Review Article
Food Science and Technology
ISSN 0101-2061 (Print)
ISSN 1678-457X (Online)
DOI: https://doi.org/10.1590/fst.12522

A theoretical approach to dairy products from membrane processes

Tamires Marques Paes da CUNHA[1], Maria Helena Machado CANELLA[1], Isabel Cristina da Silva HAAS[2], Renata Dias de Mello Castanho AMBONI[2], Elane Schwinden PRUDENCIO[1,2]*

Abstract

Milk is a food rich in nutrients and requires processing that promotes its conservation, such as concentration. The membrane process, as an emerging technology and lower energy consumption, has been applied to concentrate, purify, and adapt milk for fluid consumption or derivatives. The Microfiltration, Ultrafiltration, Nanofiltration, and Reverse Osmosis processes have already been applied to milk and the biggest challenge is in controlling phenomena such as fouling, and polarization influenced by the system's operating conditions. The membrane process has already been used in the production of cheese, ricotta, protein concentrates, powdered milk, and dairy beverages.

Keywords: milk; concentration; operation conditions; fouling; proteins.

Practical Application: The use of membrane process to produce dairy products.

1 Introduction

Milk is composed of the main macronutrients important in human nutrition, carbohydrates, proteins, and fats, in addition to the presence of vitamins and minerals (Carter et al., 2021). It can be consumed in its fluid form or transformed into different products through concentration, coagulation, and fermentation process. As it is a nutritionally rich product with high water activity, it allows the action of microorganisms, its processing being essential to ensure longer shelf life and safety in consumption.

In addition to sterilization of milk, which allows for conservation at room temperature, concentration, that is, removal of water is another industrial process capable of improving the shelf life of milk. The most common technology used by the dairy industry is concentration by evaporators, which uses high temperatures and has high energy consumption (Blais et al., 2021). As an alternative to emerging concentration technologies, there are freeze concentration and membrane filtration.

Synthetic membranes for industrial application originate from observation and intent to reproduce biological membrane functions (Habert et al., 2006; Ulbricht, 2006). Membranes are selective barriers that separate fluids into two phases. Selection may be due to particle size or dissolution/diffusion across the membrane. Membranes can be made of different materials, having a wide variety of their characteristics according to the desired application. Membrane processes have already been studied and used in several areas such as health, water treatment, and the food industry.

Membrane processes allow the concentration and separation of components of interest. In the case of selecting particles according to their size, there are the processes of microfiltration, ultrafiltration, and nanofiltration, whereas the membranes used in the reverse osmosis process are not considered porous (Habert et al., 2006). These processes generate retentate and permeate, and both can be used depending on the purpose.

These processes are the most used for dairy products (Carter et al., 2021), which presents benefits such as lower operating temperatures, promoting conservation of thermosensitive components. This work sought to present a theoretical approach to processes with membranes, concepts, challenges, and advances in dairy processing.

2 Membranes process for milk filtration

The use of membranes is possible due to their capabilities. The membrane can separate two phases of a solution, can be permeated, and can restrict passage, making it an excellent separation, concentration, and purification tool. Regarding the morphology of the membranes, they can be separated according to their porosity and density, symmetries, and asymmetries.

A driving force is necessary to occur the phase separation by the membrane. Membrane processes are associated with a driving force of pressure or concentration. The mass transfer mechanism across the membrane can be convection and/or diffusion (Habert et al., 2006).

The whole milk presents a composition of 3.6% fat, 3.6% protein, 0.7% minerals, 4.6% carbohydrates, and 87.5% of water. Skimmed milk differs from whole milk in that it contains 2.9 to 0.5% fat. Each component of milk has a characteristic size and molecular weight, making membrane processes a good way of separating and fractionating compounds (Figure 1). Milk components have approximately the following sizes or molecular

Received 15 Apr., 2022
Accepted 28 June, 2022
[1] *Programa de Pós-Graduação em Engenharia de Alimentos, Centro Tecnológico, Universidade Federal de Santa Catarina – UFSC, Trindade, Florianópolis, SC, Brasil*
[2] *Departamento de Ciência e Tecnologia de Alimentos, Centro de Ciências Agrárias, Universidade Federal de Santa Catarina – UFSC, Florianópolis, SC, Brasil*
*Corresponding author: elane.prudencio@ufsc.br

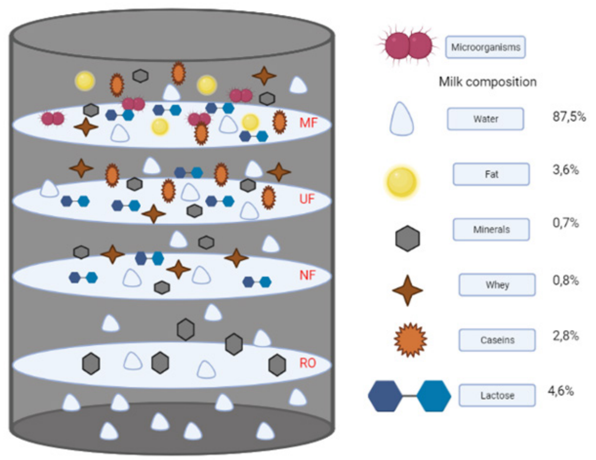

Figure 1. Membrane milk filtration - separation by component.

weights: fat globules 3.4 µm, caseins 110 nm, whey proteins 3-6 nm, and lactose 0.35 kDa (Brans et al., 2004).

2.1 Microfiltration (MF)

Microfiltration (MF) presents membranes with pores between 0.1 and 10 µm. Thus, particles larger than 0.1 µm are part of the retentate, and the pore size may vary according to the purpose of use (Carter et al., 2021). This process has as one of the applications for dairy the retention of bacteria and spores, thus it is necessary to control the size of the membrane pores, which should be small enough to retain microorganisms, but in a way, that does not compromise the composition of the permeated milk (Debon et al., 2012; Griep et al., 2018). Kara & Sert (2022) verified that MF treatment of milk improved the microbiological quality without any significant change in the chemical composition of skim milk powder. On the other hand, Saha et al. (2022) evaluated the effect of di-sodium phosphate and calcium chloride salt concentrations (10 to 90 mM) on the isolation of casein micelles from buffalo milk by MF process (0.20 µm). Based on the results found by these authors, no aggregation was recorded for buffalo micellar casein and size varied from 30 to 80 nm. In the study realized by Hooda et al. (2020) was the physicochemical properties of micellar casein concentrate prepared from skim milk (buffalo and cow), using microfiltration (MF), were assessed. In this study was not observed difference ($P > 0.05$) between the average particle size of skim milk casein micelles (cow and buffalo) and their respective micellar casein concentrate. The casein-to-whey protein ratio increased from 4.91 and 4.75 in skim milk to 135.16 and 159.22 in micellar casein concentrate of buffalo and cow, respectively (Hooda et al., 2020). Vieira et al. (2020a) confirmed advantages of microfiltration processing of goat whey orange juice beverage using mild temperatures (between 30° and 40 °C) to preserve consistency and also obtain a desirable microbial quality, beyond the preservation of many functional properties and volatile compounds. Vieira et al. (2020b) also investigated the sensory profiling using free listing task and consumer acceptance of the goat whey orange juice beverage processed by microfiltration (20, 30, 40, and 50 °C) and by conventional pasteurization. Free listing task allowed the discrimination of the beverages based on the type of processing (pasteurization or microfiltration) and microfiltration temperature. Overall, free listing task was a suitable methodology for discriminating beverages submitted to different processing, and microfiltration (20 or 30 °C) is an interesting option for goat whey orange juice beverages, resulting in products with adequate sensory properties (Vieira et al., 2020b).

The MF process has also been applied as a pre-treatment to increase the stability of UHT milk, which is increasing during long-term storage (D'Incecco et al., 2018). MF can generate dairy raw material, permeate, with adequate quality and characteristics for processing derivatives (Debon et al., 2012).

2.2 Ultrafiltration (UF)

Ultrafiltration (UF) can prevent the passage of molecules larger than 0.001 µm, as they have membranes with pores between 0.01 – 0.001 µm (Carter et al., 2021). UF can retain proteins (Oliveira et al., 2021) and fat, and allows the passage of vitamins, minerals, and lactose. The use of UF to develop dairy products benefits yield, nutritional functionality, and sensory characteristics (Faion et al., 2019). This process can be applied for protein concentration and purification (Ng et al., 2018) and stands out in cheese production, providing higher protein concentration and better nutritional characteristics to this product (Faion et al., 2019; Gavazzi-April et al., 2018). Another common application for UF is the production of milk protein concentrate (MPC) (Gavazzi-April et al., 2018).

Based on experimental data obtained by whey protein hydrolysate from UF process can be used in the technology of various Melnikova et al. (2022) dairy products to replace skimmed milk when making a normalized mixture and also as a main ingredient of beverages for sports nutrition taking into account sensory profiling of the products. This allows expanding the assortment line of domestic products for preventive nutrition of people suffering from allergies to cow's milk proteins, as well as ensuring import substitution in the segment of functional food products (Melnikova et al., 2022).

2.3 Nanofiltration (NF)

For Nanofiltration (NF) processes, the membranes used have pores between 0.001-0.0001 µm (Carter et al., 2021). NF is capable to concentrate small components of molecular weight equal to or greater than 100 Da. NF membranes can retain sugars, amino acids, dyes, and salts (Chen et al., 2018). It can also be used in the concentration of whey proteins in milk to produce derivatives. The NF process is capable of high retention of organic compounds resulting from the interaction between the membrane, the solution to be filtered, and electrostatic repulsion (Prudêncio et al., 2014).

2.4 Reverse osmosis

Reverse osmosis (RO) began in the 1960s with the creation of the first membrane for this process. Osmosis is the process that uses membranes with pores smaller than 0.0001 µm (Carter et al., 2021). These membranes can retain larger ions and compounds releasing water into the permeate and can be applied for pre-concentration of milk (Blais et al., 2021). Syrios et al. (2011) compare this process to evaporation, both of which promote water removal and osmosis may contain traces of other low molecular weight components. This process presents an increased osmotic pressure and feed stream viscosity (Blais et al., 2021) facing severe problems with fouling and permeate flow reduction. The disadvantage of this process has become the focus of several studies to promote optimization.

3 Cascade process

These processes can be joined together to form cascades to increase the efficiency of each process. Diafiltration (DF) is always used in association with one of the processes mentioned above, where water is added to the feed to overcome viscosity and allow the system to continue filtering to increase efficiency (Gavazzi-April et al., 2018), where DF also allows obtaining a purer retentate (Prudêncio et al., 2014). The union of several processes with membrane can promote different flows with different compositions that can be applied for different purposes, such as the standardization of milk to produce derivatives (Xia et al., 2020).

For the RO process, being inserted in a cascade is interesting due to its difficulties with encrustations. The addition of processes such as UF and MF before RO in milk filtration can improve its efficiency to retain somatic cells, fat globules, and protein aggregates. Due to the retention of microorganisms during the MF step, it allows RO to be performed at higher temperatures without microbiological compromise due to the multiplication temperature of microorganisms (Blais et al., 2021).

4 Membrane materials and operations conditions for milk processing

Knowledge of physical and chemical data about the solution to be filtered is essential for proper and efficient choices regarding membrane material and process operating conditions to try to predict the behavior of the solution, applied conditions, and interaction with the membrane. In the case of milk, there are proteins, fats, lactose, minerals, and a small number of vitamins. Verruck et al. (2019) related that dairy products are considered as products with substantial health benefits, such as cheese, cheese whey, dairy beverage, and powder milk. These products could be consideredand essential for the equilibrated diet from a nutritional and functional point of view. It is noteworthy that they are also excellent sources of proteins and minerals, especially calcium in a highly bioavailable form. Therefore, dairy products can improve health or well-being and, when consumed at recommended levels, their benefits include improved immune system function, reduced risk of cardiovascular, reduced risk of bone mass loss, and protection against free radical damage (Verruck et al., 2019). For this reason the use of milk to generate dairy products with new nutritional values justified its use in the membrane process.

Membranes can be separated into organic (polymers) and inorganic (metals and ceramics) (Habert et al., 2006). In dairy processing, membranes are used made of polymers or ceramics. The most used polymers for filtration in dairy products are polyvinylidene fluoride and polypropylene for UF and MF and polyamide for RO, while ceramic membranes are commonly used for MF (Carter et al., 2021). Ceramic membranes have a longer service life, but polymeric membranes are cheaper (Habert et al., 2006).

Ceramic membranes are more expensive due to the higher raw material value for fabrication and more complex production processes, but this balances out when presented with its advantages. This material allows a membrane with greater durability, high mechanical strength, resistance to chemicals and solvents, and thermal stability (Mestre et al., 2019). The difficulty in using a ceramic membrane for milk filtration is because whey proteins tend to adsorb on the surface, while caseins contribute to the encrustation layer proportionally to the applied pressure (Blais et al., 2021). Ceramic membranes are applied in the microfiltration process as an alternative for pasteurizing milk and for valuing whey (Mestre et al., 2019).

Polymer membranes are cheaper, considering that membranes represent 40% of the cost of systems is an important consideration. Although other materials are also used, polymeric membranes are the majority. Polymers provide the possibility of abundant variations in structure and barrier properties (Ulbricht, 2006).

As for the operating conditions, it is necessary to control the temperature, pressure, the feed direction being frontal or tangential, and the feed speed. Influences of these parameters on membrane filtration efficiency have already been reported. Feed speed can be important for running the process, especially when have a tangential feed. In this type of feeding, solids deposited on the membrane are dragged, making the polarization and incrustation phenomena more controlled. When using higher speeds, this drag is continuous, and the filtration resistance is lower (Atra et al., 2005).

Within the data collected in Table 1, it is observed that the temperatures for processing milk and whey, with membranes, occur between 10 ºC and 55 ºC. In general, higher temperatures generate lower viscosity, which benefits the permeation flow. Ng et al. (2018) applied UF in skimmed milk found flows with an increase of 250% when the temperature was increased from 10 ºC to 50 ºC, but this variation increased the filtration resistance. In the case of RO, Blais et al. (2021) achieved an 89% increase in filtration flow when going from 15 °C to 50 °C.

Pressure acts as a driving force in the MF, UF, NF, and RO processes. It can be seen from Table 1 that the operating values are up to 3 bar for MF, between 2 and 8 bar for UF, 2 up to 20 bar for NC, and RO is operated at much higher values and may reach 40 bar. The flow is related to the applied driving force, requiring higher pressures in tighter membranes. RO membranes are not considered porous, so they are operated at high-pressure values to have a satisfactory flow (Habert et al., 2006).

5 Development of dairy products using membranes

5.1 Cheese

Cheese is an important product of the dairy industry and is part of many traditional food preparations. Membrane filtration of milk for cheese preparation brings benefits such

Table 1. Membrane materials, geometries, objectives, and operating conditions for milk filtration.

Process	Materials	Pores/ Molecular weight	Geometry/ Modules	Objective	Operating temperature	Operating pressure	Source
MF	Ceramics	0.1 μm		Protein fractionation	10 °C		Schiffer et al. (2020)
MF	Ceramics	1.4/1.2 μm	Tubular	Removal of microorganisms and spores	6 °C		Griep et al. (2018)
MF	Organic poly(imide)	1.4 μm	Hollow fiber	Fermented Milk Production	45 °C	1-3 bar	Debon et al. (2012)
MF	Ceramics	1.4 μm	Tubular	Concentrated skimmed milk (milk powder)			Blais et al. (2021)
MF	Ceramics	1.4 μm		Increases UHT stability			D'Incecco et al. (2018)
UF	Zirconium oxide	50 kDa	Tubular	Concentration (cheese production)	50-55 °C		Deshwal et al. (2020)
UF	Polyethersulfone (PES)	10 kDa	Spiral	Temperature X Flow (skimmed milk)	10/30/50 °C		Ng et al. (2018)
UF	Polysulfone amide	10 kDa	Hollow fiber	Sheep cheese production	22 °C	1-2 bar	Faion et al. (2019)
UF+DF	Polyethersulfone (PES)	10 kDa/50kDA	Spiral	Protein concentrate (skimmed milk)	50 °C	4.65 bar	Gavazzi-April et al. (2018)
UF	Polyethersulfone (PES)	10 kDa		Biofilm investigation	15/50 °C		Chamberland et al. (2019)
UF	Polyvinyl difluoride	6-8 kDa	Flat	Milk and whey concentration	30-50 °C	1-5 bar	Chen et al. (2018)
UF	Polyethersulfone (PES)	15-20 kDa	Flat	Milk and whey concentration	30-50 °C	1-5 bar	Chen et al. (2018)
UF	Polyethersulfone (PES)	10 kDa	Spiral	Skimmed milk concentration	10-50 °C	2.76-7.58 bar	Méthot-Hains et al. (2016)
UF	Polyethersulfone (PES)	5/30 kDa	Flat	Fouling analysis in whey processing	25 °C	1-3 bar	Luján-Facundo et al. (2017)
UF+DF	Polyethersulfone (PES)	10 kDa	Spiral	Production of milk protein concentrate			Eshpari et al. (2017)
UF	Polysulfone	25 kDa		Koummis production	52 °C	2.6-3.6 bar	Kücükcetin et al. (2003)
NF	Polyamide			Milk and whey concentration			Atra et al. (2005)
NF	Polyamide		Spiral	Treatment of dairy waste	30-50 °C	10-20 bar	Chen et al. (2018)
NF+DF		150/300 Da	Spiral	Whey filtration (ricotta production)	24 °C	6.9 bar	Prudêncio et al. (2014)
RO	Polyamide		Spiral	Pre-concentration of milk	15/50°C	30.05 bar	Blais et al. (2021)
RO	Polyamide		Spiral	Skimmed milk concentration (cheese production)	50 °C	26.6 bar	Dussault-Chouinard et al. (2019)
RO	Polyamide			Skimmed milk concentration	10 °C	40 bar	Artemi et al. (2020)

Microfiltration (MF), ultrafiltration (UF), nanofiltration (NF), reverse osmosis (RO), diafiltration (DF).

as increased casein and total solids in milk, raw material, and product with greater standardization and increased yield (Carter et al., 2021), however, it can influence texture parameters in cheese (Deshwal et al., 2020). The microstructure of cheeses is also affected when the raw material is the result of filtration, being more compact, influencing the behavior of caseins, and interference from fats (Deshwal et al., 2020).

Ultrafiltration was used in the process of making pecorino cheese from sheep's milk. Faion et al. (2019) compared two formulations, one with low-fat sheep's milk and the second formulation with concentrated non-fat sheep's milk per UF. The performance of the UF process and the physicochemical characteristics of the raw material and the cheeses were evaluated. The UF favored the presence of proteins and fat in the raw material and consequently presented cheeses with higher yields.

The UF was also present in the manufacture of Halloumi cheeses made with goat's milk. From the retentate with different percentages of fat, the cheeses were produced and analyzed for physical-chemical, rheological, and sensory aspects. The researchers observed an interesting relation regarding the amount of protein, fat, and minerals in cheese, that is, cheeses with lower fat and higher protein content also have higher amounts of minerals. With an increase in milk fat, the retention of proteins by the membrane consequently also decreases the levels of minerals that bind to them and can pass to permeate during the UF process (Deshwal et al., 2020).

5.2 Cheese whey

Whey is a by-product of the cheese industry. To produce 1 kg of cheese, 9 kg of whey are generated (Luján-Facundo et al., 2017), with a large amount of waste to be treated, obtaining essential processing and utilization of it. Whey is liquid rich in proteins and lactose and can be used in processes with membranes for the concentration and purification of these components and application in dairy derivatives. However, there is a great difficulty because whey proteins influence the incrustation, reducing the efficiency of the process. Luján-Facundo et al., (2017) carried out a study focused on the analysis of 5.000-30.000 Da cutting membrane fouling in the filtration of whey. They related the percentage of calcium in the filtration feed solution with the increase in scale.

The use of NF associated with diafiltration was analyzed for ricotta production as an alternative to traditional production. Prudêncio et al. (2014) collected the whey from the result of the production of Minas fresh cheese, applied the membrane process to the collected by-product and the retentate was applied in the manufacture of ricotta cheese. Physical, chemical, rheological characteristics and images were evaluated to verify ricotta quality. Membranes, for 150-300 Da, were used for NF in spiral modules with tangential feed. The study shows that NF from whey had a decrease in permeate flux over time, which may be justified by polarization concentration, encrustation, and adsorption, effects already expected in processes with membranes. The serum underwent two different treatments, only NF and NF plus diafiltration, which were compared to the control serum. The ricotta cheeses produced with retentate from the processes with membranes had a lower quantity of total solids and proteins, which may be associated with the operating conditions and sensitivity to undergo protein alterations, and more affected coloration when compared to the control.

5.3 Dairy beverages

A fermented milk drink has already been developed from MF permeate. Using hollow fiber membranes with an average pore size of 1.4 μm, Debon et al. (2012) aimed to generate an adequate raw material that would allow the stability of a prebiotic drink. MF allowed acceptable microbiological quality and few nutritional losses for the raw material, generating a dairy beverage without the need for heat treatment.

Koumiss is an alcoholic drink traditionally made with mare's milk widely consumed in central Asia. Kücükcetin et al. (2003) applied membrane technology when adapting the production of the Koumiss drink with cow's milk. Due to the differences between the milk of the species, MF, UF, and NF were used to make the cow's milk suitable to produce the drink and, in this way, compare the characteristics with the traditional drink. Performing sensory analysis with mare's milk and modified cow's milk Koumiss showed no significant differences between samples. The use of processes with membranes proves to be effective for the compositional manipulation of milk to produce derivatives.

5.4 Powder milk

The production of powdered milk requires a great deal of water withdrawal, and approximately 87% of the milk is composed of water, making the membrane process unfeasible as a single manufacturing step. However, this does not prevent membrane technology from being applied as a form of pre-concentration, exposing the milk to heat treatments for shorter periods. Blais et al. (2021) analyzed the use of RO and MF in the concentration of skimmed milk for powdered milk production. The main objective of the research was to analyze the performance of the individual use of OR and together with MF. Another factor analyzed was the temperature (15 ºC and 50 °C). MF was used to remove microorganisms and other scales and RO to remove water. As for the permeate flux, there was no great difference between the use of RO and MF combined with RO at 15 ºC. However, when the process took place at 50 °C the flux values were a little higher, that is, the higher temperature delayed the inlay process. This improvement could be due to the lower viscosity of the milk at a higher temperature. The authors also studied the costs of the process and reported that RO can generate savings in the milk concentration process when compared to the traditional methodology.

Syrios et al. (2011) compared the UF, NF, and RO retentates for spray drying in the production of skimmed milk powder. The authors related the membrane process, as a pre-treatment, with ionic calcium retained and heat stability when powdered milk was reconstituted at 25%. The three processes showed a percentage increase of solids in the retentate, including total and ionic calcium, but it was only possible to reach the reconstituted milk powder with satisfactory stability to heat with NF treatment, at the sterilization temperature. The authors noted the difficulty of generating reconstituted powdered milk without prior heat application.

Prestes et al. (2022) reported that emerging non-thermal Technologies, such as membrane process, are promising alternatives that have been developed and explored in powder milk producing. With a purpose to decrease the negative effects of the conventional concentration processes and contribute with powder milk with high quality. These alternative procedures preserve sensory and flavor properties and maintain food pigments, original volatile compounds, vitamins, enzymes, and proteins (Prestes et al., 2022).

5.5 Protein concentrate

Protein concentrates are important products because they can be applied to different types of food, such as infant formula and bakery products (Carter et al., 2021). Milk has high levels of protein which makes it suitable raw material to produce concentrate and protein isolates. One of the difficulties in handling milk proteins is their thermal sensitivity, which can affect stability in the reconstitution of concentrates (Eshpari et al., 2017; Carter et al., 2021).

Milk protein concentrates are commonly produced using membrane processes such as UF and diafiltration followed by evaporation and a drying step, varying the percentage of protein concentration (Eshpari et al., 2017; Gavazzi-April et al., 2018). Gavazzi-April et al. (2018) evaluated the performance of UF and diafiltration in the production of milk protein concentrate. They reported favoring the flow of the system by diafiltration and considered the use of membranes with a 10 kDa cutoff as suitable to produce milk protein concentrate.

6 Performance of membrane processes in dairy processing

For the results of the membrane process to be viable, it is necessary to evaluate its performance. Good performance will depend on the combination of membrane characteristics and operating conditions that, consequently, will interfere with the characteristics and flows of currents (supply, retentate and permeate). To assess the performance of a membrane process, it is important to observe the parameters of permeate flux, hydraulic resistance (Gavazzi-April et al., 2018; Faion et al., 2019; Méthot-Hains et al., 2016), feed composition versus permeate and retentate (Blais et al., 2021; Gavazzi-April et al., 2018), retention index (Prudêncio et al., 2014; Atra et al., 2005), solute yield and concentration factor (Atra et al., 2005).

The permeate flux (J) is related to the permeate volume (V) during a given time (t) through an area of membrane (A) (Gavazzi-April et al., 2018) (Equation 1).

$$J = \frac{V}{t \cdot A} \text{ (L/h.m}^2\text{)} \quad (1)$$

For the UF + skim milk diafiltration process, Gavazzi-April et al. (2018) found values between 5.6 and 8.7 kg/hm^2 at a temperature of 50 °C and a pressure of 4.65 bar. Faion et al. (2019) had used the UF for the concentration of sheep milk, at pressures of 1 and 2 bar found initial values of 15.38 and 18.71 L/hm^2 respectively, so they opted for the process at 2 bar, but this flow was decreasing throughout the process and stabilized at 0.85 L/hm^2 after 6 min of filtration.

The total resistance (R_t) of the process can be obtained by summing the membrane resistance (R_m), the reversible resistance (R_R) and the irreversible resistance (R_i) (Méthot-Hains et al., 2016) (Equations 2, 3, 4, 5).

$$R_t = R_m + R_R + R_i \quad (2)$$

$$R_m = \frac{P}{\mu J_w} \quad (3)$$

$$R_R = \frac{P}{\mu J_E} - R_m - R_i \quad (4)$$

$$R_i = \frac{P}{\mu J_R} - R_m \quad (5)$$

where P is the transmembrane pressure (Pa), μ is the permeate viscosity (Pa.s), J_w is the water flux (m^3/m^2.s), J_R is the water flux (m^3/m2.s) after membrane rinsing, and J_E is the permeation flux (m^3/m^2.s) at the end of UF.

Faion et al. (2019) observed that when doubling the pressure during the UF process (1 bar and 2 bar), the hydraulic resistance to permeation dropped by half. Méthot-Hains et al. (2016) reported the influence of temperature on the UF resistances of skimmed milk, at a temperature of 50 °C compared to 10 °C obtained higher membrane resistance values, but lower irreversible resistance.

To evaluate the composition of the RO and MF/RO filtration process currents, Blais et al. (2021) quantified percentages of total solids, fat, proteins, casein, lactose, and somatic cells of skimmed milk, which was used as feed, during all studied processes Like previous authors, Gavazzi-April et al. (2018) evaluating the UF and UF/diafiltration currents in the production of protein concentrate (retentate), obtained a considerable loss of protein in the permeate. The compositional evaluation of currents is essential to verify the efficiency of the process, after all, it will determine the nutritional quality of the developed products and concentrate yields, in addition to indicating problematic points and the need for process optimization. Faion et al. (2019) reported differences in composition with a change in operating pressure, when a pressure of 2 bar was used, the retentate contained more values of protein, fat, and minerals than the retentate at 1 bar.

The retention index (R) is a parameter to evaluate the relation between the amounts of the compound of interest in the permeate solutions and the retentate solutions, thus determining the capacity of the process (membrane and operating conditions) to retain the component of interest (Prudêncio et al., 2014). Prudencio et al., 2014 found an R of 0.9 for protein when applying NF and diafiltration on whey from the production of Minas Frescal cheese. Atra et al. (2005) observed values between 0.92-0.98 of protein retention when they used UF to filter whey (Equation 6).

$$R = 1 - \frac{c_\rho}{c_R} \quad (6)$$

where C_P is the solute concentration in the permeate (g/L) and C_R is the solute concentration in the retentate (g/L).

The concentration factor (F) is related to the volume of solution in the feed (V_F) and the volume of retentate (V_R) (Equation 7). The solute yield (Y) is the concentration of the components in the feed by the components in the retentate (Atra et al., 2005). Atra et al. (2005) found that it is possible to achieve a Y greater than 90% in lactose recovery by applying NF (Equation 8).

$$F = \frac{V_F}{V_R} \quad (7)$$

$$Y = \frac{V_{RC_R}}{V_F c_F} \quad (8)$$

where C_R is the solute concentration in the retentate (g/L) and c_F solute concentration in the feed (g/L).

Membrane characteristics are important for the final efficiency of the process, as it is related to the selectivity and composition of permeate and retentate. Uniformity of both pore size and pore distribution across the membrane is important for the containment of the components in question (Brans et al., 2004; Gavazzi-April et al., 2018). The thinner the membrane, the easier it will be to permeate it. Very thick membranes are commercially unfeasible due to extremely low flows, but they have enough structure for adequate mechanical strength (Habert et al., 2006).

The permeate flux parameter is important for evaluating the efficiency of a membrane process. Several studies involving milk processing look at the flow behavior. The permeate flux in milk filtration tends to follow a pattern. At the beginning of filtration, the permeate flux is the highest in the process, after a while, membrane compaction and system adjustment may occur, leading to a decrease in this flux. After these steps, the flow tends to decrease over time due to incrustations, pore obstruction, adsorption of components by the membrane, increased feed viscosity, among other effects that lead to increased filtration resistance (Blais et al., 2021).

High permeate fluxes can be seen as successful for the membrane process, but caution is needed to manipulate operating conditions to increase the flux. An example of this is the increase in pressure, which is proportional to the increase in flow in the short term, as the common effect of high pressure is severe incrustations on the membrane (Artemi et al., 2020). Permeate flow is directly proportional to pressure, but above the critical transmembrane pressure, the flow becomes pressure-independent (Chen et al., 2018). Artemi et al. (2020) observed the onset of incrustation linked to linearity with the flow, when it no longer exists, they characterize it as a critical flow achieved, this was reported for the RO process.

Higher temperatures favor the flow performance, due to the lower viscosity and greater diffusivity that promotes an improvement in polarization (Atra et al., 2005). However, higher temperatures may facilitate the growth of thermophilic microorganisms and the decomposition of sensitive components (Ng et al., 2018). The flux has a common behavior of linear rise combined with temperature. Atra *et al.,* 2005 observed an increase of 0.51 m^2h at each degree °C for UF of skimmed milk. Blais et al. (2021) found that by increasing the temperature by 1°C there is a 3% improvement in the permeate flow in the RO process in the concentration of skimmed milk.

7 Challenges in milk filtration: fouling and polarization

Fouling can be subdivided into organic and inorganic. Organic fouling is caused by proteins, lactose, and organic acids, while inorganic fouling is mainly caused by calcium phosphate precipitation and biofouling (Blais et al., 2021). This phenomenon can be reversible when there is the possibility of recovering the membrane through cleaning, and irreversible when the incrustation is permanent. This phenomenon can make the process unfeasible depending on the severity (Habert et al., 2006).

Fouling can be generated due to adsorption of components across the membrane, clogging of pores, and deposition of components on the membrane surface, including concentration polarization (Habert et al., 2006). The responsibility for membrane encrustations during milk filtration has been largely associated with protein adsorption and calcium precipitation. Calcium phosphate behaves differently from other components, and its solubility decreases when the temperature of the process increases (Ng et al., 2018).

Concentration polarization will occur in all membrane filtration processes and this is due to the concentration of components close to the membrane surface. This phenomenon can be influenced by the concentration of components in the feed, flow conditions, pressures above the limiting flow, and selective capacity of the membrane (Habert et al., 2006). Protein-rich liquids, such as milk, can be polarized by high concentration, as the proteins are deposited on the surface of the membrane, favoring this phenomenon (Faion et al., 2019).

Both incrustation and polarization give resistance to the filtration process and are the focus of several studies on manipulations in membrane operations to reduce these problems. Blais et al. (2021) observed that operations using higher temperatures can reduce this phenomenon due to the association with viscosity. Using a tangential feed with higher speeds is one more way to improve the effects of polarization (Habert et al., 2006; Brans et al., 2004). Brans et al. (2004) reviewed features to reduce fouling phenomena using vibrating modules, cleaning particles, ultrasonic waves, pulsating, or high-speed crossover feed stream. Furthermore, the choice of membranes should be considered, as the more hydrophilic and negatively charged, the more they will contribute to the reduction of protein encrustation (Carter et al., 2021).

8 Costs of membrane process

The concentration of milk is commonly carried out by evaporation and drying, which are energy-demanding processes. In the quest for greater sustainability in industrial operations, membrane processes are known to save energy when compared to other concentration technologies. The energy-related cost comes mainly from the feed pumps and depends on the pressures used in the process (Blais et al., 2021).

A multi-stage evaporator has an average consumption of 83.3 Wh L^{-1} of water removed (Blais et al., 2021), depending on the combination of processes and mode of operation, membranes can be an option for reducing energy consumption in dairy products. To evaluate energy consumption in the process of concentrating milk proteins by UF and diafiltration Gavazzi-April et al. (2018) used a voltmeter connected to the system power motor, calculated Wh/kg of retentate, and reached values between 36×10^3 kWh and 114×10^3 kWh depending on diafiltration variations. Blais et al. (2021) achieved a value of 110 Wh L^{-1} of water removed from energy expenditure for the RO process in the concentration of skimmed milk, and when associating the MF before RO and raising the temperature from 15 °C to 50 °C, the spent started to be 49.4 Wh L^{-1}, generating energy savings.

Membrane costs are calculated per m^2. The values of ceramic membranes based on alumina and zirconia can vary between 500 and 3000 $/$m^2$ and polymeric membranes vary between 20 and 200 $/$m^2$ (Mestre et al., 2019). In addition to the value of membranes, Gavazzi-April et al. (2018) point out costs with cleaning and replacement of membranes, which have a useful life limit.

9 Conclusion

The membranes are a technology appropriate to milk processing and dairy milk product. The MF, UF, NF, and RO are the process used in the dairy industry. For MF, ceramic membranes are the most used for dairy products, according to the pore size range they can be used for microbiological standardization of milk, whey processing, and increased stability of fluid milk. UF has great application in cheese production, with the advantage of increased yield due to greater protein retention, it can also be applied in the filtration of whey. NF can concentrate whey proteins and recover lactose. In RO, polyamide membranes stand out, whose function is to remove water from either milk or its serum, forming a retentate rich in total solids.

To evaluate the performance of the milk filtration process with membranes, the permeate flow is an important parameter. Due to the composition of milk, a source of protein, the biggest difficulties in its filtration through membranes are related to scale and concentration polarization. These problems are the focus of several studies to evaluate manipulations in processes to reduce their effects that affect performance. The main membrane filtration costs to be analyzed are the energy costs of pumping, the membrane itself, cleaning, and maintenance.

Acknowledgements

The authors are grateful to the Coordination of Improvement of Higher Education Personnel by scholarship (CAPES, Finance code-001) and the National Council of Technological and Scientific Development by the financial support (CNPq, 405965/2016-8).

References

Artemi, A., Chen, G. Q., Kentish, S. E., & Lee, J. (2020). The relevance of critical flux concept in the concentration of skim milk using forward osmosis and reverse osmosis. *Journal of Membrane Science*, 611, 118357. http://dx.doi.org/10.1016/j.memsci.2020.118357.

Atra, R., Vatai, G., Bekassy-Molnar, E., & Balint, A. (2005). Investigation of ultra- and nanofiltration for utilization of whey protein and lactose. *Journal of Food Engineering*, 67(3), 325-332. http://dx.doi.org/10.1016/j.jfoodeng.2004.04.035.

Blais, H., Ho, Q. T., Murphy, E. G., Schroën, K., & Tobin, J. T. (2021). A cascade microfiltration and reverse osmosis approach for energy efficient concentration of skim milk. *Journal of Food Engineering*, 300, 110511. http://dx.doi.org/10.1016/j.jfoodeng.2021.110511.

Brans, G., Schroën, C. G. P. H., van der Sman, R. G. M., & Boom, R. M. (2004). Membrane fractionation of milk: state of the art and challenges. *Journal of Membrane Science*, 243(1-2), 263-272. http://dx.doi.org/10.1016/j.memsci.2004.06.029.

Carter, B. G., Cheng, N., Kapoor, R., Meletharayil, G. H., & Drake, M. A. (2021). Microfiltration-derived casein and whey proteins from milk. *Journal of Dairy Science*, 104(3), 2465-2479. http://dx.doi.org/10.3168/jds.2020-18811. PMid:33455742.

Chamberland, J., Messier, T., Dugat-Bony, E., Lessard, M. H., Labrie, S., Doyen, A., & Pouliot, Y. (2019). Influence of feed temperature to biofouling of ultrafiltration membrane during skim milk processing. *International Dairy Journal*, 93, 99-105. http://dx.doi.org/10.1016/j.idairyj.2019.02.005.

Chen, Z., Luo, J., Hang, X., & Wan, Y. (2018). Physicochemical characterization of tight nanofiltration membranes for dairy wastewater treatment. *Journal of Membrane Science*, 547, 51-63. http://dx.doi.org/10.1016/j.memsci.2017.10.037.

D'Incecco, P., Rosi, V., Cabassi, G., Hogenboom, J. A., & Pellegrino, L. (2018). Microfiltration and ultra-high-pressure homogenization for extending the shelf-storage stability of UHT milk. *Food Research International*, 107, 477-485. http://dx.doi.org/10.1016/j.foodres.2018.02.068. PMid:29580509.

Debon, J., Prudêncio, E. S., Petrus, J. C. C., Fritzen-Freire, C. B., Müller, C. M. O., Amboni, R. D. M. C., & Vieira, C. R. W. (2012). Storage stability of prebiotic fermented milk obtained from permeate resulting of the microfiltration process. *LWT - Food Science and Technology*, 47(1), 96-102. http://dx.doi.org/10.1016/j.lwt.2011.12.029.

Deshwal, G. K. R., Ameta, R., Sharma, H., Singh, A. K., Panjagari, N. R., & Baria, B. (2020). Effect of ultrafiltration and fat content on chemical, functional, textural and sensory characteristics of goat milk-based Halloumi type cheese. *LWT - Food Science and Technology*, 126, 109341. http://dx.doi.org/10.1016/j.lwt.2020.109341.

Dussault-Chouinard, I., Britten, M., & Pouliot, Y. (2019). Improving rennet coagulation and cheesemaking properties of reverse osmosis skim milk concentrates by pH adjustment. *International Dairy Journal*, 95, 6-14. http://dx.doi.org/10.1016/j.idairyj.2019.03.008.

Eshpari, H., Jimenez-Flores, R., Tong, P. S., & Corredig, M. (2017). Thermal stability of reconstituted milk protein concentrates: effect of partial calcium depletion during membrane filtration. *Food Research International*, 102, 409-418. http://dx.doi.org/10.1016/j.foodres.2017.07.058. PMid:29195966.

Faion, A. M., Becker, J., Fernandes, I. A., Steffens, J., & Valduga, E. (2019). Sheep's milk concentration by ultrafiltration and cheese elaboration. *Journal of Food Process Engineering*, 42(4), 1-9. http://dx.doi.org/10.1111/jfpe.13058.

Gavazzi-April, C., Benoit, S., Doyen, A., Britten, M., & Pouliot, Y. (2018). Preparation of milk protein concentrates by ultrafiltration and continuous diafiltration: effect of process design on overall efficiency. *Journal of Dairy Science*, 101(11), 9670-9679. http://dx.doi.org/10.3168/jds.2018-14430. PMid:30172402.

Griep, E. R., Cheng, Y., & Moraru, C. I. (2018). Efficient removal of spores from skim milk using cold microfiltration: spore size and surface property considerations. *Journal of Dairy Science*, 101(11), 9703-9713. http://dx.doi.org/10.3168/jds.2018-14888. PMid:30146287.

Habert, A. C., Nóbrega, R., & Borges, C. P. (2006). *Processos de separação com membranas* (180 p.). Rio de Janeiro: E-Papers.

Hooda, A., Mann, B., Sharma, R., & Bajaj, R. (2020). Physicochemical characterisation of native micellar casein concentrates from buffalo and cow skim milk harvested using microfiltration. *International Journal of Dairy Technology*, 73(4), 781-789. http://dx.doi.org/10.1111/1471-0307.12711.

Kara, Ü., & Sert, D. (2022). The use of microfiltration technique in the production of skim milk powder: the effect of milk transport conditions on the microbiological and physicochemical properties of milk and milk powders. *International Journal of Dairy Technology*, 75(2), 438-447. http://dx.doi.org/10.1111/1471-0307.12855.

Kücükcetin, A., Yaygin, H., Hinrichs, J., & Kulozik, U. (2003). Adaptation of bovine milk towards mares' milk composition by means of membrane technology for koumiss manufacture. *International Dairy Journal*, 13(12), 945-951. http://dx.doi.org/10.1016/S0958-6946(03)00143-2.

Luján-Facundo, M. J., Mendoza-Roca, J. A., Cuartas-Uribe, B., & Álvarez-Blanco, S. (2017). Membrane fouling in whey processing and subsequent cleaning with ultrasounds for a more sustainable process. *Journal of Cleaner Production*, 143, 804-813. http://dx.doi.org/10.1016/j.jclepro.2016.12.043.

Melnikova, E. I., Bogdanova, E. V., & Koshevarova, I. (2022). Nutritional evaluation of whey protein hydrolysate: chemical composition, peptide profile, and osmolarity. *Food Science and Technology*, 42, e110721. http://dx.doi.org/10.1590/fst.110721.

Mestre, S., Gozalbo, A., Lorente-Ayza, M. M., & Sánchez, E. (2019). Low-cost ceramic membranes: a research opportunity for industrial application. *Journal of the European Ceramic Society*, 39(12), 3392-3407. http://dx.doi.org/10.1016/j.jeurceramsoc.2019.03.054.

Méthot-Hains, S., Benoit, S., Bouchard, C., Doyen, A., Bazinet, L., & Pouliot, Y. (2016). Effect of transmembrane pressure control on energy efficiency during skim milk concentration by ultrafiltration at 10 and 50°C. *Journal of Dairy Science*, 99(11), 8655-8664. http://dx.doi.org/10.3168/jds.2016-11504. PMid:27638263.

Ng, K. S. Y., Dunstan, D. E., & Martin, G. J. O. (2018). Influence of processing temperature on flux decline during skim milk ultrafiltration. *Separation and Purification Technology*, 195, 322-331. http://dx.doi.org/10.1016/j.seppur.2017.12.029.

Oliveira, D. L., Grassi, T. L. M., Paiva, N. M., Santana, B. N., Nakamura, A. A., Bermejo-Poza, R., & Ponsano, E. H. G. (2021). Ultrafiltration for the recovery of proteins from surimi washing water. *Food Science and Technology*, 41(Suppl. 2), 458-464.

Prestes, A. A., Helm, C. V., Esmerino, E. A., Silva, R., & Prudencio, E. S. (2022). Conventional and alternative concentration processes in milk manufacturing: a comparative study on dairy properties. *Food Science and Technology*, 42, e08822.

Prudêncio, E. S., Müller, C. M. O., Fritzen-Freire, C. B., Amboni, R. D. M., & Petrus, J. C. C. (2014). Effect of whey nanofiltration process combined with diafiltration on the rheological and physicochemical properties of ricotta cheese. *Food Research International*, 56, 92-99. http://dx.doi.org/10.1016/j.foodres.2013.12.017.

Saha, P., Bajaj, R., Mann, B., Sharma, R., & Mandal, S. (2022). Isolation and characterisation of micellar casein from buffalo milk using microfiltration technique with modified buffer composition. *International Journal of Dairy Technology*, 75(2), 308-320. http://dx.doi.org/10.1111/1471-0307.12844.

Schiffer, S., Hartinger, M., Matyssek, A., & Kulozik, U. (2020). On the reversibility of deposit formation in low temperature milk microfiltration with ceramic membranes depending on mode of adjustment of transmembrane pressure and wall shear stress. *Separation and Purification Technology*, 247, 116962. http://dx.doi.org/10.1016/j.seppur.2020.116962.

Syrios, A., Faka, M., Grandison, A. S. No., & Lewis, M. J. (2011). A comparison of reverse osmosis, nanofiltration and ultrafiltration as concentration processes for skim milk prior to drying. *International Journal of Dairy Technology*, 64(4), 467-472. http://dx.doi.org/10.1111/j.1471-0307.2011.00719.x.

Ulbricht, M. (2006). Advanced functional polymer membranes. *Polymer*, 47(7), 2217-2262. http://dx.doi.org/10.1016/j.polymer.2006.01.084.

Verruck, S., Balthazar, C. F., Rocha, R. S., Silva, R., Esmerino, E. A., Pimentel, T. C., Freitas, M. Q., Silva, M. C., Cruz, A. G., & Prudencio, E. S. (2019). Dairy foods and positive impact on the consumer's health. *Advances in Food and Nutrition Research*, 89, 95-164. PMid:31351531.

Vieira, A. H., Balthazar, C. F., Guimarães, J. T., Rocha, R. S., Pagani, M. M., Esmerino, E. A., Silva, M. C., Raices, R. S. L., Tonon, R. V., Cabral, L. M. C., Walter, E. H. M., Freitas, M. Q., & Cruz, A. G. (2020a). Advantages of microfiltration processing of goat whey orange juice beverage. *Food Research International*, 132, 109060. http://dx.doi.org/10.1016/j.foodres.2020.109060. PMid:32331686.

Vieira, A. H., Balthazar, C. F., Rocha, R. S., Silva, R., Guimarães, J. T., Pagani, M. M., Pimentel, T. C., Esmerino, E. A., Silva, M. C., Tonon, R. V., Cabral, L. M., Freitas, M. Q., & Cruz, A. G. (2020b). The free listing task for describing the sensory profiling of dairy foods: a case study with microfiltered goat whey orange juice beverage. *Journal of Sensory Studies*, 35(5), e12594. http://dx.doi.org/10.1111/joss.12594.

Xia, X., Tobin, J. T., Sharma, P., Fenelon, M., Mcsweeney, P. L. H., & Sheehan, J. J. (2020). Application of a cascade membrane filtration process to standardise serum protein depleted cheese milk for cheddar cheese manufacture. *International Dairy Journal*, 110, 104796. http://dx.doi.org/10.1016/j.idairyj.2020.104796.