Chapter

Membrane Applications for Lactose Recovering

Antónia Teresa Zorro Nobre Macedo,
Joana Filipa Oliveira Monteiro,
David José Chaveiro da Silva Azedo,
Elizabeth da Costa Neves Fernandes de Almeida Duarte
and Carlos Dias Pereira

Abstract

Cheese whey, the co-product from cheese making processes, is a natural and cheap source of high value compounds, mainly proteins, small peptides, oligosaccharides, lactose, and minerals. Lactose is the main component (about 90%) of the dry extract of cheese whey. This carbohydrate has plenty of application in the food and pharmaceutical industries due to its relative low sweetening power, caloric value, and glycemic index. Besides, lactose is currently available for diverse physicochemical properties, namely particle size, bulk density, distribution, and flow characteristics, extending its use for a larger range of applications. Recovery of lactose from cheese whey can be carried out through different processes, such as membrane processes, crystallization, anti-solvent crystallization, and sonocrystallization. This chapter aims to furnish a deep insight into the performance of membrane processes for lactose recovery from cheese whey.

Keywords: cheese whey, lactose recovery, membrane processes, nanofiltration, ultrafiltration

1. Introduction

Dairy industry is one of the major food processing industries in the world, manufacturing a broad range of different products. Therefore, it generates large amounts of by-products during the processing of milk and manufacture of dairy products (e.g., cheese, butter, and yogurts), leading to problems of their management/utilization [1].

Cheese whey is the most abundant co-product in the cheese-making and casein industries. It contains about 65 g L^{-1} of dry matter, being lactose the main component (70–80%), proteins (9%), corresponding to 20% of all milk proteins, and minerals (8–20%) and, to a much lesser extent, hydrolyzed peptides from casein-k, lipids, and bacteria, which resulted from cheese manufacturing [2, 3]. Generally, for each 100 kg of milk, around 10–20 kg of cheese is manufactured, and 80–90 kg of liquid whey is released [4]. According to Food and Agriculture Organization Corporate Statistical Database (FAOSTAT), more than 114 million tons of whey were produced worldwide in 2013, with Europe producing 63 million tons in that

year [5]. Data from the European Whey Products Association (EWPA) indicated that about 6 million tons of whey (dry matter) were produced in the European Union in the year 2015 [6]. In spite of these larger volumes produced, only around 50% of the whey annually produced in the world is valorized into different added-value products. This is because, although cheese whey is an inexpensive and abundant source for developing new added-value products (e.g., foods, pharmaceuticals, and energy), its low solid content makes it difficult for direct utilization [4]. Therefore, for recovering any of its components, such as the lactose, several processes, mainly separation processes, should be used. The intended final use of lactose determines the process that should be used for its separation from cheese whey.

2. Membrane processes

Membrane separation is a filtration process based on the use of membranes for the separation of dissolved or colloidal solids in liquid mixtures, or the separation of small components in gaseous mixtures. A membrane is a permselective barrier between two phases (feed/retentate) and permeate, which preferably allows the permeation of a component (or components) of the feed retaining others, leading to their separation, purification, or concentration. The difference in permeability (membrane transport) between the components of the mixture is due to differences in size (ratio between mean pore radius of membrane and size of solute to be separated) and/or chemical selectivity for membrane material (relationship among chemical characteristics) [4].

These processes differ from frontal filtration in the following characteristics: (1) the particle size they separate; (2) tangential rather than dead-end mode of feed introduction; and (3) use of membranes, in spite of depth filters. Therefore, these processes allow to expand the scope of frontal filtration for separating components of smaller dimensions (less than 1 μm). The parallel flow limits the accumulation of substances retained on the membrane due to shear stress and two different product streams are obtained (**Figure 1**). When using membranes, the components are retained to the surface in a thin film, called the active layer or skin, and so higher retention rates are possible [4, 7].

Membrane separation processes can be classified according to the driving force that controls the mass transfer rate of the individual components from one phase to another. These driving forces can be of several natures such as concentration gradients, temperature, pressure, and external force fields. The main processes used at an industrial level are pressure-driven processes, such as microfiltration, ultrafiltration, nanofiltration, and reverse osmosis [8, 9]. In these processes, by applying a pressure, the solvent and some solutes freely permeate the membrane, while others are retained to varying extents, depending on various factors, such as solute, membrane characteristics, operating parameters, or others [8, 9]. The size of the particle or molecule to be separated as well as its chemical properties determines the

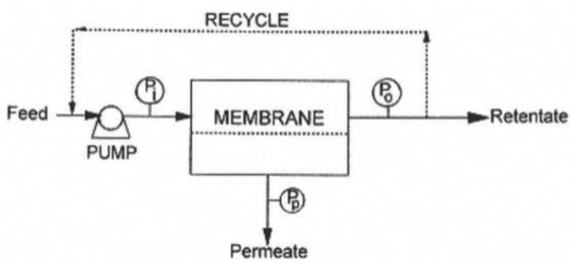

Figure 1.
Diagram of a membrane separation process [4].

structure (porous or dense, pore size, and pore size distribution) of the membrane to be used. The nature of the solvent (aqueous or organic), the cleaning method, the applied pressure, and the temperature influence the type of membrane material [10]. When it progresses toward microfiltration, ultrafiltration, nanofiltration, and reverse osmosis, the size or molecular weight of particles or molecules that are retained by the membrane, pore size, and porosity decreases. This means that the hydrodynamic resistance of the membranes to mass transfer is increasing, requiring higher applied pressures to achieve the same permeation fluxes.

Following water and wastewater treatment, the food industry ranks second in applications of those processes. Most applications are in the dairy industry (production of whey protein concentrates; milk protein standardization), followed by the beverage (wine, beer, vinegar, and fruit juice) and egg product industries [8, 11]. In the food industry, the application of membrane separation processes provides several benefits, such as food safety, competitiveness, innovation, and environmental compatibility. Food safety through membrane processes can be achieved, for example, by cold sterilization, using microfiltration. They are competitive with other concentration processes, for example, thermal processes, due to their lower energy consumption. In addition, they can be easily integrated into industrial plants due to ease of implementation, possibility of using compact modules, and good automation. So, these processes are currently present in several industrial plants, namely in the development of new value-added products, for example, from by-products (cheese whey or second cheese whey) and/or residues of the food industry. In addition, since only cleaning agents are used and the processes can be operated under mild conditions (pressure and temperature), they are recognized as green processes [3].

2.1 Membranes

The membranes can be manufactured with different types of materials (polymeric or inorganic), may have different structures (symmetrical or asymmetrical), and are usually commercialized in arrangements of membranes, with a high surface area per unit volume, called modules.

The nature of the material used is an important aspect of membrane processes because it can affect the behavior and performance and limits the use of a membrane, for a particular application. Regardless of its nature, that material must have good thermal, mechanical, and chemical stability; hydrophilicity or hydrophobicity; ease of manufacture on a wide variety of dimensions pores; modules; and configurations [4, 7]. In this respect, inorganic membranes made from ceramic materials are the most used, due to its higher thermal, chemical, and mechanical stability than polymeric membranes. These characteristics allow its use in a wider pH region and with different organic solvents. Furthermore, they are easier to clean and disinfect, since more concentrated solutions of strong acids and bases and higher temperatures can be used, keeping their life span. Some disadvantages of these membranes compared to polymeric ones are mainly associated with its higher cost, the need of using higher flow rates (greater energy consumption), and to the fact that, currently, does not exist in the market ceramic nanofiltration membranes with limit of separation less than 250 Da [12].

The classification of membranes according to their structure is shown schematically in **Figure 2**. Symmetrical membranes include microporous and homogeneous membranes (dense and nonporous). The thickness of the symmetric membranes can vary approximately from 10 to 200 μm, the resistance to mass transfer being determined by the total thickness of the membrane. Thus, the thinner the membrane, the higher the permeation rate [7]. These membranes are applied in microfiltration and can be classified, on an absolute scale, through their maximum

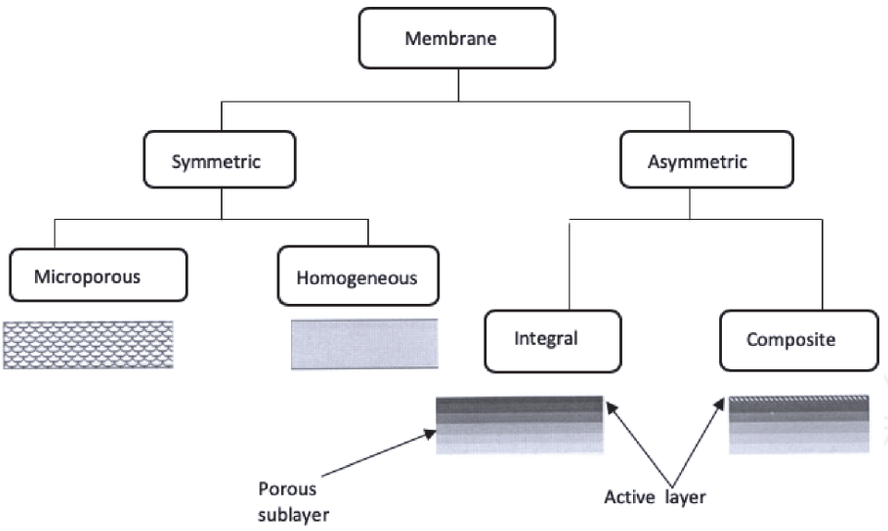

Figure 2.
Schematic representation of membrane structure.

equivalent pore diameter. Homogeneous membranes are mainly applied in gas separation. Asymmetric membranes have a structure consisted of a very thin film on their surface with a thickness in the range of 0.1–0.5 μm, called skin or active layer, which is based on a porous support layer, the thickness of which can vary between 100 and 200 μm [4, 7]. The separation occurs only at the surface, in the active layer, retaining components whose molar mass is greater than the molecular weight cut-off (MWCO) of the membrane, which is defined as the molar mass that is 90% rejected by this membrane. The manufacturing process of the membranes still leads to obtaining two different substructures: the integral asymmetric membrane design and nonintegral asymmetric membranes, the latter forming part of the composite membranes. Integral asymmetric membranes are obtained from a single polymer. Composite membranes, also called thin-film, thin, or ultrafine layer composites, are manufactured with a polymer (or other material) different from that used in the layer support and in several stages, which make it possible to optimize each of them, independently. These membranes are used in ultrafiltration, nanofiltration, and reverse osmosis.

The design of the modules is based on two types of membrane configurations: flat and tubular. Plate modules and spiral-wound modules involve flat membranes, while tubular, capillary, and hollow fiber modules are based on tubular membrane configurations. In general, an industrial membrane installation consists of the association of several modules, which are selected and configured in parallel or in series, depending on the production/specification of the final product. The selection of the module configuration, as well as the module arrangement, is based on several factors: economic considerations; type of application; ease of cleaning, maintenance, and operation; compactness of the system; and scale and possibility of replacing membranes.

2.2 Performance of membrane processes

The main parameters used to evaluate the performance of a membrane are the permeate flux that is a measure of its productivity and the apparent rejection coefficient, which allows us to estimate their selectivity. The permeate flux (J_v) is defined as the amount, in volume or mass, that passes through the membrane per unit area and time, that is,

$$J_v = \frac{V}{A \times t} \left(\text{m s}^{-1}\right) \quad (1)$$

where J_v is the volumetric permeate flux (m s^{-1}); V is the volume of the permeate (m^3); A is the membrane surface (m^2); and t (s) is the time required to collect the volume of permeate V.

The rejection coefficient is a measure of membrane selectivity for the separation of a given solute, which may be partially or totally retained by it, while the solvent freely permeates the membrane. The apparent (or observed) rejection coefficient, R, is defined as follows:

$$R = \frac{C_f - C_p}{C_f} \quad (2)$$

where C_f is the concentration of a particular solute in the feed, and C_p is the concentration of this solute in the permeate.

The apparent rejection coefficient depends on the experimental conditions, namely transmembrane pressure and feed circulation velocity. This coefficient is a dimensionless quantity, which can take values between 0 and 1, as the solute freely permeates the membrane or is completely retained by it, respectively. The latter situation corresponds to an ideal semi-permeable membrane.

The permeate flux and apparent rejection coefficient are influenced by several factors related to solute characteristics (size and shape, macro and micro solute coexistence), membranes (more hydrophobic/hydrophilic character, surface charge distribution, and surface roughness), operating parameters (transmembrane pressure, feed circulation velocity, and temperature), environmental conditions (pH, ionic strength, and osmotic pressure), and type of module (plane and tubular) [13]. These factors give rise to the resistive phenomena mass transfer across the membrane, referred to as concentration polarization and fouling, which can severely affect the performance of membrane processes. Another factor that can reduce the performance of membrane processes is the effect of osmotic pressure.

Concentration polarization consists in the formation of a concentration gradient in a thin layer near the membrane surface, caused by the accumulation of the retained species and leads to the initial decrease of permeate fluxes, which may also contribute to a reduced selectivity. It mainly affects those processes with larger pore membranes (higher permeate fluxes) such as microfiltration and ultrafiltration and can be minimized through the use of low pressures, high feed circulation rates, and low solute concentrations.

Fouling consists of pore obstruction (on or in the surface), caused by solute-membrane or solute-solute interactions, which mainly depend on the characteristics of the solutes, of the membrane, and of operating conditions and the type of module. This phenomenon can lead to a sharp reduction in permeate flux and can alter membrane selectivity [14]. In order to reduce the effects of fouling, various preventive methods can be used such as (1) use a suitable pre-treatment for the food (pre-filtration, pH adjustment, and adequate heat treatment); (2) select the most suitable membrane (narrow pore size distribution, hydrophobicity characteristics, presence of charged groups, or with certain functional characteristics on the membrane surface); (3) use the modules with spacers and work with high feed circulation rates or even at low permeate fluxes, by reducing the applied transmembrane pressure; and (4) use the rotary (or vibratory) modules, in which the membrane moves on a rotating cylinder, creating greater turbulence close to the membrane, compared with conventional tangential modules, while maintaining low shear rates within the fluid [15].

The effect of osmotic pressure in the decline of permeate fluxes is generally neglected in microfiltration and ultrafiltration since the solutes to be separated in these cases have very high molar masses. However, if the concentration of macromolecular solutes is very high, then this effect will have to be accounted for. The phenomenon is especially important in reverse osmosis and also nanofiltration, since in these processes, the solutes that separate are of low molar mass, so the osmotic pressures can be high, decreasing the effective pressure.

In addition, the performance of the overall membrane process should also take into account economic factors, such as membrane prices and shelf life, cleaning and disinfection reagents, and energy consumption.

3. Lactose recovery through membrane processes

In the industrial process that is currently used for lactose production, membrane separation techniques have already been introduced because lactose is currently recovered from the whey ultrafiltration permeate. The whey proteins separated have different and interesting applications (e.g., whey protein concentrates, WPC, or whey protein isolates, WPI), thus contributing to the valorization of cheese whey. The Commonwealth Scientific and Industrial Research Organization (CSIRO) developed a method for the possible commercial production of pharmaceutical quality lactose, which integrates the following operations: ion exchange (for calcium and magnesium removal), nanofiltration/diafiltration (for lactose separation, concentration, and purification), evaporation, crystallization, and chromatography, allowing not only to obtain high purity lactose, as well as mineral salts and calcium from cheese whey. This process has several benefits because through the use of nanofiltration/diafiltration, it is produced by a purified lactose concentrate, minimizing simultaneously the evaporation costs due to the reducing volume. Besides, the nanofiltration permeate can be subjected to reverse osmosis, producing water of good quality (e.g., for cleaning and diafiltration).

The recovery of lactose from cheese whey by membrane processes is mainly carried out by nanofiltration (NF) of the ultrafiltration permeates, due to their physical-chemical composition. Those permeates are composed of small solutes, being lactose the major compound of the dry matter, followed by several ions such as, sodium, potassium, calcium, magnesium, chloride, phosphate, and citrate. Therefore, the specific selectivity of NF to this type of solutes and its lower energy consumption, compared with other processes such as reverse osmosis and evaporation, has boosted their use in dairy [8, 16, 17] and other agroindustrial sectors.

One of the most important uses of nanofiltration is the production of whey-demineralized lactose concentrates in the food industry, or even, if enough purification is achieved, for pharmaceutical purposes. The demineralization of dairy fluids is very important to reduce their high salt content (8–20% of dry matter) [3, 18], which causes several difficulties in processing. A high salt content leads to slow lactose crystallization rate because it reduces lactose solubility in supernatant liquor during crystallization.

The major drawback of the NF process is the fouling caused by mineral precipitation of salts, namely calcium phosphates. Another reason for the decrease of permeate flux is the increase of osmotic pressure and concentration polarization, due to the accumulation of lactose and salts (sodium, potassium, and chloride) near the membrane surface, causing a reduction in the effective pressure [19, 20].

Guu and co-workers [21] found that the application of NF for sweet whey or UF permeates allowed to increase the production of lactose crystals by about 10 and 8%, respectively, for a VRR of 3.0. This behavior was attributed to the partial

demineralization of the permeate, especially in terms of the monovalent ions, sodium and potassium. These results raised the interest for the integration of NF membranes in the lactose production plants at the industrial level.

Rice and co-workers [22] carried out nanofiltration of ultrafiltration permeates using polyamide membranes NF270 and observed a severe flux decline during filtration at high temperatures and pH, due to calcium phosphate precipitation, because of its lower solubility in these operating conditions. Those authors suggested that if the pH of the feed was reduced, fouling could be avoided, despite changing the separation properties of the membrane.

Cuartas-Uribe and co-workers [23] studied the concentration of lactose from whey ultrafiltration permeates, combining concentration by nanofiltration with continuous diafiltration modes, and found that the best operating conditions were a transmembrane pressure of 2.0 MPa and a volume dilution factor of around 2.0 because a good removal of chloride was possible with the lowest lactose loss for the permeate. Although these authors claimed that no fouling problems were detected during NF tests, experiments at a larger scale should be performed to evaluate the economic feasibility of the process.

Ferg and co-workers [24] also investigated the recovery of lactose through a combination of membrane processes, namely MF (nominal pore size 0.2 μm), UF (5 kDa MWCO), ion exchange, and RO, and obtained an overall lactose recovery of 74%, with a purity of 99.8%.

Bertoluzzi and co-workers [25] compared the performance of two double-stage membrane processes for treatment of dairy wastewaters: (1) microfiltration (MF) plus NF and (2) MF plus OI. For MF, a hollow fibber module was used, being membranes made of poly(ether sulfonate)/poly(vinyl pyrrolidone) (PES/PVP) mixture with a pore size of 0.20 μm. In the NF and RO experiments, polymeric flat-type membranes were used, being these membranes made of polyamide composites. For the NF experiments, they used two different membranes (NF90 and NF), which are made of the same material but have different rejection properties, since NF90 is a tighter membrane, while the other one is a looser membrane, as can also be confirmed by their hydraulic permeabilities to pure water. Before the experiments, the dairy wastewater was prefiltrated across a filter of 0.25 μm to remove solids and to avoid a quick fouling of membranes. After that, microfiltration was also used as a pretreatment for the next operation (NF or OI) with the objective of improving their performance. The authors found that the sequence of MF followed by RO allowed a better removal of total solids and organic matter. Besides, the composition of the final permeate was compatible with the discharge on receiving waters according to the Brazilian environmental regulations or could be used in cleaning-in-place processes in the dairy factory. Although the results of this study are a good basis for other similar dairy wastewaters, since the variety of manufacturing processes involved in dairy products used is too large, for each type of sample/desired goal, a previous study is always necessary.

Macedo and co-workers [20] used a combination of UF/NF and UF/DF followed by NF/DF of the previous permeates, to recover lactose from the permeates both of sheep cheese whey (PUF-S) and of goat cheese whey (PUF-G) (**Figure 3**).

Both samples were subjected to the following pretreatment: filtration (using traditional cotton cloths), skimming for fat removal, and low pasteurization. NF of both permeates was carried out with NFT50 (NF) membranes until a volume concentration factor (VCF) of about 2.5. It was observed a sharp decrease (around 60%) in the permeate flux in the case of PUF-S and a smaller reduction (about 20%) in PUF-G (**Figure 4**). The authors attributed this different behavior to the following factors: the higher concentration of lactose and applied pressure used in the case of PUF-S (higher permeate fluxes) led to a greater and faster accumulation

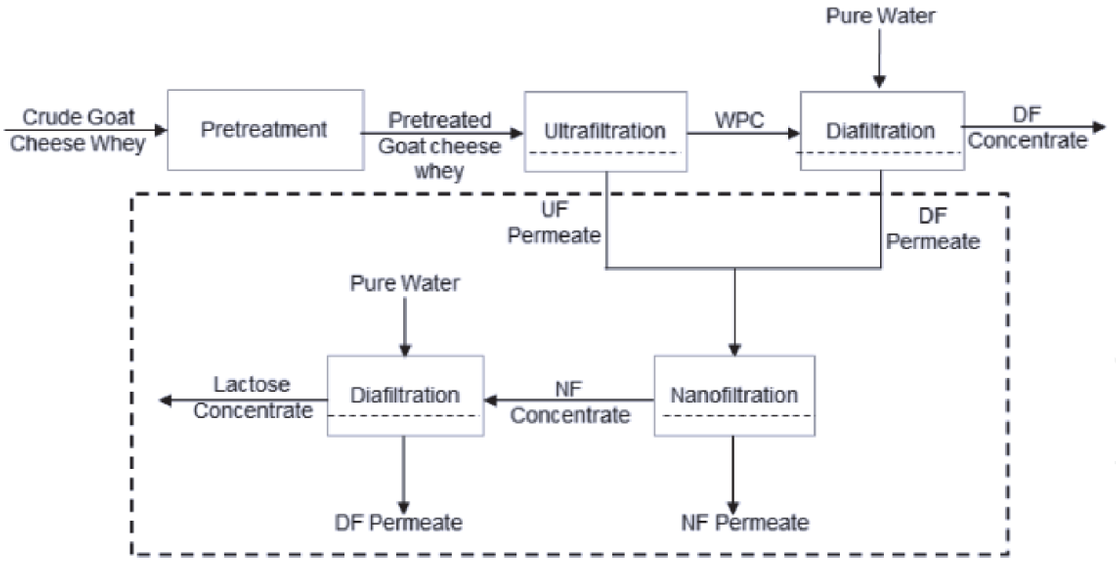

Figure 3.
Recover of lactose (lactose concentrate) and whey proteins from cheese whey: WPC = whey protein concentrate; DF concentrate = whey protein concentrate of UF/DF; lactose concentrate (obtained after NF/DF) [20].

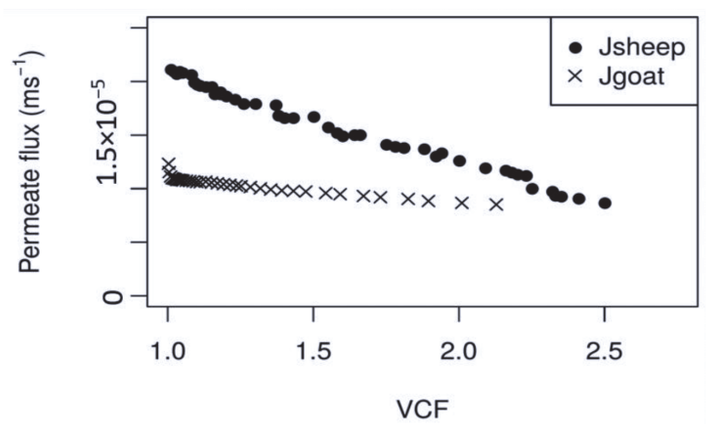

Figure 4.
Variation of average (three replicates) permeate fluxes with the volume concentration factor (VCF) for the concentration by nanofiltration of PUF-S ($\Delta P = 3.0 \times 10^6$ Pa; $\langle v \rangle = 1.42$ m s^{-1}) and PUF-G ($\Delta P = 2.0 \times 10^6$ Pa; $\langle v \rangle = 0.94$ m s^{-1}), at T = 25°C [20].

of lactose near the membrane surface, causing a higher increase in the osmotic pressure and concentration polarization phenomena. On the other hand, since the pH was 6.06 and the initial concentrations of calcium and phosphate were also higher than those of PUF-G, most probably, mineral fouling occurred due to the formation of insoluble calcium phosphates. In the case of PUF-G, the lower pH (5.43) and calcium and phosphate concentrations, due to the effect of dilution by diafiltration, were less prone to mineral fouling, leading to a more stable permeate flux. In spite of that, the permeate fluxes were lower during all the run, likely because of the highest concentration of chloride ions in goat cheese whey, which caused a greater initial osmotic pressure and therefore a lower effective transmembrane pressure. Beyond this, it is likely that also protein fouling contributed to this behavior since the pH of PUF-G was closest to the isoelectric point of β-lactoglobulin, the most abundant whey protein.

These results suggest that, in order to reach a better NF performance for recovering lactose, the following procedures should be applied: (1) precipitate calcium or use ionic exchange resins with the objective to reduce calcium concentration in the

permeates, avoiding the decline of permeate fluxes during NF, due to the formation of calcium phosphates and (2) optimize NF/DF process to improve the performance of recovery process of lactose.

Membrane processes, for example, nanofiltration, also play a role in the recovery of mother liquor (or delactosed permeate) resulting from the crystallization process. This co-product was investigated after fractionation by membrane processes (NF and reverse osmosis) for salt substitute in soup formulations [26, 27]. By NF, the residual lactose was recovered and recycled to the crystallization tank, enhancing the yield of this process. On the other hand, the permeate was subjected to reverse osmosis producing a retentate enriched in salt, which will be used in the food industry. A detailed review about the possible valorization of the mother liquor is described by Oliveira and co-workers [28].

Several processes for lactose production involving advanced technologies are commercially available. Most of them include membrane processes, namely nanofiltration and reverse osmosis for the production of edible lactose, crystalline lactose, and lactose syrup, which can be used for the production of galactooligosaccharides.

The integration of membrane processes for recovering bioactive compounds from cheese whey, in small and medium dairies, in spite of the initial cost of the equipment, must be investigated in each case. The economic viability of these plants will depend on the valuation to be given to the different separated fractions. Cheese producer's associations may play a decisive role in the concentration of all the cheese whey released in a given region, in a single plant for processing/recovery of value-added compounds.

4. Conclusions

The recovery of lactose from cheese whey allows not only the valorization of this co-product in the cheese industry, but also to mitigate the environmental damage caused by it. This work is focused on the use of membrane processes for lactose recovery. The selection of the most suitable process depends on several factors such as composition of the initial cheese whey (quite varied, especially in the case of those resulting from artisanal cheese production), volume produced, and final intended application for lactose. Progress in these processes will lead to an overall improvement in the process of recovering lactose from cheese whey. In the case of membrane separation, its implementation at the industrial level is increasing. Hitherto, its use in small and medium scales is conditioned by the initial economic investment, depending rather on the synergy of the various producers, which in turn should be driven by their associations.

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Conflict of interest

The authors declare no conflict of interest.

Author details

Antónia Teresa Zorro Nobre Macedo[1,2*], Joana Filipa Oliveira Monteiro[3], David José Chaveiro da Silva Azedo[1], Elizabeth da Costa Neves Fernandes de Almeida Duarte[2,3] and Carlos Dias Pereira[4]

1 Higher Agriculture School, Polytechnic Institute of Beja, Beja, Portugal

2 Linking Landscape, Environment, Agriculture and Food (LEAF), Higher Institute of Agronomy, University of Lisbon, Lisbon, Portugal

3 Higher Institute of Agronomy, University of Lisbon, Lisbon, Portugal

4 Higher Agriculture School, Polytechnic Institute of Coimbra, Coimbra, Portugal

*Address all correspondence to: atmacedo@ipbeja.pt

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