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## Process efficiency of casein separation from milk using polymeric spiral-wound microfiltration membranes

D. Mercier-Bouchard,\* S. Benoit,\* A. Doyen,\* M. Britten,† and Y. Pouliot\*<sup>1</sup>

\*STELA Dairy Research Center, Institute of Nutrition and Functional Foods, Department of Food Sciences, Université Laval, Québec, QC, Canada, G1V 0A6

†Food Research and Development Centre, Agriculture and Agri-Food Canada, St-Hyacinthe, QC, Canada, J2S 8E3

### ABSTRACT

Microfiltration is largely used to separate casein micelles from milk serum proteins (SP) to produce a casein-enriched retentate for cheese making and a permeate enriched in native SP. Skim milk microfiltration is typically performed with ceramic membranes and little information is available about the efficiency of spiral-wound (SW) membranes. We determined the effect of SW membrane pore size (0.1 and 0.2  $\mu\text{m}$ ) on milk protein separation in total recirculation mode with a transmembrane pressure gradient to evaluate the separation efficiency of milk proteins and energy consumption after repeated concentration and diafiltration (DF). Results obtained in total recirculation mode demonstrated that pore size diameter had no effect on the permeate flux, but a drastic loss of casein was observed in permeate for the 0.2- $\mu\text{m}$  SW membrane. Concentration-DF experiments (concentration factor of 3.0 $\times$  with 2 sequential DF) were performed with the optimal 0.1- $\mu\text{m}$  SW membrane. We compared these results to previous data we generated with the 0.1- $\mu\text{m}$  graded permeability (GP) membrane. Whereas casein rejection was similar for both membranes, SP rejection was higher for the 0.1- $\mu\text{m}$  SW membrane (rejection coefficient of 0.75 to 0.79 for the 0.1- $\mu\text{m}$  SW membrane versus 0.46 to 0.49 for the GP membrane). The 0.1- $\mu\text{m}$  SW membrane consumed less energy (0.015–0.024 kWh/kg of permeate collected) than the GP membrane (0.077–0.143 kWh/kg of permeate collected). A techno-economic evaluation led us to conclude that the 0.1- $\mu\text{m}$  SW membranes may represent a better option to concentrate casein for cheese milk; however, the GP membrane has greater permeability and its longer life-time (about 10 yr) potentially makes it an interesting option.

**Key words:** microfiltration, polymeric spiral-wound membrane, skim milk, process efficiency, energy consumption, membrane fouling

### INTRODUCTION

Pre-concentration of milk before cheesemaking has been extensively performed by UF (Pouliot, 2008) but it can now be achieved by microfiltration (MF). With a process using a pore size diameter of approximately 0.1  $\mu\text{m}$ , CN micelles of average size 0.13 to 0.16  $\mu\text{m}$  are rejected, whereas serum protein (SP), lactose, and soluble calcium are recovered on the permeate side (Saboya and Maubois, 2000; Fox and McSweeney, 2003). Pre-concentration of milk by MF offers many benefits: CN-enriched retentates retain their functionality and nutritive properties and permeates with native SP do not contain any residual coagulant, starter culture, lactic acid, or color from the cheesemaking process, which allows SP to have superior functional advantages compared with whey (Britten and Pouliot, 1996; Nelson and Barbano, 2005; Karasu et al., 2010).

Two types of membranes are commonly used for MF processes: tubular ceramic membranes and spiral-wound (SW) membranes (Cheryan, 1998; Hu et al., 2015). Tubular ceramic membranes are a popular choice of membrane material for MF applications due to their high thermal resistance, narrow pore size distribution and high hydraulic performance (Zulewska et al., 2009; Fernández García and Rodríguez, 2015). Over time, alternatives have been developed to prevent membrane fouling, which is the main challenge in MF (Guerra et al., 1997). Fouling of MF membranes was reduced by the emergence of the hydraulic concept of the uniform transmembrane pressure (UTP) membrane and then by graded permeability (GP) membranes, which are based on a variation of the porosity in the structure (Saboya and Maubois, 2000; Garcera and Toujas, 2002). More recently, 0.1- $\mu\text{m}$  GP membranes were shown to be efficient in separating CN from skim milk (Hurt et al., 2015). Although several studies have character-

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<sup>1</sup>Corresponding author: yves.pouliot@fsaa.ulaval.ca

ized the separation performance of MF membranes, very few evaluated process efficiency and quantification of energy consumption in MF. The study done by Tremblay-Marchand et al. (2016) showed that when the CF was increased to  $3.0\times$  for a  $0.1\text{-}\mu\text{m}$  GP membrane, energy consumption and total CN loss increased, whereas the SP removal rate decreased. Diafiltration allowed an increase in total SP removal but resulted in a substantial increase in energy consumption and CN losses (Tremblay-Marchand et al., 2016).

The recent introduction of SW MF membranes has attracted some attention. Zulewska and Barbano (2013) studied the separation performance of  $0.3\text{-}\mu\text{m}$  SW membranes, but important fouling of the membrane elements by CN micelles occurred whereas the much smaller SP contributed to a minor amount of membrane pore constriction. With this pore size diameter, the cake layer accumulating at the membrane surface dominates the separation, rather than the membrane itself (Hu et al., 2015; Steinhauer et al., 2015). Compared with GP and UTP membranes, the  $0.3\text{-}\mu\text{m}$  SW membrane produced the lowest SP removal rate with a value of 39%, whereas GP and UTP membranes could remove up to 61 and 64%, respectively (Zulewska and Barbano, 2013). Lawrence et al. (2008) also evaluated the performance of SW membranes for CN and SP separation in milk, using 0.3 and  $0.5\text{ }\mu\text{m}$  pore diameters. Casein rejection increased with the TMP, from 96% to almost 100%, whereas SP transmission decreased from 22 to 1%, as the TMP increased from 50 to 258 kPa (Lawrence et al., 2008). After one sequential DF, about 50% of the total SP was removed to the permeate side. Beckman et al. (2010) also evaluated milk protein separation performance using a  $0.3\text{-}\mu\text{m}$  SW membrane at  $50^\circ\text{C}$  with a TMP of 100 kPa to a CF of  $3.0\times$  and successfully removed 70% of the SP after 2 sequential DF. Zulewska and Barbano (2014) evaluated the effect of DF on the efficiency of SP removal during MF of skim milk. After reaching a CF of  $3.0\times$  and 2 sequential DF, an additional 22 and 7% of the SP was removed after each DF, respectively, giving a total SP removal of 97% (Zulewska and Barbano, 2014). Govindasamy-Lucey et al. (2007) showed that SW membranes with a pore size of  $0.2\text{ }\mu\text{m}$  could produce retentates with an increased CN to true protein (TP) ratio and reduced SP content. It can be hypothesized that with a smaller pore size diameter ( $0.1$  or  $0.2\text{ }\mu\text{m}$ ), the process efficiency of CN and SP separation in milk increases, especially because these membranes offer important advantages in cost, compared with ceramic membranes (Papadatos et al., 2003; Beckman and Barbano, 2013; Hu et al., 2015).

The objective of this study was to characterize the performance of  $0.1\text{-}$  and  $0.2\text{-}\mu\text{m}$  polyvinylidene difluoride (PVDF) SW membranes by MF at  $50^\circ\text{C}$  in terms

of gain in yield and process efficiency for the separation of CN from milk, compared with GP membrane performance. The investigation on each type of MF membrane was designed to help guiding industrial decisions based on the needs, values, and constraints of dairy processors.

## MATERIALS AND METHODS

### Raw Material

Pasteurized skim milk was purchased from a local dairy supplier (Natrel, Québec, Canada) and stored at  $4^\circ\text{C}$  until use in MF experiments. For total recirculation experiments performed with SW membranes of  $0.1$  and  $0.2\text{ }\mu\text{m}$  pore diameters, 2 batches of skim milk, each divided into 3 equal parts, were microfiltered. For single-stage concentration-DF modes with  $0.1\text{-}\mu\text{m}$  SW membrane, one batch of skim milk was divided into 3 equal parts of about 280 L. Total recirculation and concentration DF experiments were carried out in triplicate.

### MF System

The MF system (model 393, Tetra Pak Filtration Systems, Champlin, MN) was the same as described by Tremblay-Marchand et al. (2016). Briefly, it was equipped with 2 feed pumps (model LKHP-10, Alfa Laval and model H-25, Wanner Engineering Inc., Minneapolis, MN) and 1 recirculation pump (model LKHP-10, Alfa Laval). Flow rates were measured with electromagnetic flow meters (model SM2001, IFM Electronic, Malvern, PA). A pressure sensor (model PF265x, IFM Electronic) measured the inlet and outlet retentate pressures as well as permeate pressure at the outlet. The system is composed of 3 stages with 3 filtration loops but this study used only one stage with 1 or 2 SW MF membranes installed in series in the loop. The  $0.1\text{-}$  or  $0.2\text{-}\mu\text{m}$  pore diameter SW MF membranes (models V0.1-2B-3838 and V0.2B-3838, Synder Filtration, Vacaville, CA) with surface areas of  $6.69$  and  $7.06\text{ m}^2$ , respectively, were mounted horizontally. The SW membrane elements had feed spacers of  $0.79\text{ mm}$  (31 mils), diameter of  $9.65\text{ cm}$  (3.8 inches), and a length of  $96.52\text{ cm}$  (38 inches). The average cross-flow velocity was estimated between  $0.9$  to  $1.12\text{ m/s}$  for cross-flow rates at  $5.9\text{ m}^3/\text{h}$  in the element, as determined according to the scroll-end area method (Johnson, 2013).

### Operational Modes

**Total Recirculation Mode.** In total recirculation mode, the MF system was operated in a closed loop

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