NONTHERMAL CONCENTRATION OF MILK BY FORWARD OSMOSIS

A Thesis

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Master of Science

by

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ABSTRACT

Concentration of milk in the dairy industry is typically achieved by thermal evaporation or reverse osmosis (RO). Heat concentration is energy-intensive and leads to cooked flavor and color changes. RO is affected by fouling, which limits its final achievable concentration. The objective of this work was to evaluate forward osmosis (FO) as an alternative method for concentrating milk, and study the effects of fat content and temperature on the process. Pasteurized skim and whole milk were concentrated at 4, 15, and 25 °C, using a benchtop FO unit. Water flux and concentration were monitored, and the quality of the concentrates evaluated. All runs were conducted in triplicate, and data analyzed by ANOVA. Flux decreased with time under all processing conditions. Higher temperatures led to less pronounced flux drops and faster concentration for both skim and whole milk. For skim milk, 40 °Brix was reached after 7h at 25 °C, 8.5h at 15 °C, and 10h at 4 °C. Whole milk concentration was slower, with 30 °Brix achieved after 7h at 25 °C, 8h at 15 °C, and 9h at 4 °C. The sensory quality of FO concentrated and thermally concentrated milk, diluted to single-strength and HTST milk was evaluated by a panel, who did not find significant differences between concentrated and un-concentrated milks. This data suggests FO is a viable nonthermal alternative for concentrating milk.

BIOGRAPHICAL SKETCH

Andreea Beldie was born and raised in Mangalia, a small town on the Black Sea shore in Romania. She is the youngest of three siblings, having an older sister, Alina and an older brother, Ştefan. In middle school, she fell in love with chemistry and this love led her to the College of Applied Chemistry and Material Science at the Politehnica University of Bucharest. As an undergraduate, Andreea pursued research on different subjects and became fascinated by the idea of research.

In 2015, she was one of nine students accepted into the Food Science Summer Scholar Program at Cornell University. The summer scholar experience at Cornell has changed Andreea's perspective of life and career and she decided to pursue a graduate degree at Cornell. In 2016, she graduated as a valedictorian on her specialization and started working as a food engineer for the National Institute of Research and Development for Food Bioresources in Bucharest.

In 2017, she joined Carmen Moraru's lab as a Masters Student and started working on Forward Osmosis. In her two-year program, Andreea participated in two product development competitions. In 2017, her team was awarded the second prize for the product they created for the National Dairy Council Product Development Competition. In 2018, she joined the Food Science Graduate Student Organization as a co-social chair and the Graduate and Professional Women Network as a communications chair.

She was fondly named "the snack queen" by her lab mates because she loves snacking. Thanks to this passion for snacks she was part of the Professional Manufacturing Confectioners Association Student Outreach Program two years in a row, getting to learn more about the confectionery industry.

After graduation, Andreea will be starting a Ph.D. program in the same lab. She is looking forward to continuing her research in nonthermal processing and is hoping to make discoveries that would advance the field of food science, or at least the processing side of it. Additionally, she would love to get the chance to participate in the Cornell SMART program in order to apply the knowledge obtained from her studies.

To my parents, my sister, brother, my nieces and sister in law for love and support.

To my friends, who made me feel like home while far from home and to the best dog that could ever exist, Tasha <3.

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I feel like this list could go on forever, but I could not stop without thanking my SQUAD (Cedric, Ola, and Keqin) from the bottom of my heart! Thank you all for hugs, support, hot pot, ice cream, and gym assistance in times of need! May our friendship only grow stronger! Many thanks to Ana Gaby, my "Mexican sister", fellow TA and amazing friend for being a constant reminder of kindness and happiness.

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LIST OF ABBREVIATIONS

ECP – External Concentration Polarization

FO – Forward Osmosis

HTST – High temperature short time

ICP – Internal Concentration Polarization

IR- Infra Red

MF – Microfiltration

MQIP – Milk Quality Improvement Program

NFDM – Nonfat Dry Milk

NMR – Nuclear Magnetic Resonance

PRO – Pressure Retarded Osmosis

QDA- Qualitative Descriptive Analysis

RO – Reverse Osmosis

UF – Ultrafiltration

(%T3B) – Top three boxes scale

CHAPTER 1
Concentration of milk - a review

1.1. Introduction

Milk concentration is a common unit operation in the dairy industry. There are numerous reasons for which eliminating water from milk is useful or important. Cow's milk consists of ~87.3% water, 4.8% lactose, 3.7% fat, 3.5% protein and 0.7% minerals (TetraPak Dairy Processing Handbook, 1995). When removing water, its main component, the volume decreases significantly, which allows for an increased capacity of storage and transportation. Moreover, milk requires refrigeration in order to prevent microbial growth during storage. The cooling water and the energy required for the cooling process are reduced when the volumes that need cooling are reduced. Additionally, milk concentration is used as a preliminary step in the manufacture of certain dairy products, such as yogurt and particularly before drying in the manufacture of milk powder. When used before drying, which is an energy-intensive process, an intermediate concentration step decreases the energy expenditure.

1.2.1. World production of evaporated milk

While the world production of condensed milk has been showing a predominantly increasing trend, the world production of whole and skim evaporated milk has been decreasing. Concentrated milk is also known as evaporated milk, due to the fact that this has been the main way of concentration used so far in the dairy industry. Concentrated or evaporated milk represents the base for condensed milk, also known as condensed or sweetened condensed milk. Condensed milk contains approximately

45 % of sugar (TetraPak Dairy Processing Handbook, 1995). Starting in the early 2000s, the production of evaporated skim milk has been decreasing steadily, while the one of skim condensed milk has been increasing rapidly, reaching almost 1 mil tonnes (Fig. 1.1).

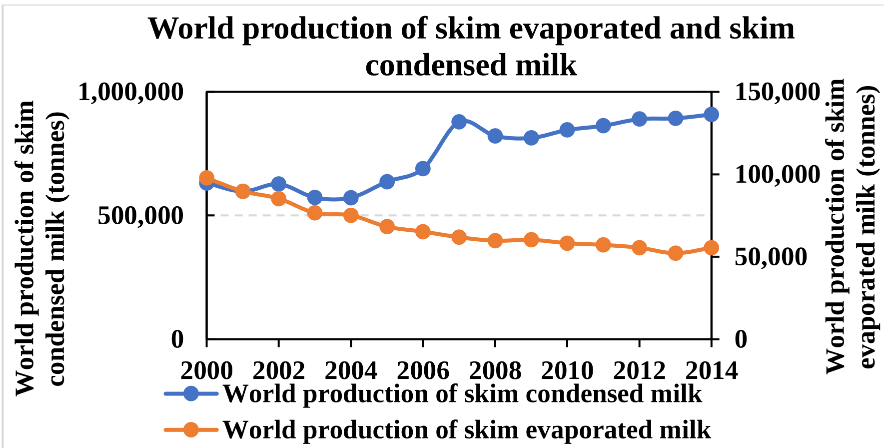

Fig. 1.1. World production of skim evaporated and skim condensed milk. Adapted from: FAOSTAT, July 2019

The world production of whole condensed milk has been slightly increasing between 2009 and 2012 and experienced a slight decrease between 2012 and 2014. The whole condensed milk trend has experienced an overall increase of about 400,000 tonnes from 2000 to 2014. (Fig.1.2).

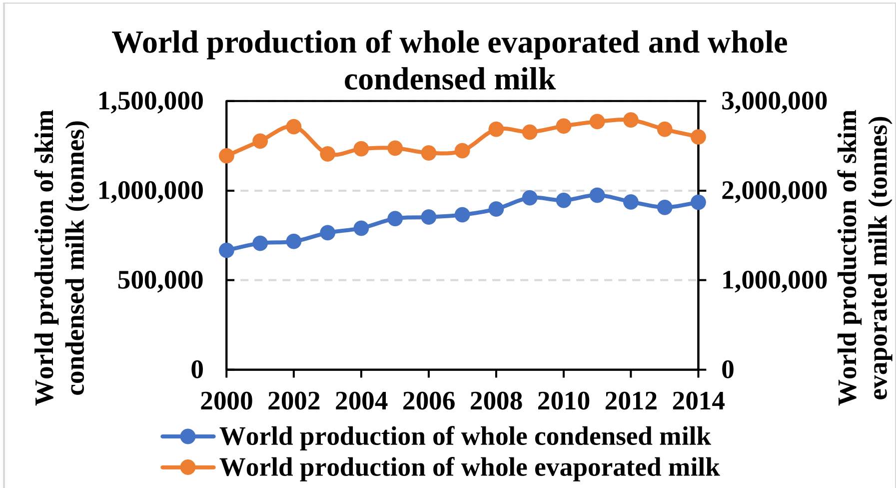

Fig. 1.2. World production of whole evaporated and whole condensed milk. Adapted from: FAOSTAT, July 2019

1.1.1. Milk concentration methods

The traditional method used for milk concentration is thermal evaporation. Other methods include reverse osmosis (RO) (Glover, 1971; Hiddink et al., 2010), , freeze-drying (Hartel and Espinel, 1993) and more recently, forward osmosis (FO) (Chen et al., 2019).

Although milk concentration has been used for more than a century, and new concentration techniques are emerging, there is no review that assesses the various methods of concentration. This work intends to assess these methods and their advantages and disadvantages. In this paper, focus will be placed on thermal evaporation and RO, which are the most widely used techniques, and FO, as a new promising method of concentration.

1.2. Thermal evaporation

The evaporation of milk under vacuum is also known as the “classical” concentration method for milk (Carić et al., 2009). This process was developed and patented in the mid-1800s by Gail Borden Jr., who was the first to create a method of milk preservation that would maintain the quality of fresh milk (Hunziker, 1914; Hickey, 2009)

Thermal evaporation consists of water removal from solution in the form of vapors. It is commonly done under reduced pressure, or partial vacuum, which leads to decreased boiling temperature of the product, with the main purpose of preserving heat-sensitive components. Usually, the liquid comes in contact with a heat exchange surface that is heated up by hot steam from the other side. As the product reaches the boiling point, water vaporizes, and as vapors are removed the concentration of the product increases.

1.2.1. Challenges of thermal evaporation

Evaporation is known to be one of the most energy-intensive operations in the food industry. For optimization purposes, it is run in stages (Fig.1.1). A three-stage evaporation process requires ~800 kJ/ kg of water removed, and a six-stage process needs ~230 kJ/ kg of water removed, excluding mechanical energy. In comparison, RO uses 20-35 kJ/ kg of water removed (Walstra, 2006).

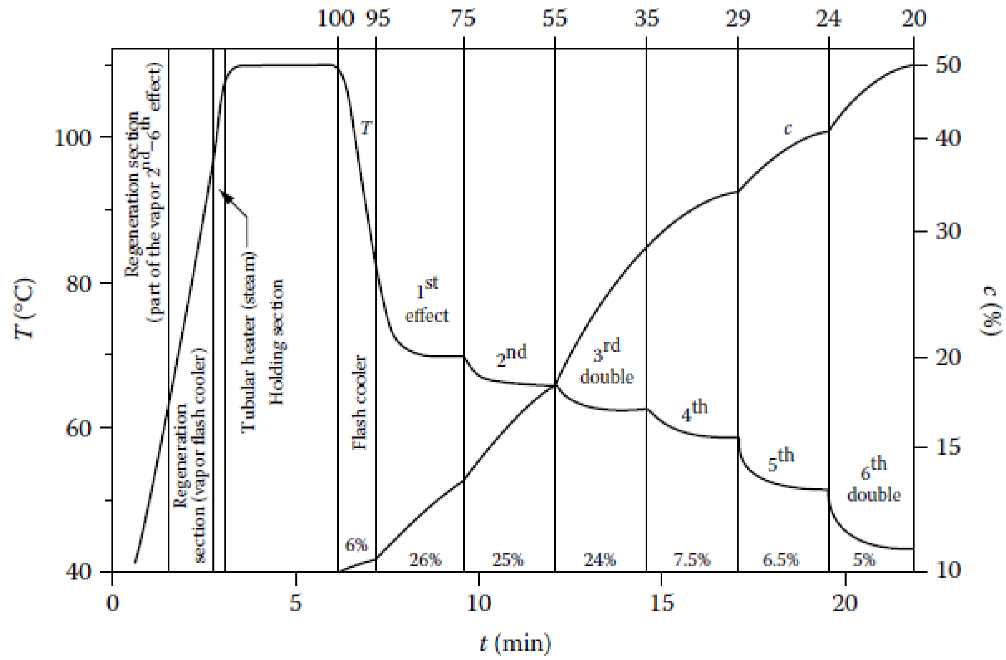

Fig.1.3. Example of the course of temperature (T) and dry matter content (c) of skim milk as a function of time (t) in a six-effect evaporator with preheating. Source: (Walstra, 2006)

In addition to the high energy consumption, during thermal evaporation, this processing step has several undesirable effects on the product quality. First of all, dairy products are heat sensitive and some components, particularly serum proteins proteins and water soluble vitamins, can be damaged by long exposure to high heat. Therefore, the evaporators used in the dairy industry operate under vacuum conditions that enable boiling at moderate temperatures, that can be as low as 40 °C. They are also designed for the shortest possible residence time (Carić et al., 2009).

Despite the fact that thermal evaporation is still the most utilized method of concentration for liquid foods in general, and milk in particular, it is known that it can have a negative impact on the taste, flavor, color, and even the nutritional value of the concentrates (Coday et al., 2014).

Moreover, thermal evaporators provide perfect conditions for survival and even growth of thermophylic spore-forming bacteria. On one hand, the range of temperatures used in the later stages of the evaporation process in multiple effect evaporators (40-65 °C) is ideal for thermophilic bacteria. On the other hand, minerals, particularly calcium, which can be found abundantly in milk and is concentrated in solution as the water evaporation proceeds has been shown to increase the expression of genes promoting the sporulation process (Walstra, 2006). Additionally, it was reported that thermophilic bacilli form biofilms at elevated temperatures (Burgess et al., 2010). Mesophilic and thermophilic bacteria could also have detrimental effects on product quality and safety. Some strains produce proteases, lipases, phospholipases, and even β-Galactosidase, therefore leading to textural defects and off-flavors. Certain spore-formers (e.g. *Bacillus cereus*) are also known to be producing toxins responsible for food poisoning symptoms (Lücking et al., 2013).

In order to prevent the negative effects of bacterial contamination, current methods of controlling these bacilli include shorter production times, the use of sanitizers, altering the process temperatures, and reducing the time spent by the product in the optimal temperature growth zone (Burgess et al., 2010).

1.3. Reverse Osmosis (RO)

The dairy industry uses many membrane separation techniques, including RO, nanofiltration (NF), ultrafiltration (UF), and microfiltration (MF). In membrane processing, a liquid is put in contact with a semi-permeable membrane, which allows only the passing of specific components – either under a pressure or concentration gradient. Different membrane processes can be differentiated according to the membrane pore size, and can be used for the separation of particles with molecular weights within the range of the membrane pore sizes.

Membrane processes can be and are successfully used in the food industry to concentrate liquid foods at low temperatures. Therefore,membrane separation is a promising technique for water removal form milk and other dairy streams, as membrane concentration can be performed without the traditional downsides of thermal concentration that were reviewed above (denaturation of heat-sensitive components, change in sensory attributes or proliferation of mesophilic and thermophilic spore-forming bacteria).

For example, the dairy industry uses RO for water removal, leading to volume reductions and the recovery of total solids and water (Walstra, 2006; Kumar et al., 2013). The nature of RO concentration will be discussed in detail in the following subsection. Another emerging technology that has the ability to remove water from a liquid product is FO, that will also be described later in this paper.

1.3.1. Principle of operation

RO, the membrane technology that is currently used on an industrial scale in the dairy industry for water removal, uses membranes that have pore sizes of about $10^{-4} – 10^{-3}$ μm. RO uses high hydraulic pressures (30- 60 bars) applied to the feed side of the membrane. The water molecules pass through the membrane to the other side, forming the permeate (Fig. 1.4.) (TetraPak Dairy Processing Handbook, 1995). Due to the selectivity of the membrane, the permeate is close to pure water. The concentrated liquid that cannot pass through the membrane is called retentate.

RO concentration is typically operated at temperatures below or around 40 °C when cellulose acetate membranes are used, or at 70-80 °C with composite membranes (Kumar et al., 2013).

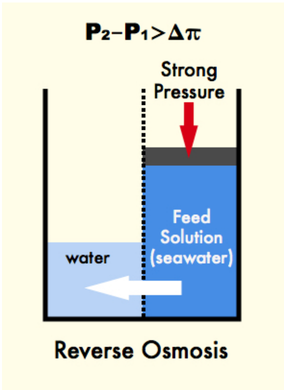

Fig. 1.4. RO principle. Adapted from: https://wernerantweiler.ca/blog.php?item=2015-09-01

1.3.2. Process fundamentals

The efficiency of a membrane separation process can be assessed through the value of the permeate flux passing through the membrane. Permeate flux can be calculated using equation 1.1:

$$J = \frac{m}{A \times t \times \rho} \quad (1.1)$$

Where m is the permeate mass, in kg, A is the membrane filtration area and ρ is the density of the permeate (Menchik and Moraru, 2019).

1.3.3. Challenges of RO

Despite being successfully used in the dairy industry, RO presents a series of disadvantages. Due to long operation times, combined with the high applied pressures, RO membranes are prone to fouling due to blockage of pores, adsorption of particles on the pores, deposition of protein and minerals, cake formation, depth fouling and also biofilm formation (Kumar et al., 2013).

In case of RO of dairy streams (milk, whey), fouling can be attributed to native and denatured dairy proteins, fat or minerals, particularly calcium phosphate, which can precipitate and/or promote the formation of Ca bridges between proteins or between proteins and the membrane constituents. Fat affects the viscosity of the product, therefore reducing the level of concentration that can be achieved by RO alone, leading to the use of a combination of thermal evaporation and RO for the concentration of whole milk (Carić et al., 2009).

Membrane fouling contributes to costs increases of RO operations, due to both a need for additional pre-treatment units to prevent fouling, and chemicals required for cleaning. Additionally, the increasing concentration leads to an increase in pumping energy to maintain the transmembrane pressure, due to increased osmotic pressure on the feed side, therefore increasing the energy consumption (Wenten and Khoiruddin, 2016). Concentration polarization represents a challenge as well, but this will be described later, in the FO section.

Research has focused on finding more efficient cleaning methods by using various chemicals or physical methods such as osmotic backwash. Membrane modification solutions have also been found to be effective in reducing fouling tendencies. More research is however needed to find a membrane material that would increase the performance and efficiency of RO systems (Wenten and Khoiruddin, 2016).

Another disadvantage of RO is the high energy required by the pumps that circulate the feed under high-pressure conditions (Wenten and Khoiruddin, 2016).

1.4. Forward Osmosis (FO)

FO is currently regarded as an innovation in the field of membrane separation. In recent years, it has gained more importance for its potential as a concentration method for liquid foods, such as juices (An et al., 2019), natural colors (Babu et al., 2006), byproducts (acid whey) (Menchik and Moraru, 2019), and also for milk (Chen et al., 2019).

1.4.1. Principle of operation

Compared to RO, UF, MF, and NF, FO is not a pressure-driven membrane process. Like RO, it is an osmotic process that uses a semi-permeable membrane, through which only water molecules can pass. Unlike RO, its driving force is an osmotic pressure gradient, given by the difference in concentration of two solutions placed on either side of the membrane. On one side, there is a highly concentrated sugar or salt solution, also known as the osmotic agent or draw solution and on the other side, there is a less concentrated solution, the feed (Fig. 1.5). Water permeates from the side of the most diluted liquid to the more concentrated side, in order to achieve an equilibrium in concentration.

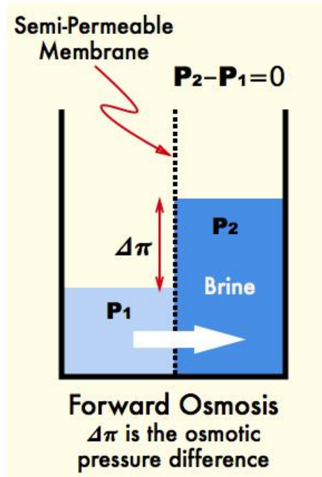

Fig. 1.5. FO principle; Adapted from: https://wernerantweiler.ca/blog.php?item=2015-09-01

Due to the fact that it does not use high pressures, FO is less energy-intensive, having the potential of being more sustainable than pressure-driven processes like RO. Additionally, FO can be used at low temperatures, preserving the organoleptic properties of milk, and preventing microbial growth.

1.4.2. Process fundamentals

In an ideal situation, without concentration polarization, the FO flux can be calculated according to the following equation:

$$J = A(\pi_{D,b} - \pi_{F,b}) \quad (1.2)$$

Where A is the water permeability coefficient of the membrane, $\pi_{D,b}$ is the bulk osmotic pressure of the draw solution, and $\pi_{F,b}$ is the bulk osmotic pressure of the feed.

However, FO can be affected by concentration polarization and reverse solute flux, and as a consequence, it was observed that the experimental flux values in FO are lower than the predicted, theoretical values. Other flux equations were developed in order to take into account those issues.

1.4.3. Challenges in FO

The membranes used for FO are asymmetric, meaning that they are composed of a porous support layer and a more compact one, the active layer. This structure leads to concentration polarization, which represents a major challenge for FO processing. In FO, there are two types of concentration polarization: external (ECP) and internal (ICP). Concentrative ECP occurs on the feed side of the membrane when a build-up of solute accumulates on the active layer. Dilutive ECP occurs on the draw solution side of the membrane, where the draw solution is being diluted by the water that permeates. ICP takes place in the support layer and is exclusive to FO (Gray et al., 2006).

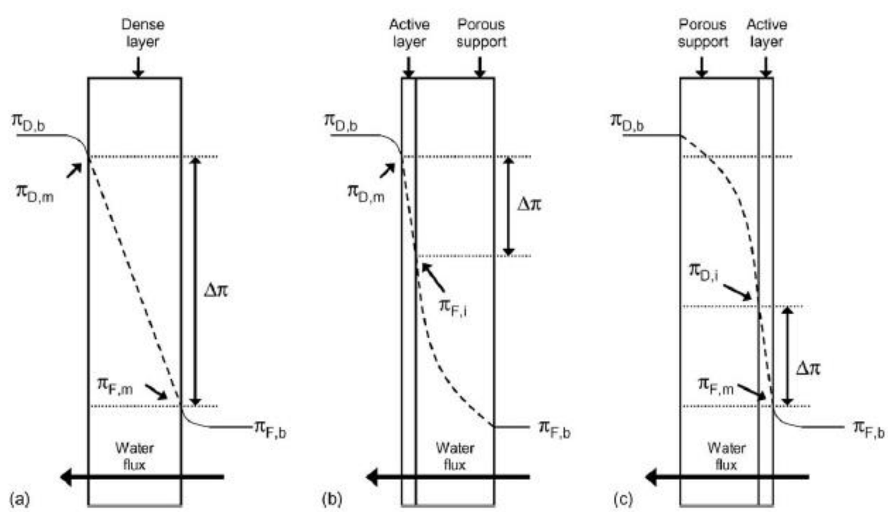

Fig. 1.6. Illustration of osmotic driving force profiles for osmosis through several membrane types and orientations, incorporating both internal and external concentration polarization. (a) A symmetric dense membrane; the profile illustrates concentrative and dilutive ECP. (b) An asymmetric membrane

with the dense active layer against the draw solution (PRO mode); the profile illustrates concentrative ICP and dilutive ECP. (c) An asymmetric membrane with the porous support layer against the draw solution (FO mode); the profile illustrates dilutive ICP and concentrative ECP Source: (McCutcheon and Elimelech, 2006)

When the feed faces the support layer, concentrative ICP occurs as the concentration of solutes is building up in the support layer. When the draw solution faces the support layer, dilutive ICP occurs, as water crosses through the membrane to equilibrate the concentration (Gray et al., 2006).

The need for a draw solution represents another challenging aspect of FO. When used for food applications, the draw solution must be food grade. This way, in case of reverse solute flux, represented by the flow of small amounts of draw solution into the feed in case of a membrane breach, the safety of the food product is not impacted. However, reverse solute flux is not the only reason why the osmotic agent represents a challenge. After the FO process, the diluted draw solution needs to be regenerated, which means that another concentration step needs to be performed. If done by thermal evaporation, by example, the overall energy requirements of the FO process can be significantly increased (Menchik and Moraru, 2019).

1.4.4. FO of milk

FO has been successfully used for water desalination and reclamation, and, more recently, it started to be adopted into the food industry for juice concentration. So far only one study on the FO of milk is available in literature.

Chen et al., 2019 used a cellulose triacetate membrane with an active area of 24 m^2 and sodium chloride (4 – 57 g/L) as the osmotic agent for the concentration of skim milk and whey by FO. They achieved a concentration factor of 2.5 and fluxes of 3.9 L/(m^2h) at 9.3 °C and 4.5 L/(m^2h) at 16.5 °C. They hypothesized that water is not the only substance passing into the draw solution. Accordingly, they determined that lactose was another compound that passed through the membrane, at a flux of 3 g/(m^2h), encompassing 50% of the total organic matter that can be found in the draw solution at the end of the concentration process. This group also investigated the influence of transmembrane pressure on the FO process. Their findings show that an increase in the feed side pressure was more effective than a more concentrated draw solution in enhancing water flux. This study shows that FO shows significant potential for the nonthermal concentration of dairy streams (Chen et al., 2019).

1.4.5. Conclusions

Membrane concentration represents a viable, nonthermal alternative to thermal evaporation. In case of milk, a major advantage is the preservation of its heat-sensitive compounds, the preservation of taste and aroma that would not be negatively impacted by heat, and the avoidance of warm conditions that are crucial in the development of spore-forming bacteria and biofilms. While membrane concentration can be achieved using RO, FO may have some additional advantages as a method for concentration of milk. FO is not a pressure-driven process, and therefore it is not prone to irreversible fouling. Moreover, recent research suggests that FO could be more energy efficient than RO or thermal concentration. There are still many areas to be improved in FO in terms of membrane development, osmotic agent improvement and even in the determination of appropriate parameters to be used for the efficient concentration of milk. Therefore, FO should be further studied and improved as both the demand and production of concentrated milk are increasing.

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CHAPTER 2
Forward osmosis of skim and whole milk

2.1. Introduction

Concentration of milk and its derivatives is a common unit operation in the dairy industry. Often times, concentration is used as an intermediate step in obtaining milk powders for the purpose of saving energy, drying being more energy-intensive when used by itself (Bloore and O'Callaghan, 2009). However, other reasons for which concentration is conducted are the facilitation of subsequent processing, such as crystallization, the decrease in costs needed for storage capacity, cooling, and shipping. Methods used for concentrating milk or other dairy fluids include thermal evaporation, considered the "classical" method (Carić et al., 2009), freeze concentration (Hartel and Espinel, 1993) and membrane separation techniques, namely reverse osmosis (RO) (Carić et al., 2009).

Even though evaporation is considered to have improved significantly in recent years, it is still very energy-intensive (Carić et al., 2009). In addition, thermal evaporation is known to reduce the quality of the final concentrated product, due to prolonged exposure to heat, which can negatively impact the color, taste and the nutritional value of the final concentrate (Coday et al., 2014). Moreover, evaporators are prone to biofilm formation (Deeth and Hartanto, 2009).

RO is another concentration technique presently used in the dairy industry, which has as main advantages lower energy consumption and lower heat exposure of the product as compared to thermal evaporation, and can be used either standalone (Abbot et al., 1979; Hiddink et al., 2010) or combined with UF (Glover, 1971). However, RO is significantly affected by membrane fouling, which leads to flux decline and limits the final achievable concentration of the product, which is why RO is often used in combination with thermal evaporation (Carić et al., 2009). Additionally, the very high pressure used in RO systems can damage the fat globules in milk.

Forward osmosis (FO) is also an osmotic membrane process that can be used to concentrate liquid foods. As opposed to RO, it does not use externally applied pressure, but a large osmotic pressure differential across the membrane, generated by two aqueous solutions that have different osmotic pressures situated on either side of the membrane (Rastogi, 2016). FO represents a very attractive alternative for food applications, due to the gently, nonthermal process and low energy consumption. Since it is less prone to fouling, FO can attain higher concentration levels than RO (Rastogi, 2016).

FO has recently gained more attention in the food industry as a means of concentrating products like: fruit juices (An et al., 2019; Jiao et al., 2004), natural colors (Babu et al., 2006, Nayak and Rastogi, 2010) or acid whey (Menchik and Moraru, 2019).

The objective of this paper is to evaluate the potential of FO as a method to concentrate whole and skim milk at low temperatures.

2.2. Materials and methods

2.2.1. Materials

Batches of 15 L of skim or whole pasteurized milk were purchased from the Cornell Dairy Plant (Ithaca, NY) and kept under refrigeration at 5±1°C for one day before processing.

Thermally concentrated milk of about 50 °Brix and the base for it were kindly provided by Land O'Lakes (Carlisle, PA).

2.2.2. FO processing

The FO concentration runs were conducted using batches of 8 L of milk. The remaining milk was used for physicochemical, microbiological and sensory analysis. The FO rig was equipped with a spiral-wound cellulose triacetate membrane (Ederna, Toulouse, France) with an outside diameter of 63 mm, a length of 530 mm, and a filtration area of 0.5 m^2. The milk was circulated from the feed tank through a countercurrent plate heat exchanger and then into the membrane, using a centrifugal pump. The temperatures chosen for the FO runs were refrigeration temperature (4 °C), ambient temperature (25 °C) and one temperature in between (15 °C). The concentrate was recirculated into the feed tank. The concentrate valve on the outlet of the membrane was used to adjust the pressure as needed. In parallel, the OA at 60 ° Brix was circulated by another pump on the other side of the membrane, where it collected water, becoming dilute or spent OA. The spent OA was collected in a different vessel. The difference in mass over time was recorded using scales and used for calculating flux. The temperature of the product was monitored using a thermocouple incorporated in the membrane rig. The pressure was measured using pressure gauges installed in the feed, concentrate, and osmotic agent sides, respectively. The concentration of the retentate, expressed as °Brix, was measured every 15 min using a digital refractometer (Sper Scientific 300053 – Scottsdale, AZ). The process was run until the pressure limits of the membrane were reached, or until the desired concentration was achieved.

FO membrane cleaning and storage. After each experiment, the rig was cleaned. First, it was rinsed three times with DI water for 2 min each time. The rinse was performed between all cleaning steps. An initial citric acid wash (0.4%) of pH ~ 4 was applied for 11 min at 3 bars, then for 4 min at 1 bar. Citric acid was used as it is known to be less corrosive and milder to the membrane surfaces and because citrate is known to chelate calcium ions more strongly than phosphate (Carić et al., 2009). The acid wash was followed by an alkaline wash using 40 drops of Ultrasil-110 (Ecolab, Saint Paul MN) in 2 L of water, and was performed for 11 min at 3 bars and for 4 min at 1 bar, at a pH of about 8; then by an enzymatic cleaning with 4 mL of Prolyve 1000 protease (Soufflet Biotechnologies, Colombelles, France) in 2 L of water at pH 7 ran for 1 h at 1 bar. After that, an additional citric wash was applied using the same procedure as the first time, followed by a disinfection step with hydrogen peroxide (1%) for 20 min at 3 bar and 10 min at 1 bar. The OA side was rinsed with water until the refractometer read 0.0 °Brix and then was sanitized concomitantly with the membrane side using the same concentration of hydrogen peroxide. For whole milk runs, the plate heat exchanger was disassembled, washed with soapy water, rinsed, disinfected with 200 ppm bleach solution, rinsed again and then reassembled.

This cleaning procedure was developed in consultation with the FO equipment manufacturer (Ederna, Toulouse, France). To verify the cleaning efficiency, water fluxes were performed before and after every run. After processing, the membrane rig was left soaking in 0.5% sodium metabisulfite solution to prevent microbial growth.

2.2.2. Osmotic agent regeneration

The spent OA (40–50 °Brix) was regenerated using an Anhydro Laboratory Vacuum Evaporator, Model Type E. When the regenerated OA had more than 60 °Brix, it was diluted with DI water to 60 ± 0.2 ° Brix before the next use, to ensure consistency among the different experimental runs. After use, the evaporator was rinsed with water and then a caustic wash was performed with 0.5 % Interest (Diversey Inc, Sturtevant, Wisconsin). After an additional water rinse, the evaporator was sanitized using a 0.6 % solution of Divosan Plus (Diversey, Inc., Charlotte, NC).

2.2.4. Physicochemical analyses

The initial milk, the FO concentrate and reconstituted FO milk to single strength concentration were analyzed for concentration level as °Brix (Sper Scientific 300053 – Scottsdale, AZ), water activity (AquaLab series 3 – Meter, Pullman, WA), pH (Thermo Scientific Orion Star A214 pH/ISE Benchtop Meter, Waltham, MA), color (Konica Minolta CR-400 Chromameter, Tokyo, Japan), total solids (AOAC 920.107 + 925.23 A) (Blue M Stabil-Therm Pro-Tronix II, Electric Oven, Blue Island, Illinois), (Mettler Toledo AE 240, Columbus, OH) and milk composition (FOSS, Milkoscan Minor, Hillerød, Denmark). Conductivity (Conductivity (TDS), Type 700, Chemtrix Inc., Hillsboro, OR) and nuclear magnetic resonance (NMR) were used to check for the presence of lactate as an indication of reverse solute flux. Lactate was chosen instead of potassium since this mineral naturally occurs in milk and the reverse solute flux would add only a negligible amount of potassium compared to its initial concentration in milk. Lactate, on the other hand, only occurs in small quantities in milk and changes in its concentration can be easily identified by NMR.

2.2.5. Sensory analysis

The sensory analysis consisted of three parts:

1. An initial assessment of the skim milk that was used for nonthermal and thermal concentration;
2. A large-scale consumer test that looked at nonthermally and thermally concentrated milk reconstituted to single strength, and;
3. A Qualitative Descriptive Analysis (QDA) in which nonthermal and thermal concentrates were assessed.

All the tested samples were assessed microbiologically for Standard Plate Count and coliforms by the Milk Quality Improvement Program (MQIP) at Cornell University (Ithaca, NY).

2.2.5.1. HTST skim milk assessment

The first part consisted in assessing the starting material (HTST skim milk) used for FO and thermal concentration by a group of highly trained individuals (n=5, 4 women and 1 man) of the Cornell Milk Quality Improvement (MQIP) Panel.

2.2.5.2. Large scale consumer test

For this test, over 120 participants (77 females, 44 males), with ages ranging from 18 to greater than 54, were recruited after being prescreened as skim, 1% and 2% milk drinkers, that consume milk no less than once per week as a plain glass of milk or with their cereals.

The nonthermally concentrated milk and the thermally concentrated milk were reconstituted to single strength (around 10 °Brix) by adding filtered water, purchased from the store (Great Value). After achieving the same °Brix as the initial milk, the milk composition was assessed by IR (FOSS, Milkoscan Minor, Hillerød, Denmark) and adjusted if needed to match the composition of the unconcentrated milk. Consumers were given three test samples: one of HTST skim milk (Cornell Dairy), one of reconstituted nonthermally (FO) concentrated milk and one of reconstituted thermally concentrated milk. About 30 mL milk samples were poured into 150 mL cups that were coded with random 3-digit blinding codes obtained from the RedJade software and then covered with lids. The samples were tempered to 10 ± 2 °C prior to the sensory tests. The consumers were asked to answer a few demographic questions and then proceeded into the test. They were presented with the samples monadically, according to the distribution determined by the RedJade software (Curion, Deerfield, IL). The consumers assessed the milk samples for the overall liking of appearance and flavor on a 9-point hedonic scale, where 1 is "dislike it extremely" and 9 is "like it extremely". Just-about-right (JAR) questions were asked for sweetness, cooked flavor, milk flavor intensity, color, and consistency/mouthfeel/viscosity. The JAR questions were developed on a 5-point scale, ranging from 1, that was "not sweet enough", "not cooked enough”, “not nearly thin/viscous enough for a skim milk”, “too white”, and “tastes much too weak/watered down/flat in flavor” to 5, that was “much too sweet”, “much too cooked”, “much too concentrated/intense in flavor”, “too dark/yellow”, and “much too thick/viscous for a skim milk”, for each category. They were also asked if they could perceive any off-tastes and asked to describe them.

Additionally, consumers were asked to conduct a triangle test, in which they compared reconstituted FO concentrated milk to HTST milk that was never concentrated. During this test, they were offered three milk samples, two of which were the same (either reconstituted samples that were initially nonthermally concentrated or HTST pasteurized skim milk samples). The samples were randomized using the RedJade software, and consumers were asked to determine which sample was different. RedJade software was used for data collection and analyses. The participants of the study were compensated with $5.

2.2.5.3. QDA panel test

The Qualitative Descriptive Analysis was performed by a descriptive panel (n=7) selected from the MQIP panel and people who received the MQIP training but were not panelists. The panelists received an initial training of 1h, during which a lexicon for concentrated milk was developed. During the test, they received three samples of nonthermally concentrated milk and three of thermally concentrated milk, respectively. The samples were served simultaneously. The descriptive terms for aroma were: milky - associated with fluid milk; milk powdery – associated with milk powder/NFDM; chemical/medicinal – associated with machines, equipment, medicine, cough syrup, metallic, electrical burn; cheesy – associated with old or aged cheese, stale, macaroni and cheese powder; cooked – associated with cooked milk). Descriptors were identified for other attributed, as follows: for appearance (color); for texture (viscosity, adhesiveness, grainy); for taste (sour, bitter, salty, savory/umami, sweet); for flavor (cooked, caramel, cheesy, milky, milk powdery, chemical/medicinal); for aftertaste (astringent, bitter, salty, sour, chemical/medicinal), and other (film-like mouth coating, irritation). The samples were evaluated using a 0 to 100 scale in which 1 is "none" and 100 is "extremely".

2.3. Results

2.3.1. FO concentration of skim vs whole milk

Fig. 2.1. and 2.2. show the change in concentration and flux versus time in FO of whole milk and skim milk, respectively. In both cases, the permeate flux decreased with time, which likely happened due to the gradually increasing viscosity of the concentrate, as well as internal and external concentration polarization phenomena. In all cases, cleaning enabled the full recovery of the membrane function, as indicated by water fluxes recorded after each cleaning.

FO was able to achieve higher concentration levels for skim milk (40 ° Brix) compared to whole milk (35 ° Brix). Permeate fluxes were also higher for the FO of skim milk compared to whole milk. The negative impact of fat on FO concentration was likely caused by the presence of fat globules, which have high rigidity at low temperatures and may interfere with the transport of water. This hypothesis is supported by the increase in the final concentration level with increasing processing temperature for whole milk, as a final concentration of : 32.4 ± 0.7 °Brix was obtained at 25 °C compared to ~29.1 ± 3.1 °Brix at 15 °C and 27.8 ± 1.4 °Brix at 4 °C (Fig.2.1). Additionally, the permeate flux after 8h dropped to 52 % of the initial flux at 25 °C, 30 % at 15 °C and 24 % at 4 °C (Fig. 2.1).

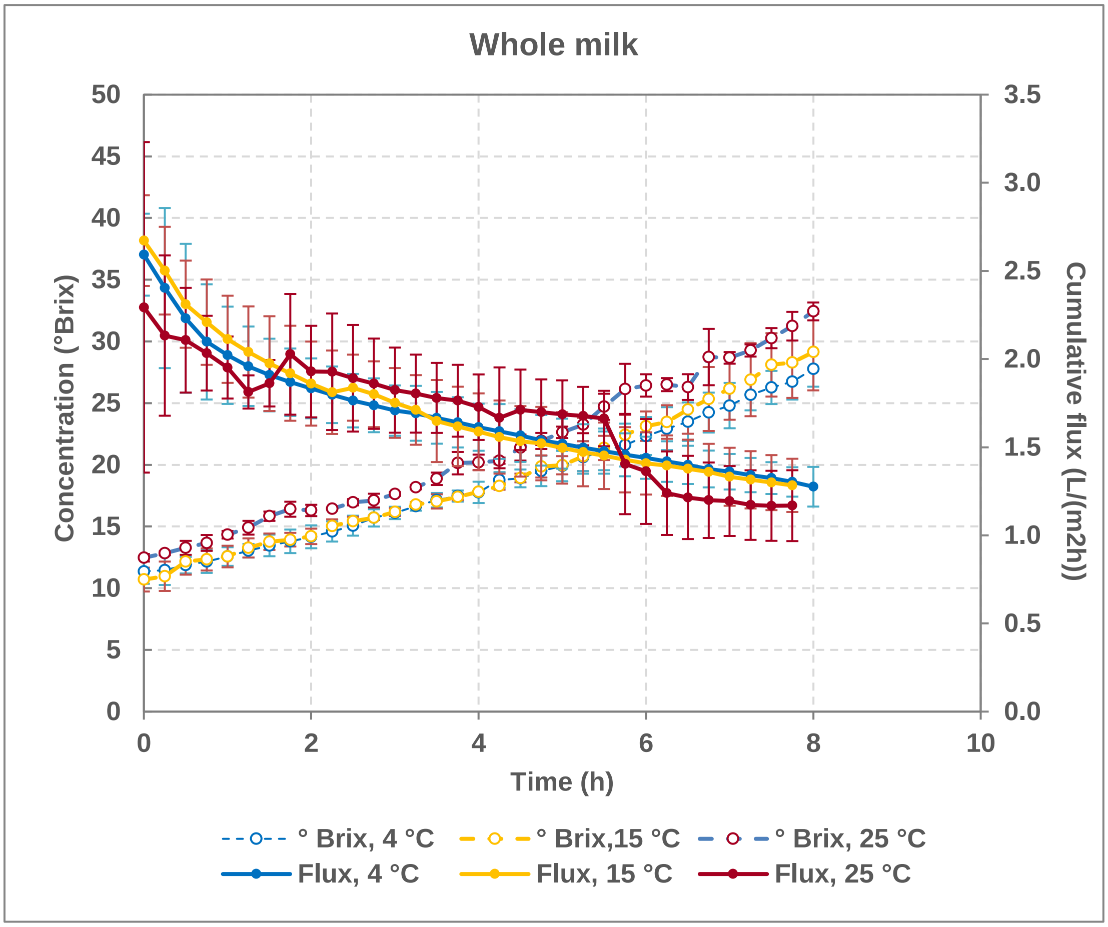

Fig. 2.1- Concentration and flux versus time in FO of whole milk

Surprisingly, for skim milk, although the higher temperatures allowed for faster FO concentration, the highest concentration (~40 °Brix) was achieved at 4 °C. After 5.75 h, a concentration of 35 °Brix was achieved at 25 °C and 15 °C, while at 4 °C the concentration only reached ~30 °Brix (Fig. 2.2). For skim milk, water flux at the beginning of the process (15-20 min) was 2.21 ± 1.64 L/ (m^2h) at 25°C, 2.98 ± 0.62 L/ (m^2h) at 15°C, and 2.52 ± 0.39 L/ (m^2h) at 4 °C. These values were not statistically different ($P > 0.05$).

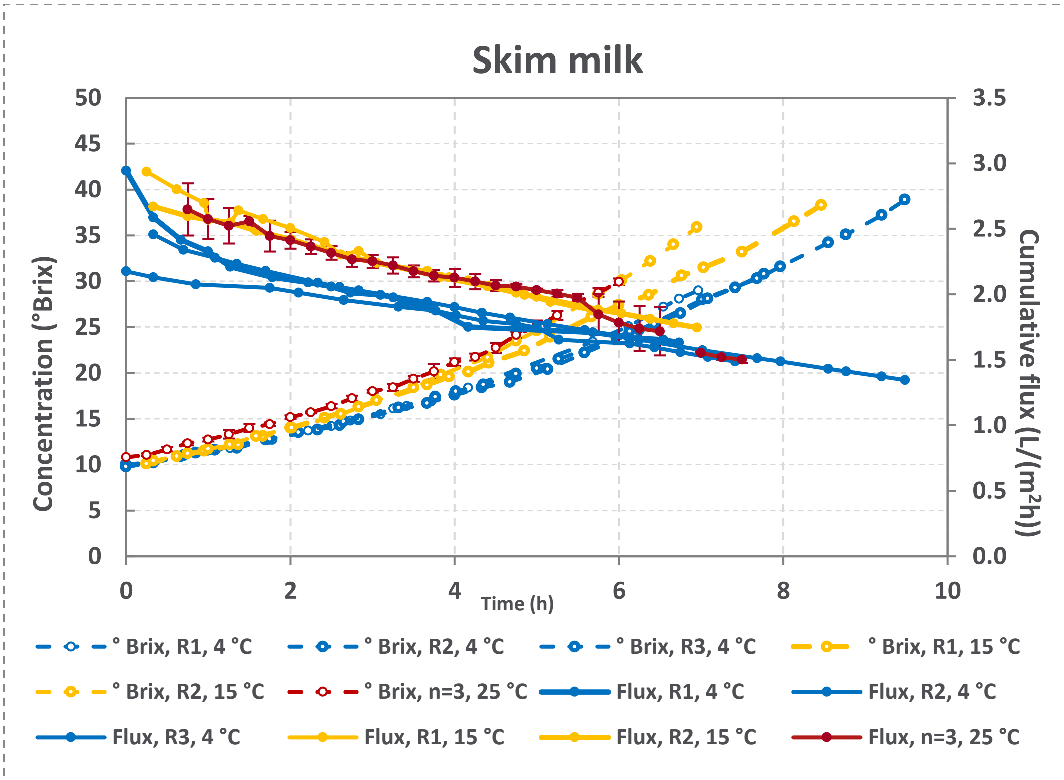

Fig. 2.2- Concentration and flux versus time in FO of skim milk

2.3.2. Quality of the FO concentrates

Concentration of milk has the potential to increase its shelf life due to the reduction of water activity and pH. The water activity decreased significantly after concentration from 0.994 to 0.973 in skim milk, and from 0.993 to 0.975 in whole milk ($p < 0.01$). The pH values were found to be significantly lower as well ($p < 0.01$). For skim, pH decreased from 6.72 to 6.36 and for whole milk, from 6.74 to 6.47.

No significant differences ($p > 0.05$) in color, represented by L values, were found between the initial, concentrated, and reconstituted milk, for both skim and whole milk.

2.3.3. Assessment of reverse solute flux

Previous work on forward osmosis of milk has shown the presence of reverse solute flux (Chen et al., 2019). For this work, conductivity measurements and NMR were used to determine whether the reverse solute flux was present. When comparing the initial sample to one that was concentrated by a factor of ~3.5, conductivity increased with concentration from an average of 3962.5 μS to an average of 5814.17 μS ($p < 0.05$) for whole milk, and from 4416.7 μS to 7650 μS for skim milk.

Pasteurized milk and reconstituted milk were analyzed. While the conductivity values increased from an average of 3962 μS to of 4075 μS for whole milk and from 4416.7 μS to 4800 μS for skim milk, the difference was not statistically significant. ($p > 0.05$). Since these values could have been a result of the precision of the instrument, NMR analyses were conducted to check for the presence of lactate in the concentrated milk. These analyses were performed only on skim milk. NMR spectra showed a modification at 1.33 ppm, which indicates the presence of lactate only in the concentrated sample (Fig. 2.3). This confirms, therefore, the occurrence of reverse solute flux. Its potential impact on sensory attributes will be discussed in subsection 2.3.4.4.

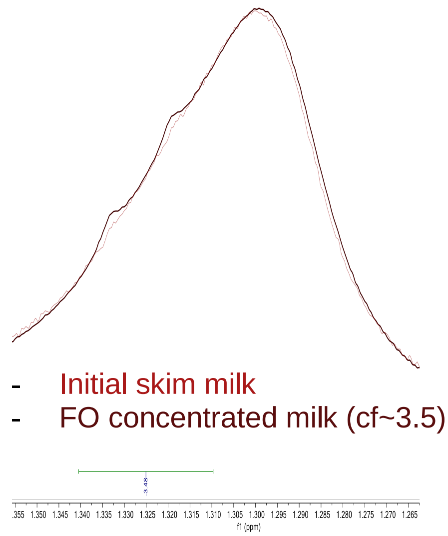

Fig. 2.3. NMR spectra of initial and FO concentrating skim milk showing a modification at 1.33 ppm, that corresponds to potassium lactate

2.3.3. Sensory evaluation of the FO concentrated milk

2.3.3.1. Sensory evaluation of the initial (unconcentrated) skim milk

Both initial milk samples that were to be concentrated either thermally or nonthermally were found to be within "good" quality range (Clark, Costello, Drake, and Bodyfelt, 2009) with the mean Overall Score of 9.4 out of 10 and no sensory criticism for the non-thermal concentrate base and 8.7 out of 10 with "cooked" flavor noted by 4 of 5 panelists for the thermal concentrate base. Based on these results it was decided that having a similar quality of the initial material, the eventual differences that would appear when comparing the samples diluted to single strength could be attributed to the processing technique used for concentration.

2.3.3.2. Large scale consumer test

On a Top three Boxes scale (%T3B), that sums up the results for “Like it extremely”, “Like it very much” and “Like it moderately” for appearance, flavor and overall liking, the three types of milk were appreciated similarly. In terms of appearance, 71% of the consumers preferred the single strength thermally concentrated milk, 66% preferred the control and 65% preferred the nonthermally concentrated milk. These values were not statistically significant ($p > 0.05$).

In terms of overall liking alone, the participants chose in a proportion of 60% the reconstituted thermally concentrated milk. 57% preferred the reconstituted nonthermally concentrated milk and only 53% preferred the HTST milk. This difference is not statistically significant ($p > 0.05$), which suggests that both types of reconstituted milk would not be perceived as different from a regular HTST milk purchased from the store. Even though the results are similar for thermally and nonthermally concentrated milk, the advantage of the nonthermal consists of increased safety and potentially lower energy expenditure when compared to thermal evaporation.

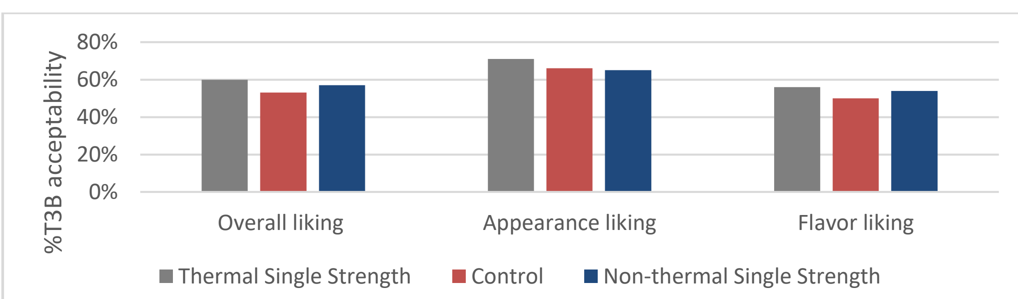

Fig. 2.4 - (%T3B) of HTST skim milk, reconstituted thermally concentrated milk and reconstituted nonthermally concentrated milk

2.3.3.4. QDA panel test

On a scale of 1 to 100, in which 1 is "none" and "100" is extremely, the QDA panel assigned an average score (n=3) of 40 ± 6.57 for the chemical/medicinal aroma to the nonthermally concentrated milk, and a score of 15 ± 8 to the thermally concentrated one ($p = 0.02$) (Fig. 4).

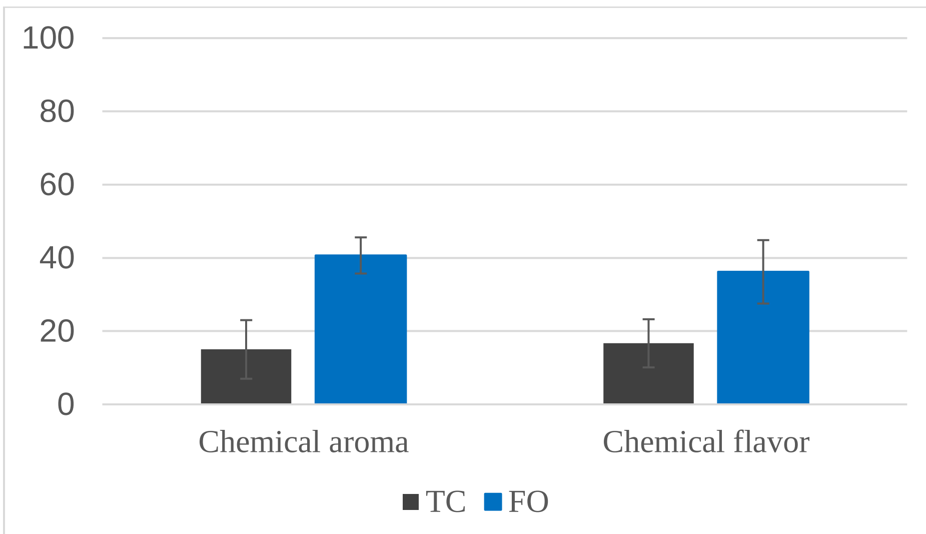

Fig. 2.5 – Chemical/ Medicinal aroma intensity of thermally and nonthermally concentrated milk at ~35 ° Brix

This aroma could be attributed to the long exposure to the membrane rig, as this is a small batch process. This effect might diminish once this process would be upscaled and converted to a continuous process. Another reason for this aroma could be represented by the existing reverse solute flux (confirmed by NMR qualitatively) (Fig 2.3). , and also by conductivity (Subsection 2.3.2). Another significant difference was found in viscosity ($p = 0.01$). The score for nonthermal was 34.28 ± 2.5 and for thermal was 48.71 ± 1.0 (n=3). No other significant differences were found among the two groups.

When looking at the averaged values for the intensity of “cooked” flavor, the values though different, are not statistically significant ($p > 0.05$) (Fig. 2.6.). This could be attributed to the fact that the panelists that analyzed these samples were trained to find specific defects in single strength milk and their additional training on concentrates might not have been enough to have them accustomed to this product. Therefore, the term “cooked” might have been misleading because its perception varied randomly among thermal and nonthermal samples as shown in Fig. 2.7.

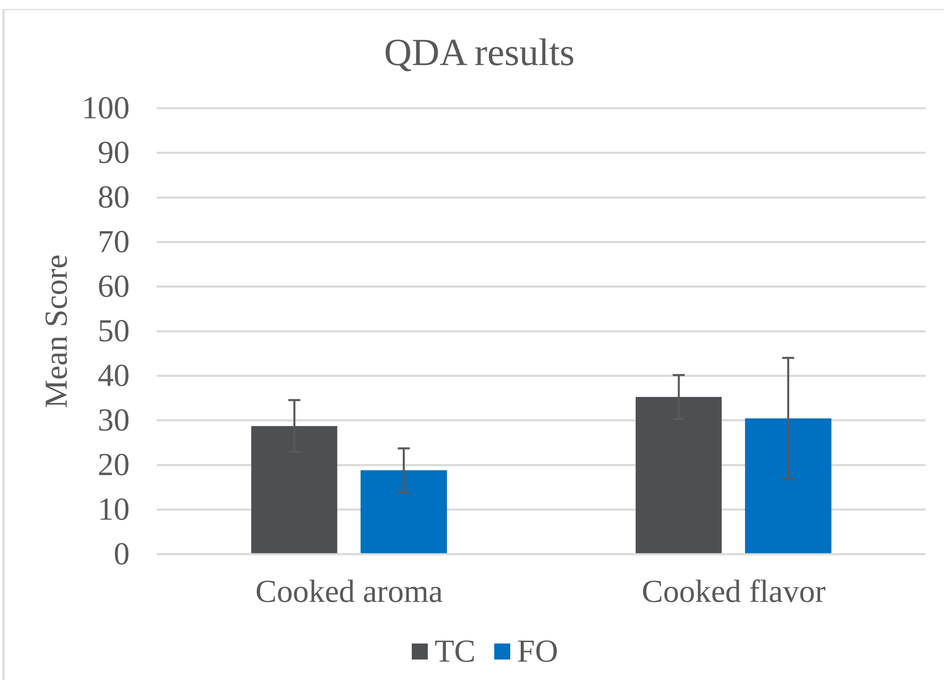

Fig. 2.6 – Averaged cooked aroma intensity of thermally and nonthermally concentrated milk at ~35 ° Brix

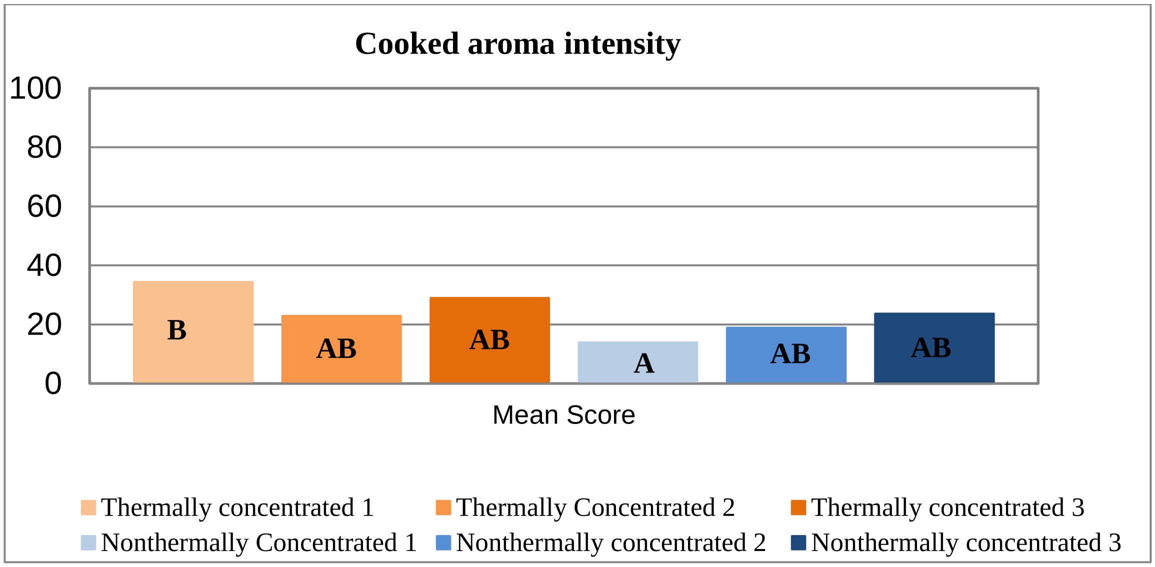

Fig. 2.7 – Cooked aroma intensity of thermally and nonthermally concentrated milk at ~35 ° Brix

2.4. Conclusions

FO can be successfully used for the concentration of skim and whole milk, achieving concentrations as high as 40° Brix, and 35 ° Brix, respectively. It can also be used at low temperatures, assuring the safety of the product.

As shown by the large-scale consumer test, when reconstituted, nonthermally concentrated milk can't be differentiated by HTST skim milk or thermally concentrated milk, proving that milk retains its sensory attributes.

We developed a lexicon that can be used for future studies on milk concentrates. However, more training might be needed for getting a better understanding of the term "cooked".

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