a mechanical vapor recompression heating system) (Ramírez et al., 2006; Schuck et al., 2015)
		- Increased shelf-life	- Intense heat treatment can affect the pH sensibility and minerals equilibrium;	300 kJ.kg^{-1} (traditional multi-stages evaporators with mechanical vapor recompression- MVR) (Moejes et al., 2020; Ramírez et al., 2006; Tanguy et al., 2015)
			- In specific products, undesirable changes of sensory properties (flavor, color and texture);	
			- High installation and operating costs	
	Spray drying	- Increased shelf-life;	- Decrease in milk thermolabile compounds;	5256 kJ.kg^{-1} for a skim milk spray-dried from 50% to 96% total solids (Ramírez et al., 2006; Schuck et al., 2015);
		- Lower storage and transportation costs;	- High energy consumption (10-20 times higher than evaporation process);	One single stage: 4900 kJ/kg water evaporated; two stages: 4300 kJ/kg; three stages 3400 kJ/kg (Ramírez et al., 2006; Schuck et al., 2015; Verdurmen & Jong, 2003)
		- Reduction of the product volume;	- To reduce energy costs, there is a need for pre-concentration (90% of the water is removed in the evaporator and only 9-10% in the spray dryer)	
		-Expansion of logistic distribution;	- Changes in the fat and lactose conformation, resulting in undesirable physicochemical and sensory characteristics	
		- Requires a noticeably short time		

Table 1. Continued...

	Process	Advantages	Disadvantages	Studies about energy expenditures
alternative concentration processes	Freeze concentration	- Preservation of highly heat-sensitive milk compounds;	- High investment cost in large-scale production;	335 $kJ.kg^{-1}$ water removed (which is equivalent to approximately 15% of heat addition in a single-effect evaporator) (Sánchez et al., 2010)
		- Maintenance of color and flavor;	- High refrigeration and operating costs;	
		- Low deterioration due to decreased of enzymatic and microbiological activities;	- Low production rate;	
		- Increased shelf-life;	- Loss of soluble solids in the ice fraction	
		- Non- polluting process;		
		- Without the use of preservatives and additives;		
	Membrane separation	-Low energy consumption;	- Membrane pores are often clogged;	14.0-36.0 $kJ.kg^{-1}$ water removed by pressure driven membrane filtration (Moejes et al., 2020; Ramírez et al., 2006);
		- High flow rates;	- Requires high quality water for cleaning;	50.08-62.54 $kJ.L^{-1}$ of milk retentate, 18.18-21.65 $kJ.L^{-1}$ of permeate and 87.08-107 kJ for cleaning at different pressures (Bahnasawy & Shenana, 2010);
		- Improvement of the yield;	- High maintenance costs	0.6-1.5 $kJs.kg^{-1}$ for ultrafiltration of a Greek-style yogurt (Paredes Valencia et al., 2018)
		- Increased shelf-life;		
		-Removal of bacterial pathogens and spores;		
		- Non-polluting process;		
		- Without the use of preservatives and additives		
	Freeze drying	-Preservation of bioactive compounds;	- High energy consumption;	
		-Maintenance of flavor and color;	- Inviable to produce in large-scale;	
		- Increased shelf-life;	- Low production rate;	3820-5500 $kJ.kg^{-1}$ water removed. In 24 h, the energy consumption of all the consumers (cooling chamber, sublimation, and vacuum pump) in a freeze-dryer is 937,177.42 kJ (Bando et al., 2017; Keselj et al., 2017)
		- Facility of transport and storage;	- Requires sterile diluents on reconstitution;	
		- Non-polluting process, low waste water;	- High cost and complexity of the equipment	
		- Without the use of preservatives and additives;		
		-Process with absence of oxygen, preventing against oxidative reactions		

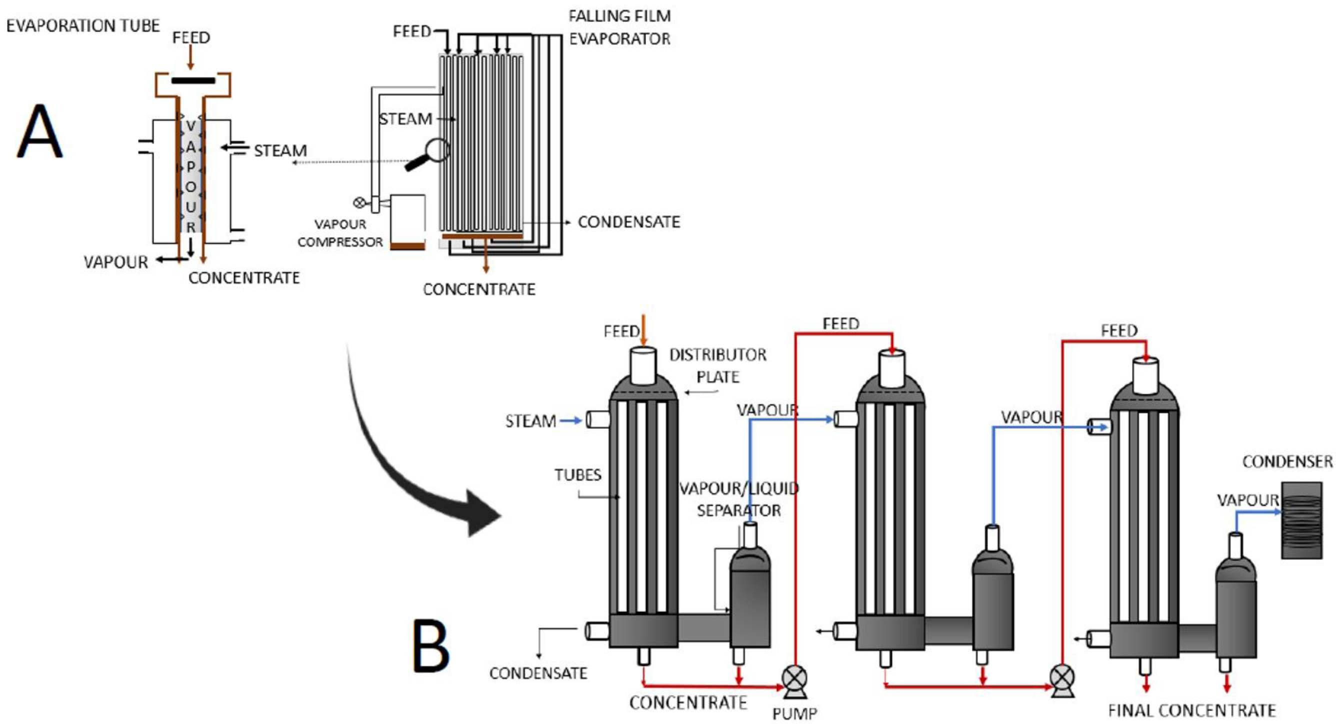

Figure 1. A: A long tube vertical falling film evaporator. Adapted from Verdurmen & Jong (2003); B: Multiple effects of a long tube vertical falling film evaporator.

Dumpler & Kulozik, 2015, 2016). The consequent isomerization and thermal degradation of lactose are parallel reactions to the Maillard reaction and, products such as formic and acetic acid, from lactose oxidation, also lead to a decrease in pH of milk (Dumpler et al., 2020; Fox et al., 2015).

The heat stability and the pH sensitivity of milk are often attributed to the presence of casein micelles formed by calcium bridges and linkages by colloidal calcium phosphate through complex hydrogen, hydrophobic bonds, and electrostatic interactions (Dumpler et al., 2020; Koutina & Skibsted, 2015). During a heat processing of milk below 80 °C, whey proteins denature and there are changes in the size and structure of casein micelles, reducing the amount of ionic calcium and phosphate, which will also decrease the pH due to remaining as free ions in the whey fraction for further interactions during acid clot formation (Deeth & Bansal, 2019; Dumpler et al., 2020; Koutina & Skibsted, 2015). This consequent coagulum from denaturing whey proteins and casein micelles may result in heat-induced fouling inside of evaporators, increasing the viscosity, decreasing the heat transfer and the energy efficiency of the equipment (Dumpler & Kulozik, 2015; Wu et al., 2021). In addition, heat-induced protein accumulation on the inner contact surface of the evaporator can induce the proliferation of microorganisms and problematic decreases in product quality, such as changes in color, flavor, and texture. The growth of pathological microorganisms can also be a risk to the consumer's health, with the industries being responsible for all the quality and food safety, from processing to the market.

2.2 Spray drying process

Milk powder and powdered dairy products present a globally consolidated market and a particular interest by food industries due to their high acceptability and added value. The milk powder is an interesting solution for those who lack direct access to adequate refrigeration, which characterizes it's practically of consumption (Ding et al., 2021a; Kalyankar et al., 2015). Due to the functional and nutritional properties of milk and the high value of its components, powdered milk and powdered dairy products are considered a valuable concentrated ingredient for diverse applications in food formulations, such as bakeries, confectionaries, infant formulas, meat products, and nutritional foods (Kalyankar et al., 2015; Khan et al., 2021).

The storage of fresh milk presents obstacles due to the vast volume of dairy processing and the need for refrigerated storage, transport, and marketing. Drying is a conventional way of concentrating food and facilitating logistics, handling milk, increasing the shelf life and stability of the product. The main purpose of converting fresh milk into milk powder is to transform a liquid perishable matrix into a product that can be stored for years without loss in physicochemical, microbiological, nutritional, and sensory quality (Deshwal et al., 2020; Ding et al., 2021a; Kalyankar et al., 2015).

The usual process of drying in dairy industries is spray drying and, by definition, is a transformation of feed from a solution, suspension, or paste into a concentrated/dried form by spraying this fluid into a hot drying medium (Verdurmen & Jong, 2003). To improve the thermal efficiency of the drying procedure and avoid overheating of powder particles, the equipment (spray dryer) can consist of one, two, or three stages (Figure 2). Different from evaporators, there is no recovering of latent heat of the vapor in the spray dryer. Drying is responsible for up to 15% of the industrial energy requirement and, if compared to a concentration by evaporation, the energy demand by the spray drying process is 10-20 times higher per kilogram of water removed (Table 1). Consequently, it is usual to proceed with a primary concentration by evaporation before drying, however, spray-drying is still the most energy-intensive process in dairy industries and has received much attention (Ramírez et al., 2006; Schuck et al., 2015; Verdurmen & Jong, 2003).

In the first stage, the preheated feed solution (< 100 °C) is pumped from a product tank to the atomizing dispositive, which contains the drying chamber. The drying air, composed of filtered atmospheric, is inserted through the hot chamber at 150-250 °C by an air disperser. The atomized feed solution meets the hot drying air and occurs the solvent evaporation, which occurs simultaneously with the cooling of the air. The concentrated product is converted into droplets of 10-200 μm and depending on the dimensions of the spray-dryer, the residence time of the dried particles is around 5 to 30 s (Schuck et al., 2015; Verdurmen & Jong, 2003). Most powder particles fall to the bottom of the dryer and are submitted to a pneumatic transport and an immediate cooling system, which is essential to preserve better flavor, physicochemical characteristics, and long shelf life. The particles with the smallest diameters remain in the air and, if necessary, the air passes to a cyclone to separate the solid fraction. After drying, the powders are transported to the next drying stage or a packing system (Kalyankar et al., 2015; Masum et al., 2020).

Effects in dairy composition through the spray drying process

The dairy concentration by spray drying process is a traditional, important, and economic operation due to the flexibility in handling a variety of products. However, the high temperature needed in this process reduces the heat-sensitive nutritional and sensory components of the original matrix. In addition, the drying air temperature can affect the physicochemical properties of powder milk by changes in the distribution of the majority components, particle morphology, color characteristics, and water activity (Deshwal et al., 2020; Habtegebriel et al., 2018; Perusko et al., 2021).

One of the components of the solid fraction that is most affected during the spray drying process is the milk fat, which is dispersed in a colloidal system with proteins, water, and soluble components. Aromatic compounds of powdered milk undergo several complex changes during processing and, in spray drying conditions precisely, the thermal procedure can cause damage of droplet shrinkage and release free fat, which is easily oxidized and cause sweet taste, fatty and creamy flavor of powdered milk (Feng et al., 2021). In addition, after drying, the fat is distributed on the surface of whole milk particles and this conformation influences the size of dried flakes and affects the interconnecting among the powder particles, forming an undesirable pasty characteristic (Birchal et al., 2005; Habtegebriel et al., 2018; Lin et al., 2018). Milk protein is also an important macronutrient that can be used as indicator of milk quality after a technological treatment and is also affected by the intense temperature of the spray dryer. According to Vincenzetti et al. (2018), who studied the effects of spray drying and freeze-drying processes on the β-lactoglobulin and lysozyme content in donkey milk, the high temperature to which the donkey milk was subjected decreased

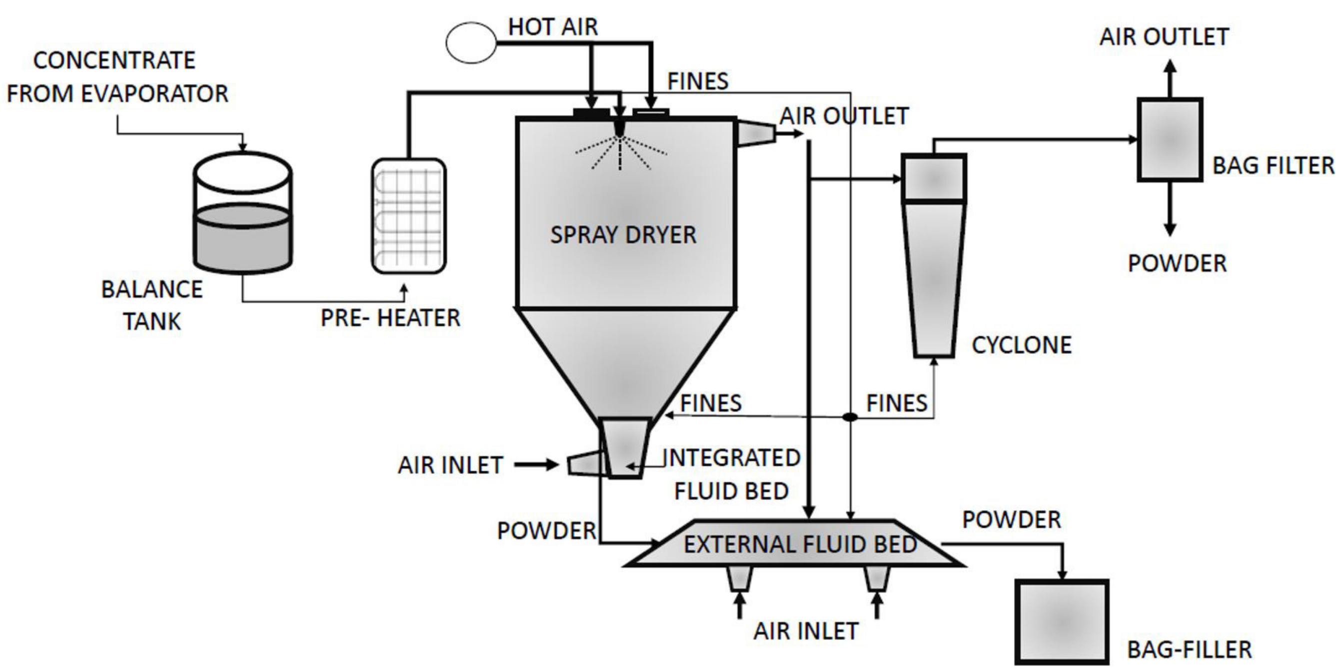

Figure 2. Stages of a spray dryer. Adapted from Verdurmen & Jong (2003).

significantly the lysozyme enzymatic activity (58% of residual activity) and β-lactoglobulin content (6.43 mg/mL in fresh milk *vs*. 5.51 mg/mL in spray-dried milk). The denaturation of milk proteins can also cause encrustations inside the spray-dryer, reducing the efficiency of heat exchange in the equipment and blocking nozzles, causing a low-quality powder and requiring product rework (Bista et al., 2021; Cheng et al., 2018; Deshwal et al., 2020).

The lactose fraction has also proven to be responsible for important roles in the sensory, nutritional, functional, and physicochemical properties of powdered dairy products (Fialho et al., 2018; Park et al., 2016; Rongsirikul & Hongsprabhas, 2016; Zhou & Langrish, 2021). Depending on the processing conditions, in addition to high temperatures, water evaporation, high concentration of lactose and lysine-rich proteins, Maillard reactions may occur and causes several effects, including the unattractive formation of melanoidins (nitrogen-containing brow pigments), the loss of nutritional value, changes in the sensory properties, presence of off-flavors and formation of potential mutagenic products (Perusko et al., 2021; Zhou & Langrish, 2021). Due to the quick changes in the temperature and moisture composition in the hot chamber of the equipment, Maillard reactions in spray dryers may be different from those liquid systems. Furthermore, due to the fast rate of water removal, the lactose structure is converted into its amorphous glassy state, which is unfavorable to the crystallization phenomenon (Habtegebriel et al., 2018). This lactose form, at high temperatures or moisture content in the storage of powdered dairy products, develops molecular mobility and converts into a rubbery state, which occurs at a temperature range known as glass transition temperature (Tg). If the storage occurs at temperature higher than Tg, the mobility of this lactose conformation increases and the viscosity decreases, initiating the lactose crystallization. According to studies, the extension of Maillard reactions can be reduced by improving the design of spray dryers, as well as the control of dairy concentration processes and all the external variables and internal properties (Deshwal et al., 2020; Habtegebriel et al., 2018; Masum et al., 2020; Perusko et al., 2021; Zhou & Langrish, 2021).

Due to these undesirable changes in physicochemical and sensory aspects of concentrated dairy products that may occur in traditional concentration processes, evaporation and spray drying can be replaced recently by new and alternative concentration processes from the rise of studies of food engineering and food technology, developing alternative and non-thermal technologies which allow the permanence of highly heat-sensitive milk compounds (Balde & Aïder, 2017; Camelo-Silva et al., 2022a; Casas-Forero et al., 2020; Liz et al., 2020; Petzold et al., 2016; Canella et al., 2020; Moejes et al., 2020; Muñoz et al., 2018). Recently, the dairy sector around the world aims for product quality. Dairy products with high functional and nutritional quality have become the most desired option for consumers, being one of the main focuses of manufacturing. Studies, researches, and development of new food concentration processes allow new choices for industries, improving the production chain.

3 Alternative concentration processes in milk manufacturing

Non-conventional concentration processes gain increasingly attention from dairy industries as means to decrease the negative effects of conventional processing technologies. Non-thermal technologies maintain the maximum of milk bioactive compounds, obtaining a concentrated dairy product with high quality, nutritional and functional value, accepted sensorially and offsetting the total production costs. The advances of new studies support industries to apply emergent technologies in the dairy manufacturing according to the production flow, method of operation and the type of dairy products, since alternative technologies develop products with specific properties to each type of method, which also becomes a factor of choice for the industrial sector on large-scale application (Barros et al., 2022a; Camelo-Silva et al., 2022b; Deshwal et al., 2020; France et al., 2021; Canella et al., 2020; Merkel et al., 2021; Shabbir et al., 2021; Verruck et al., 2019b).

3.1 Freeze concentration

The freeze concentration process is a non-thermal concentration technology applied in liquid foods based on a solid-liquid separation at low temperatures, which the water fraction is frozen, transformed into pure ice crystals, and removed from the concentrated solution (Ding et al., 2021b; Sánchez et al., 2010). This method can be used to concentrate or pre-concentrate heat-sensitive compounds, which is an interesting alternative for dairy manufacturing, due to the maintenance of the nutritional value, volatile compounds, and flavor of dairy products (Barros et al., 2022b; Benedetti et al., 2015; Camelo-Silva et al., 2022a; Liz et al., 2020; Canella et al., 2020; Muñoz et al., 2018; Prestes et al., 2022; Sánchez et al., 2011a). Freeze concentrated dairy products can be used in diverse food formulations or as an intermediate matrix in dairy processing (Balde & Aider, 2016; Balde & Aïder, 2017; Barros et al., 2022b; Camelo-Silva et al., 2022b; Muñoz et al., 2018).

The process is based on lowering and controlling the temperature of the solution below its freezing point to avoid freezing all the components simultaneously, at the eutectic point. The purity of ice crystals increases with controlled freezing above the eutectic point of the solution, preserving all the properties of the original liquid matrix. Usually, the upper limit of freeze concentration, concerning the original liquid food, range from 40 to 50% of solid content, depending on the level of soluble solids and the food matrix composition (Morison & Hartel, 2018; Prestes et al., 2022; Sánchez et al., 2011b).

Since this process does not involve a liquid-vapor interface, there is the maximum preservation of thermolabile compounds, increasing the quality of the concentrated product. In addition, from a thermodynamic point of view, the freeze concentration is highly interesting due the energy consumption is lower when compared to evaporation and spray drying processes. The latent heat of water freezing is almost one-seventh of latent heat of water evaporation (335 $kJ.kg^{-1}$ *vs* 2260 $kJ.kg^{-1}$), which is a potential for energy saving for de-watering of liquid foods (Table 1) (Balde & Aïder, 2017; Casas-Forero et al., 2020; Ding et al., 2021b;

Prestes et al., 2022). In freeze concentration methods, the energy savings can also be related to the possibility of using passive thawing as a recovery step of the concentrated phase, which enhances the energy efficiency and quality of the concentrated products (Balde & Aïder, 2017). Since the yield of the concentrate is not high compared to other concentration methods, the separation of frozen water from the concentrate solution can be carried out once or through several successive freezing steps, in the same liquid food. This procedure, which increases the soluble solids content, will depend on the original composition of the liquid matrix, the desirable objective, and the yield of each freeze concentration stage. In the freeze concentration technology, ice crystals can be formed from liquid solutions in different methods: suspension, progressive, and block freeze concentration, with distinct freeze techniques and ice crystals separation (Casas-Forero et al., 2020; Ding et al., 2021b; Samsuri et al., 2018; Sánchez et al., 2011a, 2011b; Zambrano et al., 2018).

The suspension freeze concentration is the most complex and expansive method of freeze concentration, based on the formation of individual ice crystals when the liquid matrix is submitted at low temperatures. An initial phase of crystallization occurs and the ice crystals position themselves into large ice particles, increasing the volume and, in the second phase, ice nuclei grow inside the solution (Morison & Hartel, 2018; Sánchez et al., 2011a). The size of these ice crystals is limited, and the separation of the concentrate is complex with the use of typical equipment, with specific purposes (Figure 3A). The system is composed of a crystallizer (heat exchanger) to promote the crystals' growth. At a pre-freezing temperature, the liquid matrix is cooled and advances to the crystallizer to form ice crystals, which are removed from the concentrate fraction inside of a separator. The remains of frozen pure water are separated from the concentrated liquid in a wash column with compression to handle the ice crystals with high purity (Aider & Halleux, 2009; Morison & Hartel, 2018; Sánchez et al., 2011a).

The progressive freeze concentration is one of the most important methods for concentrating liquid foods and is based on the layer crystallization, in which a large mass of ice is formed on a cold surface, with an easier separation due to the ice crystals adhering to the surface (Moharramzadeh et al., 2021; Morison & Hartel, 2018; Samsuri et al., 2018). This process is simpler and must be developed according to specific properties of the liquid food, the concentrate fraction, and its yield at the end of the procedure. The costs of operation, equipment, and maintenance are low, which makes this process promising to be applied on a large scale and replace the complexity of a concentration by suspension crystallization (Morison & Hartel, 2018; Sánchez et al., 2011a). This type of freeze concentration has been proven to be efficient in studies about the separation and concentration of dairy products, which systems were developed and improved according to the dairy matrix and specific needs (Chabarov & Aider, 2014; Dantas et al., 2021; Muñoz et al., 2019; Samsuri et al., 2018). A progressive freeze concentration system was proposed for the first time in the separation of skimmed

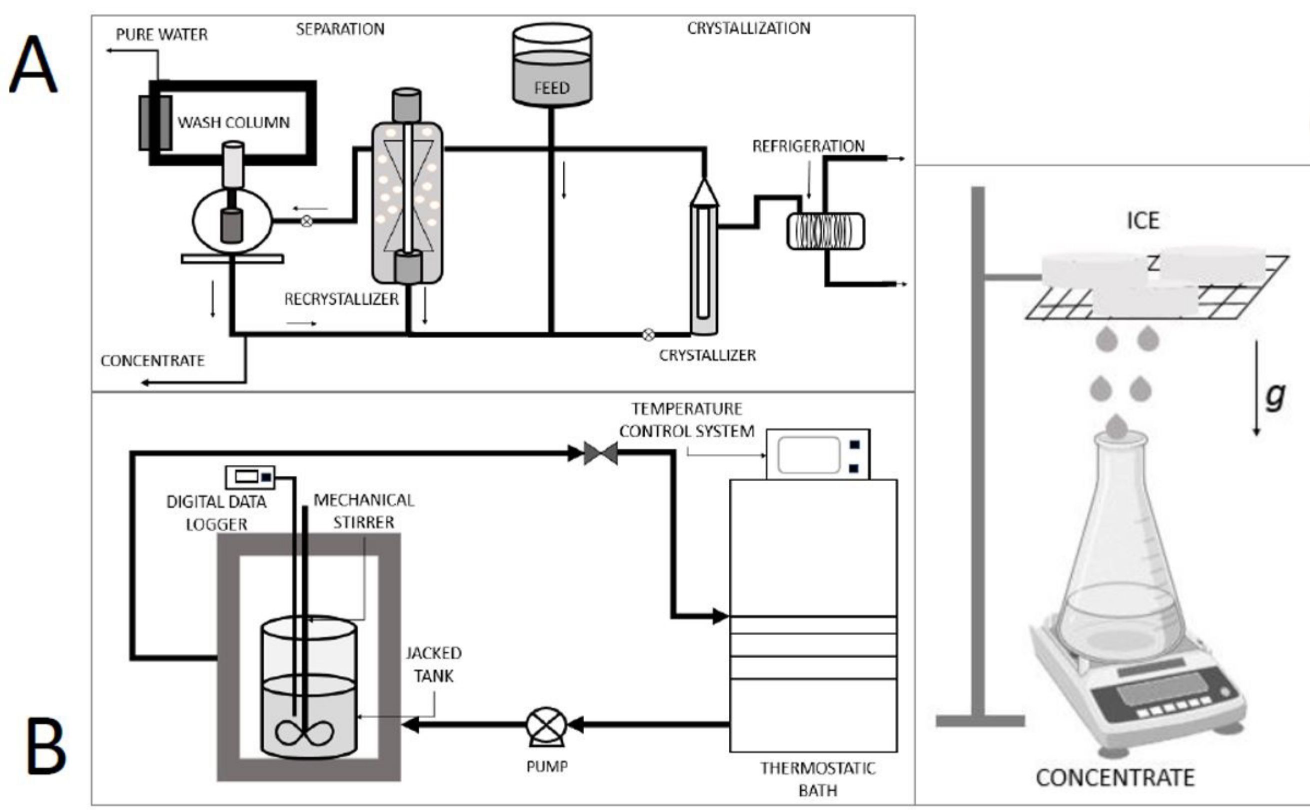

Figure 3. A: Suspension freeze concentration scheme. Adapted from Aider & Halleux (2009) and Prestes et al. (2022); B: Vertical progressive freeze concentration scheme. Adapted from Muñoz et al. (2019); C: Block freeze concentration system.

milk by Muñoz et al. (2019), proving to be an effective method to concentrate milk and offering interesting energy savings, when compared to the suspension method and nutritional preservation of the concentrated skimmed milk. In this experimental vertical system, the liquid matrix is placed in an agitated tank with a cooling jacket. The low temperature causes the formation of an ice layer on the cooling walls of the tank and a mechanical stirring is applied to decrease the solute accumulation in the ice fraction (Figure 3B) (Ojeda et al., 2017).

In the block freeze concentration technique, the liquid food is frozen and partially thawed by the assisted gravitational defrost method to separate ice and concentrate fractions (Zambrano et al., 2018). At a controlled temperature, the ice block performs as a solid carcase and, by the phenomenon of diffusion, the concentrate is drained (Aider & Halleux, 2009) (Figure 3C). About all the freeze concentration methods, this technique is promising due the facility of operation, simpler equipment and the lower total cost, with diverse applications and studies in the dairy sector (Aider & Halleux, 2009; Barros et al., 2022a; Camelo-Silva et al., 2022a; Liz et al., 2020; Canella et al., 2020; Morison & Hartel, 2018; Muñoz et al., 2018; Sánchez et al., 2011a).

Effect of freeze concentration process in dairy products

Studies and equipment development about freeze concentration of dairy products have resulted in knowledge about the performance of dairy fluids at low temperatures, the impact on the milk physicochemical properties and the influence on the behavior of the main components such as lactose precipitation, protein conformation, and fat dispersion in both the concentrate and the ice fraction (Balde & Aider, 2016; Barros et al., 2022a, 2022b; Muñoz et al., 2019; Sánchez et al., 2011a).

During the concentration, there is an increase of soluble solids in the concentrated dairy fraction, with a tendency to increase the viscosity of the concentrate. The high concentration of caseins and their consequent dehydration causes an increase of micelles volume and in the inter-micelles interaction, which strongly contribute to the viscosity of milk. Any chemical or physical effect, as well as the concentration, that may change the aggregation state of casein micelle, certainly will modify the viscosity of milk. As a result, the increase of the viscosity is inversely proportional to the ability of separate the concentrate from the ice fraction, which limits the concentration efficiency (Balde & Aider, 2016; Balde & Aïder, 2017; Bienvenue et al., 2003). In addition, high milk soluble solids content (proteins, fat, or lactose) may affect the crystallization and development of pure ice crystals, affecting the efficiency in the separation step and restricting the phenomenon of heat and mass transference.

Alternative studies employing previous treatments to remove fat from whole milk or directing skimmed milk to freeze concentration were carried out to reduce the effect of fat during the concentration and improve the efficiency of ice crystals separation (Aider & Ounis, 2012; Balde & Aïder, 2017; Barros et al., 2022b; Camelo-Silva et al., 2022a; Muñoz et al., 2019). Compared to skimmed milk, the fat content in the dairy matrix increases the resistance in removing the ice fraction of concentrated whole milk due to the interaction/adsorption of caseins with fat globules. This interaction is responsible for the formation of large particles and the presence of a clumped that interferes in the separation of ice crystals (Tribst et al., 2020). In addition, the milk fat fraction can also influence the lactose distribution in the concentration process, directing the high lactose content in the ice fraction, which contains most hydrophilic compounds (Aider & Halleux, 2009).

The milk color is one of the physical parameters that most influence sensory acceptance, mainly whiteness. In the freeze concentration process, the whiteness and luminosity can be improved according to the conformation of caseins in the concentrate. Comparing the physicochemical parameters of skimmed powdered milk from traditional concentration methods (evaporation) and alternative processes before drying, such as the freeze concentration, Balde & Aïder (2017) obtained products with high luminosity, good flow, and heat stability of milk powders from a previous freeze concentration. The high whiteness and luminosity can be attributed to an aggregation of denatured whey proteins and casein micelles, forming large particles. Compared to the traditional evaporation, significant differences can be attributed to the high temperatures involved in this process, which causes the formation of melanoidins or Maillard Reaction Products (MRP), with a typical caramel-brown color (Perusko et al., 2021).

Freeze concentration processes can be easily applied in dairy manufacturing with the purpose to produce stable concentrated products with high dry matter content without the need to enrich the formulation with whey proteins, powdered milk, or additives to improve the color of dairy products (Balde & Aïder, 2017). Studies report that freeze concentration is an important technology to promote high lightness, viscosity, and overrun for ice creams with skimmed milk freeze concentrated (Camelo-Silva et al., 2022a) and replaced with whey concentrate (Barros et al., 2022a) (Table 1). In addition, recent researches indicate that the high dry matter of the concentrate can offer a large concentration of nutrients and substrates for the development of probiotic cells and be a good carrier in dairy products, ensuring a high quality and functional value (Camelo-Silva et al., 2022b; Canella et al., 2018; Liz et al., 2020; Muñoz et al., 2018).

3.2 Membrane separation process

Membrane separation is an emerging food processing technology that can provide milk preservation at low temperatures, increasing the shelf-life (Carter et al., 2021). In dairy manufacturing, this popular separation process can be used to improve the microbial quality of dairy products and maintain the nutritional and functional properties of milk bioactive compounds (Henriques et al., 2020; Prudêncio et al., 2014; Verruck et al., 2019b). Compared to traditional separation processes, the membrane technology is favored due to the operation at moderate temperatures, pressure, and high selectivity during the procedure, which do not change organoleptic characteristics of dairy products (Galvão, 2018; Kim & Min, 2019; Velpula, 2017). In addition, unlike conventional methods, membrane separation is a very attractive alternative technology due to the required temperature does not involve solvent phase change, which increases energy savings in this concentration process and represents an energy-efficient alternative to the concentration of dairy products (Table 1) (Bahnasawy & Shenana, 2010; Faucher et al., 2021; Marx & Kulozik, 2018; Prudêncio et al., 2014).

The principle of this process occurs similarly to a pressure filtration system with membranes (thin film) to separate two solutions acting as a selective barrier. The separation is based on the permeability of membrane pores, separating immiscible solids and soluble solids, and the systems of separation are developed according to the direction of feed flow, which can be tangential or perpendicular. When the liquid passes through the membrane in a single direction, pores clogging may occur. On the other hand, in the tangential system, there are two directions of current passing through the membrane: one that flows parallel to the membrane removing the trapped solids, and the other purified, that passes through it. In this system, the parallel flow assists in the removal of particles that could clog the membrane pores. After passing through the pores, the liquid matrix is separated into two fractions: the permeate or microfiltrated, which is the liquid that passes through the pores, and the retentate (Figure 4). This fraction contains a higher concentration of solids that have a bigger size than the pore diameter of the membrane (Bahnasawy & Shenana, 2010; Valencia et al., 2018; Verruck et al., 2019b).

The selectivity in separation depends on the process conditions and the type of membrane, relating the pore size and molecular-weight cutoff. The separation procedure can be classified as Microfiltration (MF), Ultrafiltration (UF), Nanofiltration (NF), and Reverse Osmosis (RO) (Figure 5) (Carter et al., 2021). In dairy manufacturing, beyond to being used for the separation of solid milk components, MF is widely applied in reducing microbiological counts, with the biggest pore size (0.1-10 μm) and the lowest process pressure (0.01-0.2 MPa). According to specific pore size, micellar caseins (50-500 nm), whey proteins (3-6 nm), lactose (1 nm), minerals and water can permeate the membrane, while fat globules (10 μm) are separated, and bacteria (10-100 μm), spores (1 μm), and somatic cells are removed (Carter et al., 2021; Schäfer et al., 2018; Smith, 2013; Verruck et al., 2019b). In this case, microfiltration is important in ensuring safe food for consumption, removing spores that are not inactivated in the milk pasteurization/sterilization process, in addition to extending shelf life, since somatic cells induce lipolytic and proteolytic milk reactions, damaging the sensory characteristics, color, flavor, and texture (Ma et al., 2000; Verruck et al., 2019b).

For UF processes, the pore size ranges from 10 to 100 nm, operating at pressures of 0.1 to 1.0 MPa and, in milk concentration, proteins and fat fraction are retained providing a retentate rich in the dry matter that can be used in the production of high added value concentrated products. The permeate is composed of water, minerals, and lactose, which can be isolated and purified (Carter et al., 2021; Smith, 2013; Verruck et al., 2019b). The NF (1-10 nm of pore size and pressure of 1.5-3.0 MPa) is a fractionation technique with a concentration of substances having a molar mass between 100 and 1000 Da (g mol^{-1}) and are applied for whey processing in order to increase protein content (Carter et al., 2021; Marx & Kulozik, 2018; Merkel et al., 2021; Prudêncio et al., 2014; Smith, 2013). RO is a concentration technique, with the smaller pore size (< 1 nm) and the higher processing pressure (3.0-5.0 MPa), which are related to increasing the stability and the shelf life of whey concentrates (Marx et al., 2018; Marx & Kulozik, 2018).

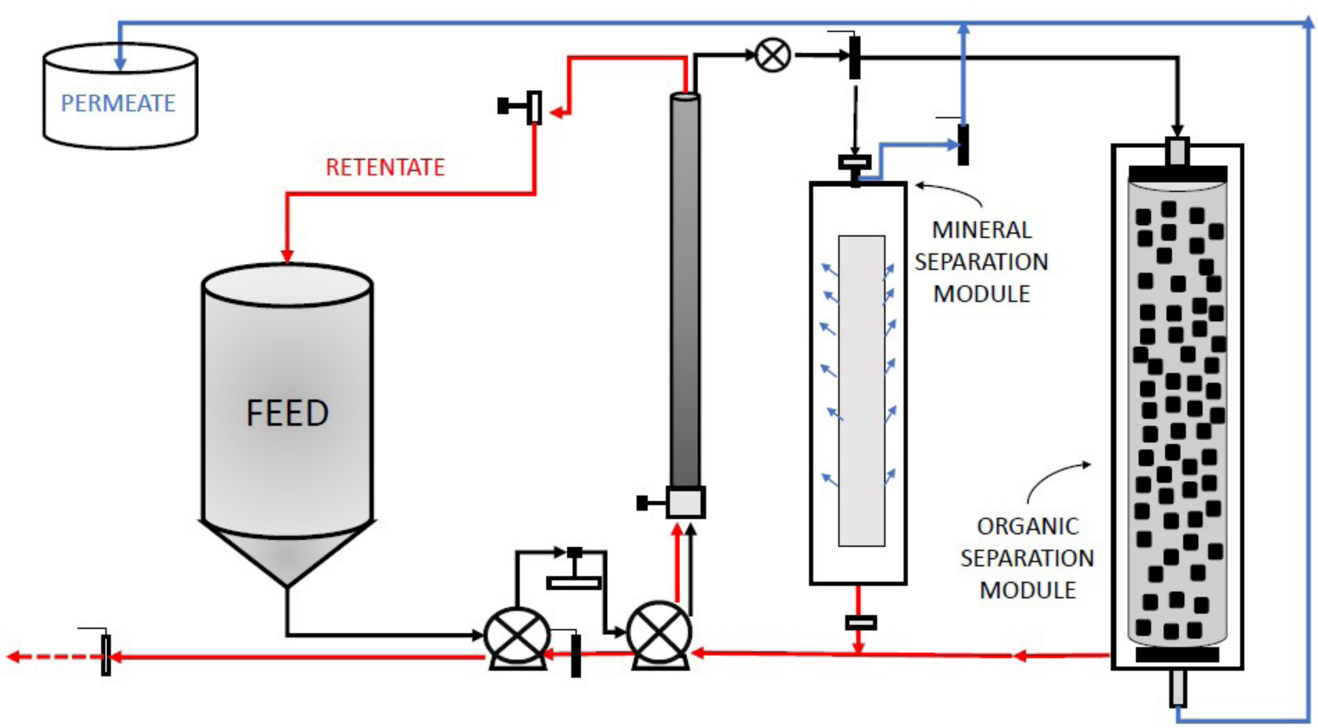

Figure 4. A schematic overview of the membrane separation method.

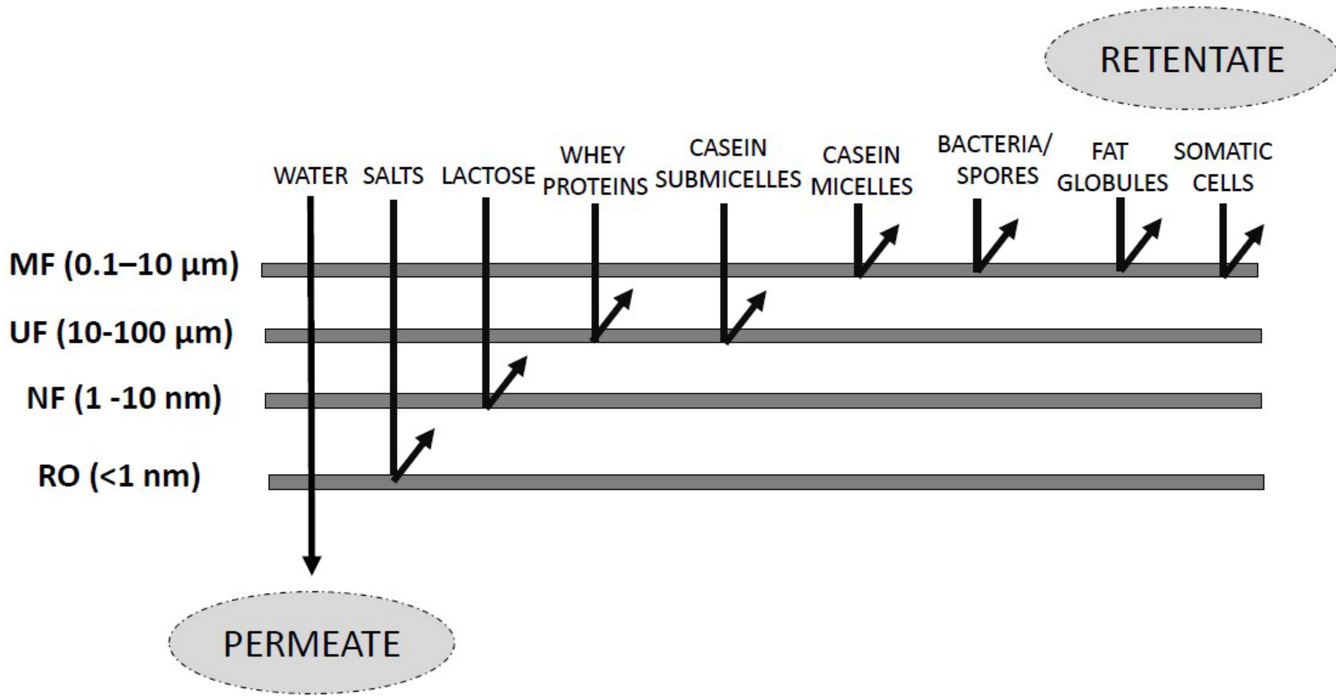

Figure 5. Passed and rejected dairy components based on membrane pore size. Adapted from Carter et al. (2021). Note: MF = microfiltration, UF = ultrafiltration, NF = nanofiltration, RO = reverse osmosis.

Effect of membrane separation processes in dairy products

Membrane separation systems at high-pressure processing have become an interesting alternative to concentrate dairy products, since this technology, in addition to acting on the removal of pathogens and spores, can reduce the loss of nutritional compounds, maintain the maximum of milk properties, enable high flow rates and improve the yield (Stratakos et al., 2019; Verruck et al., 2019b).

The separation of the milk protein fraction (caseins and whey proteins) is gaining considerable attention from dairy and beverage manufacturers due to its unique protein profile and functionality. Membrane separation technologies, in special the MF and UF, are promising in cheesemaking, which provide a micro/ultrafiltered milk that can be used for protein standardization and improve the texture and yield of cheeses (Carter et al., 2021; Faion et al., 2019). In a Peccorino cheese production from ultrafiltered sheep's milk by Faion et al. (2019), the ultrafiltered milk provided a nearly fourfold increase in protein (37%) and fat (29%) content, increasing the cheese yield by 17%. In addition, the time and moderate temperature of the ultrafiltration process (22 °C for 30 min) can favor the development of lactic acid bacteria for fermented dairy products (Table 2).

In the permeate, minerals and lactose fraction can be affected due to protein interactions and deposition on the membrane surface. According to the specific pore size, these interactions result in a gel layer formation, clogging the pores and exerting resistance to the passage of water, minerals, and lactose, which contains low molecular mass. For minerals, only two-thirds that are bound with micellar proteins (usually calcium phosphate) remain retained in the membrane, while the soluble ones are permeated through it (Faion et al., 2019). Minerals that pass with the whey fraction by interactions or in the form of free ions and salts, may impact the functional properties of the permeate, in its buffering capacity and influence the cheesemaking (Carter et al., 2021; Marx et al., 2018; Verruck et al., 2019b). In addition, the temperature in membrane separation can influence the distribution of constituents and physicochemical properties of milk. According to France et al. (2021), MF of skimmed milk was performed at 4, 8, and 12 °C. During the separation, the mechanical and thermal energy requirements during the MF of skimmed milk were strongly dependent on processing temperature (at 4 °C = 27.6 KWh, at 8 °C = 24.6 KWh and 12 °C = 22.9 $\times$ 10^{-3} KWh) with the highest energy requirements for UF at 4 °C due to the high viscosity and the generation of heat during pumping, increasing the energy required to maintain the lower temperature. For protein contents, the fouling of the membrane caused by casein micelles decreased the concentration of β-casein, β-lactoglobulin and α-lactalbumin in the permeate throughout MF. However, the dissociation of β-casein into the whey fraction is higher at lower temperatures allowing for increased partitioning thereof into the permeate at 4 °C (Coppola et al., 2014). The MF at lower temperatures may enable the production of next-generation dairy streams with novel protein fractions (France et al., 2021).

During the flow of milk through the membranes, the applied pressure minimally affects the protein content and fat globules when compared to a concentration in processes with high temperatures and mechanical impacts (Jukkola et al., 2018). The process must be monitored to maintain a suitable functioning of the membranes due to possible cake formation

Table 2. Recent studies on alternative technologies applied to dairy concentration.

Process	Dairy product	Conclusions	Authors
Freeze concentration	Ice cream	The concentrated skim milk provided high viscosity and overrun. The total solids and protein content were high, influencing in the color of ice cream, with high lightness.	(Camelo-Silva et al., 2022a)
Microfiltration	Skimmed milk	Lower temperatures (4, 8, 12 °C) influenced in the MF performance, with the highest membrane fouling at 4 °C and the highest energy requirements for separation at 4 °C. The permeate, also at 4 °C, obtained the highest solid content and whey proteins dissociated.	(France et al., 2021)
Freeze concentration	Ice cream replaced by whey concentrate	The presence of whey concentrate provided a greenish yellow color in ice cream, increased the viscosity, soluble solids and overrun. The replacement with whey did not affect the microstructure of the ice crystals.	(Barros et al., 2022a)
Ultrafiltration	Milk proteins	Development of bipolar membrane electrodialysis coupled with ultrafiltration enabled the concentration of caseins in their soluble form (caseinates) or precipitated without obstructing the pores of the membranes.	(Mikhaylin et al., 2018)
Freeze drying	Petit Suisse cheese	Rheological properties slightly differed between fresh and freeze-dried samples after rehydrating. This concentration method maintained the color properties and influenced in the microstructure of protein conformation, changing the product viscosity.	(Silva et al., 2021)
Freeze concentration	Fermented lactic beverage	Whey concentrate was used to develop a probiotic and symbiotic (probiotic + inulin) fermented beverage. The use of whey concentrate and inulin contributed to an increase in apparent viscosity and soluble solids. The concentrate provided high macro and micronutrients to probiotic cells development.	(Canella et al., 2018)
Ultrafiltration	Greek-style yogurt	A Greek-style yogurt was developed with the retentate of milk ultrafiltration and avoid an acidified whey from permeate and compared to a yogurt ultrafiltered. Both samples obtained similar protein (~10%) and solid content (~17%) and the energy requirement was higher due to the membrane fouling during the procedure.	(Valencia et al., 2018)
Freeze concentration	Probiotic microcapsules produced with goat's whey concentrate	Powders produced from goat's whey concentrate and inulin (with high total solids content) presented water solubility and cohesiveness and provided high lightness, a greenish color and a good stability of probiotic cells at cold storage.	(Liz et al., 2020)
Freeze drying	Camel milk powder	Freeze-dried camel milk powder had the highest dispersibility (67.15%), solubility (88.77%) and the lowest acidity (0.193% vs 0.211%) when compared to a spray drying process, attributing to the non-thermal concentration process a high stability of camel milk metabolites.	(Deshwal et al., 2020)
Freeze concentration, reverse osmosis	Skimmed powdered milk	The powdered milk from a previous concentration by freeze concentration and reverse osmosis provided high solubility, whiteness and lightness when compared to an evaporation process. In addition, the powder particle size was highest for the two alternative concentration processes.	(Balde & Aïder, 2017)
Freeze drying	Symbiotic yogurt	Survival rates of *L. plantarum* added in microcapsules were higher after freeze-dried when compared to free cells (91.2 *vs* 61.7%). Cryoprotected *L. plantarum* microcapsules tolerate the freeze drying process, classifying the yogurt powder as a probiotic product for 10 weeks at 25 °C.	(Jouki et al., 2021)
Ultrafiltration	Cheese with concentrated sheep's milk	The sheep milk ultrafiltration (22 °C for 30 min at 2 bar) increased the Peccorino cheese yield (17%) and provided an approximately fourfold increase in the protein (37%) and fat (29%) content.	(Faion et al., 2019)
Nanofiltration	Sweet and acid whey	Nanofiltration and electrodialysis provided 88% and 91% desalination of sweet and acid whey, which provided a high removal of minerals and organic acids with a decrease by 88% of lactic acid.	(Merkel et al., 2021)

on the surface or pore blocking caused by casein micelle sizes (Verruck et al., 2019b). According to studies, whey proteins of pasteurized skimmed milk (> 78 °C) can denature and form aggregates with minerals or can adhere to casein micelles, clogging the membrane pores (Carter et al., 2021; Saboyainsta & Maubois, 2000; Verruck et al., 2019b). This problem can affect the flux and modify the composition properties of the permeate and the retentate, which will not contain casein micelles but a product similar to milk protein concentrate due to the ratio of casein to whey protein (Carter et al., 2021). Recently, researches were developed to reduce the membrane fouling caused by casein micelles during the milk ultrafiltration (Mikhaylin et al., 2016, 2018). A bipolar membrane electrodialysis was coupled with ultrafiltration, allowing the production of H^+ and OH^- ions from water under the application of an electric current. The milk is acidified in the electrodialysis module and caseins are precipitated and separated from whey proteins, without clogging. In addition, the base generated by the bipolar membrane can be applied in the conversion of insoluble caseins micelles into their soluble form of caseinates, without the use of chemicals and is considered a more environmentally sustainable process (Mikhaylin et al., 2018). Sustainable strategies are also developed for application in ultrafiltration and nanofiltration of whey, with an obtention of nonacidified whey permeate before further application and reducing this major by-product generated by dairy industries (Faucher et al., 2021; Merkel et al., 2021). Although membrane fouling is something recurrent, these study alternatives can improve the efficiency of the membrane separation process, which is currently one of the emerging technologies with great potential to replace traditional dairy concentration methods in large-scale.

3.3 Freeze-drying

The freeze-drying technology, or lyophilization, is a concentration process based on the phenomenon of water sublimation of the food composition. At low temperatures, the water fraction is separated from the food by crystallization below its triple point (0.01 °C) and then is directly transformed from the solid-state to the vapor phase at high pressures (approximately 611 Pa) (Bando et al., 2017; Waghmare et al., 2021). The concentration at freezing temperatures limits the damage of thermolabile compounds and is an advantageous technology to retain more taste, aroma, and color when compared to other concentration methods, being also an interesting alternative to concentrate dairy products, providing an increase of their quality (Deshwal et al., 2020; Vincenzetti et al., 2018; Zhu et al., 2020).

The market of freeze-dried products is increasing; however, it is necessary to reduce the high energy consumption of the process, ranging from 3820 to 5500 $kJ.kg^{-1}$: the highest energy requirement among all the concentration processes (Table 1) (Bando et al., 2017; Keselj et al., 2017). Compared to conventional drying, which dehydrates foods in a single stage, the freeze-drying time is longer and consumes large amounts of energy (almost double to remove 1 kg of water) (Duan et al., 2016). In addition, energy is required to freeze the food, maintain the high pressure, sublimate water crystals and condense water vapor. This concentration process becomes expansive and is usually applied in high value-added products and makes the processing of common and accessible concentrated products unfeasible (Garcia-Amezquita et al., 2016; Waghmare et al., 2021).

The design of a freeze-dryer is composed of four basic components: drying chamber, vacuum pump, heat source, and condenser (Figure 6). For milk processing, it becomes usual to apply a vacuum freeze dryer. In this method, freezing can be done directly in the freeze-dryer or other equipment that makes this step possible. The vacuum system is a combination of a water circulation pump and oil-sealed pump. During the initial stage of the vacuum process, the air is removed by the high-power oil sealed pump causing a decrease in pressure. After this procedure, the vacuum is maintained by a low-power pump. For the frozen water fraction to change state, passing from the solid-state directly to steam, a heating system is needed to raise the temperature and prevent the change from the solid to the liquid phase. In this system, it can contain plates with steam circulation or hot water at 120 °C. Cooling for steam condensation is equipped by a system with liquid refrigerant circulating on plates behind or on the sides of the freeze dryer (Duan et al., 2016; Garcia-Amezquita et al., 2016; Waghmare et al., 2021).

Effects of freeze-drying process in the dairy products composition

Due to the constant consumer demand for foods with higher functional and nutritional quality, the food formulation has been adapted with the replacement of natural components by freeze-

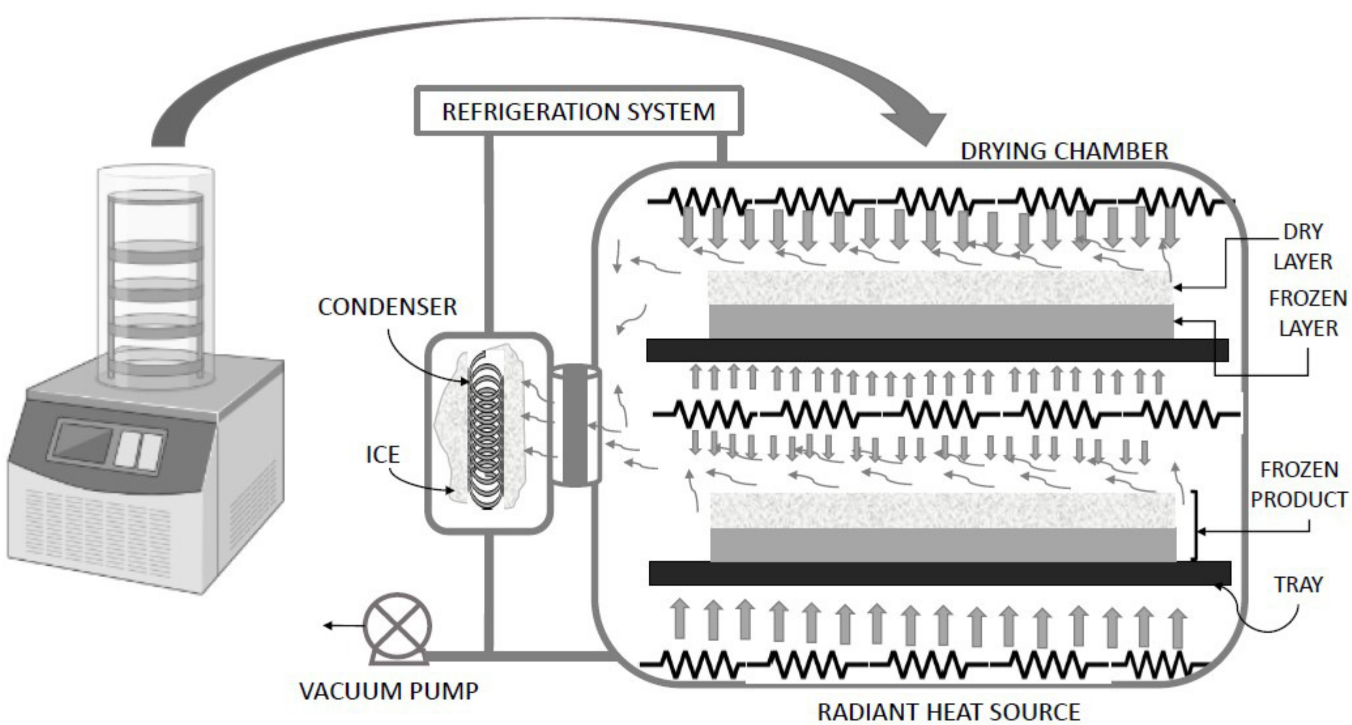

Figure 6. A typical freeze-dryer system. Adapted from Garcia-Amezquita et al. (2016).

dried products. As well as other emerging technologies, freeze-drying in addition to concentrating food can also be considered a method of preserving food products (Deshwal et al., 2020; Vincenzetti et al., 2018). Due to the low water activity, microbial development and enzymatic oxidations are delayed, allowing the concentrated product to be stored for a long time at room temperature. Furthermore, especially in dairy manufacturing, the use of low temperatures during the concentration step enables to maintain the color and flavor of the product (Deshwal et al., 2020; Duan et al., 2016; Silva et al., 2021; Vincenzetti et al., 2018; Waghmare et al., 2021).

The first investigation about the effect of freeze-drying on raw milk metabolites was proposed by Zhu et al. (2020). About the contents of some organic acids, amino acids, and dipeptides, slight changes were detected, however, the authors pointed that these alterations may happen as a result of the incomplete re-dissolution process of the freeze-dried milk powder rather than the freeze-drying process. The concentration of orotic acid, a fatty acid naturally occurring in raw milk, was stable after the freeze-drying treatment. To the low storage temperatures (4 °C and -20 °C), metabolites barely changed when stored in a freezer over a long period, relating the freeze-drying as an effective concentration and preservation method for milk concentration with minimal changes on the metabolites.

Effects in milk composition were also proposed by Deshwal et al. (2020), who compared camel milk powder produced by freeze-drying with the traditional method of spray drying (Table 2). According to this study, freeze-dried camel milk powder had the highest dispersibility (67.15%) and solubility (88.77%). This can be attributed to this process, which makes the products lighter than other drying methods. Low solid feed during freeze-drying results in porous particles as a large amount of water is removed during the stages of drying. The lowest acidity was obtained in freeze-dried camel milk powders when compared to the spray drying method (0.193% *vs* 0.211%), which can cause an increase of minerals precipitation, lactose degradation, and Maillard reactions with a considerable and irreversible pH decrease. These reactions, due to the application of high temperatures, also decreased the calcium and iron contents of camel milk powders when compared to a freeze-drying process (0.011-0.012 g/kg *vs* 13.71 and 15.33 g/kg, respectively), highlighting the important maintenance of nutritional and functional compounds in a concentration by freeze-drying. The structure of milk fat globules can also change according to specific concentration methods. In a study about the effects of freeze-drying and spray drying on the microstructure of milk fat globules, Yao et al. (2016) pointed that the surfaces of some fat globules after freeze-drying became thicker than those from raw milk and after spray-drying. This technology can cause the formation of irregular flaky translucent sheets with sharp edges, whereas spray-dried fat globules are spherical particles (Zhu & Damodaran, 2011). This phenomenon can be explained due to the amphiphilic phospholipids that tend to accumulate on the surface during freezing and then, the fat globules stick together during the freeze-drying process. In addition, the freeze-drying method caused an increase in fat globules, explained by the authors due to the formation of ice crystals and possible repulsion of foreign material away from the interstitials, causing globule aggregation. In addition, the osmotic pressure of the globules may have caused recombination in larger globules (Yao et al., 2016).

The concentration by freeze-drying also can influence the physicochemical properties of a dairy product. In a development of a freeze-dried *Petit Suisse* cheese, Silva et al. (2021) pointed that this concentration method can influence the conformation of protein networks of the product composition (Table 2). Microscopy of the freeze-dried samples showed structures with large porosities and low agglomeration, indicating minimal interaction between the particles. This characteristic is attributed to the sublimation process of the larger ice crystals that filled the formed cavities with available water in the protein matrix. The formation of large pores in a protein network may be caused by an increase in the positive electrical charge of casein micelles at pH < 4.6, reducing intercellular interactions and resulting in the formation of an opening (porous). The protein dehydration promoted by freezing can change the textural and rheological properties, attributing different viscosities after rehydrating. In addition to changes in the composition and dairy structure, freeze-drying has become an interesting and useful method to extend the shelf life of probiotic bacteria in dairy products. Due to damage to cell membranes by the formation of ice crystals and decreasing cell viability, microencapsulation is an alternative for the incorporation of probiotics into the medium, which was proposed by Jouki et al. (2021) in development of a symbiotic freeze-dried yogurt powder using microencapsulation of *L. plantarum* (Table 2). After freeze-drying, the survival rate of probiotic cells ranged from 67.1-91.2%, with minimal effect of this process on microencapsulated cells (9.8-10.6% loss). *L. plantarum* microcapsules enriched tolerated the freeze-drying process, featuring the yoghurt powder as a probiotic product for 10 weeks at 25 °C.

Therefore, the use of freeze-drying technology, as well as all alternative concentration technologies, allows the development of functional dairy products, with their nutritional properties maintained and quality enriched, pointing out these unconventional methods as potential substitutes in the concentration of dairy products in the dairy sector.

4 Future perspectives in the replacement of traditional technologies by emerging processes in the concentration of dairy products

The concentration of dairy products becomes one of the main challenges for the food industry, due to the guarantee of products of high quality and linked to a low-cost process. Recent studies, even if carried out on a small scale, show an industrial potential in replacing traditional concentration technologies by emerging non-thermal alternatives.

New research and equipment refinement must be conducted with adaptations of promising laboratory results for large-volume production, as well as the development of specific equipment at low costs, in addition to increasing speed, yield, and soluble solids content in the concentration/separation steps, which can also be developed into a single operation in the same equipment's section. These improvements would be fundamental mainly for freeze-drying and block freeze concentration technologies that carry out an efficient concentration process, but with low yields and, therefore, are not viable in industrial production today. The application of non-thermal concentration processes, in addition to ensuring a high nutritional and sensory quality to the dairy product, can be linked to processes with an environmental appeal, with the reduction of the use of non-renewable energy resources such as the block freeze concentration, and alternatives for the treatment of by-products from milk manufacturing in recent aspects of membrane separation processes.

With constant research and development in the dairy sector, the industrial replacement of traditional concentration technologies by non-thermal methods becomes a future potential and a differential in the final quality of the product, winning consumer preference and becoming an interesting alternative in the competitive industrial sector from an economic and technological perspective.

5 Conclusion

Concentrated dairy products are one of the most important manufactured goods for the dairy industry due to the high added value, reduced volume, and lower transport costs. In addition, concentration processes increase the shelf-life, which can expand distribution logistics. Evaporation and spray drying processes are thermal concentration methods that are still traditional in the dairy industry, however, they are unfeasible processes due to the requirement of high energy expenditure and the decrease of nutritional, functional, and sensory properties of milk and dairy products. Nowadays, studies about the application of emerging non-thermal concentration technologies have been explored as means to decrease the adverse effects of traditional processing and present promising alternatives for dairy industries. Among the emerging dairy concentration methods reported in this review, membrane separation processes are advantageous due to enabling high flow rates, capable of removing bacteria and spores from the matrix, low energy expenditure, and reduced total costs. On the other hand, freeze concentration and freeze-drying, however advantageous in keeping bioactive compounds in milk and enable an increase in the quality of concentrated products, become energy-expensive, have a low yield, and are unfeasible for large-scale production. Recent studies involving the application of unconventional processes in the concentration of dairy products are fundamental for the industrial sector, which makes it possible to implement emerging processes on a large scale and increase the quality of commercialized concentrated products.

Conflict of interest

The authors have no conflicts of interest.

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