Effect of transmembrane pressure control on energy efficiency during skim milk concentration by ultrafiltration at 10 and 50°C

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ABSTRACT

The efficiency of the ultrafiltration process during skim milk concentration was studied using both dynamic and constant (465 or 672 kPa) transmembrane pressure experiments at refrigerated temperature (10°C) and high temperature (50°C). The pilot-scale module was equipped with a 10-kDa polyethersulfone spiral-wound membrane element with a surface area of 2.04 m². Permeation flux, resistance-in-series model, mineral and protein rejection, and energy consumption were studied as a function of temperature and transmembrane pressure applied. Higher permeation flux values were systematically obtained at 50°C. Also, a significant temperature effect was found for calcium rejection, which was lower at 10°C compared with 50°C. Total hydraulic resistance and reversible fouling resistance were higher at 50°C than at 10°C. No change in protein rejection was observed, depending on the operating mode studied. Permeation flux, which was higher at 50°C, had lower pumping energy consumption compared with ultrafiltration at the colder temperature. Also, the low ultrafiltration temperature required a higher total energy consumption to reach the 3.6× retentate compared with ultrafiltration at 50°C. Overall, our study shows that the operating parameters and temperature can be optimized using an energy efficiency ratio.

Key words: ultrafiltration, energy consumption, efficiency, transmembrane pressure

INTRODUCTION

Ultrafiltration is a process for concentrating skim milk proteins to produce protein concentrates (MPC) with a range of protein content from 42 to 85% (dry basis). Milk protein concentrates are widely used to

standardize cheese and milk protein content to improve profit by increasing yield, reducing fat losses in whey, and increasing the capacity of a cheese-making plant (Rehman et al., 2003; Guinee et al., 2006). Milk protein concentrate powders have several functional benefits such as emulsification and high reconstitution solubility, in addition to their nutritional value (functional ingredient) for a wide range of food products (Kelly, 2011; Sikand et al., 2011; Luo et al., 2015). Although producing MPC is relatively simple, it is essential to control and optimize technological parameters [temperature, transmembrane pressure (TMP), diafiltration, and so on] because they affect MPC composition, physical functionality, and operating costs. Producing high-quality products at minimal cost with low environmental impact is the major goal of the dairy industry's eco-efficiency concept. Eco-efficiency can be achieved by decreasing energy consumption while maintaining high permeation flux values. Operating temperature and TMP adjustments are 2 possible approaches for maximizing permeation flux, but their effect on energy consumption by the filtration units must be characterized.

Several studies have compared the 50°C and 10°C operating temperatures usually used to produce MPC from UF skim milk in terms of UF performance or retentate composition (Pompei et al., 1973; Kapsimalis and Zall, 1981; St-Gelais et al., 1992; Liu et al., 2014; Luo et al., 2015). Concentrating skim milk at low temperature is the best option for controlling microbial growth and minimizing protein denaturation (Pompei et al., 1973). During UF at 10°C, permeation flux values decreased 4- to 5-fold compared with UF performed at 50°C (Pompei et al., 1973). Moreover, UF processing at low temperature increases water viscosity but also generates increased concentration polarization. Kapsimalis and Zall (1981) and St-Gelais et al. (1992) also studied the effects of temperature on permeation flux and microbial quality of UF skim milk retentates. Both studies showed that permeation flux values obtained with the same molecular weight cut-off (MWCO)

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membrane were improved at high temperature, but bacterial growth was also more important during UF at 50°C. Conversely, Luo et al. (2015) found better permeation flux values, less membrane fouling, and shorter treatment time to final volume concentration factor at low temperature during UF of skim milk. Recent studies also focused on the effect of UF temperature on the composition and functionality of MPC. Luo et al. (2015) found better functionality (emulsification, solubility, and heat stability) when UF was performed at 15°C, rather than at 30 or 50°C. These authors also reported a decrease in casein micelle size at high temperature (from 92.7 to 83.6 nm) at the end of the UF, disagreeing with the work of Liu et al. (2014), who observed a smaller change in the average micelle diameter during UF concentration. Luo et al. (2015) also demonstrated that a lower processing temperature removes more calcium. St-Gelais et al. (1992) showed that a low UF temperature reduced the buffering capacity of the skim milk concentrates, which increases the removal of soluble calcium and phosphorus.

The effect of TMP adjustments on UF membrane performance during skim milk concentration has been extensively studied. Grandison et al. (2000) characterized the effects of TMP on permeation flux and fouling during UF of skim milk at 50°C. Similar to other studies, these authors showed that at constant wall shear stress, the fouling layer, which forms at higher TMP, governs the flux (Cheryan and Kuo, 1984; Razavi et al., 2003; Rinaldoni et al., 2009; Huang et al., 2014). Furthermore, according to Cheryan and Kuo (1984), the TMP and the temperature directly affect energy consumption by UF systems. More mechanical energy is required when the TMP is higher (Cheryan and Kuo, 1984). Using a 10-kDa UF cassette module, Rinaldoni et al. (2009) estimated the mechanical and thermal energy contributing to the UF system at different operating pressures (TMP of 50, 100, and 150 kPa) and temperatures (20, 30, and 40°C), assuming ideal thermal insulation of the system. Their results suggested that maximum energy efficiency, in terms of thermal and mechanical energy contributed during processing, was achieved at room temperature and low pressure. Mechanical energy consumption increased with the TMP, but the thermal energy used to maintain the system's temperature was higher than the mechanical energy needed for feed pumping. They also showed that increasing the TMP increased the permeate volume. Increasing the temperature did not have the same effect.

To achieve the highest possible efficiency, losses in mechanical energy must be minimized. The energy losses of membrane systems are related to electric drives, pumps, membranes, valves, and system design. The study of technological alternatives to reduce the

use of energy, including mechanical and thermal energy, can help dairy processors to improve the eco-efficiency of their operations.

Our aim was to characterize the performance (permeate flux decline, fouling resistance) and energy consumption (in situ measurement for mechanical and theoretically calculated for thermal energy) of a 10-kDa polyethersulfone (**PES**) spiral-wound membrane during skim milk UF, as a function of TMP (2 constant TMP of 672 or 465 kPa, and one dynamic TMP ranging from 276 to 758 kPa to maintain constant flux) and temperature (10°C and 50°C).

MATERIALS AND METHODS

Milk Supply and Preparation

Whole raw milk (400 kg) purchased from a local cheese factory was warmed to 40°C with a heat exchanger and skimmed with a centrifugal cream separator (model MP1254, Westfalia, Oelde, Germany). The cream (40 kg) was discarded and the resulting raw skim milk (360 kg) was pasteurized with a HTST heat exchanger (APV, Goldsboro, NC) at 74°C for 16 s, cooled to 4°C, and stored at the same temperature until used.

Ultrafiltration Process

The pilot-scale UF system (GEA NIRO, Hudson, WI) included a stainless tank (60 L), a positive displacement feed pump (D/G-10, 576V, 5 HP Wanner International Ltd., Minneapolis, MN), 2 pressure gauges to monitor inlet and outlet pressure, and 1 membrane module. We used a PES spiral-wound UF module (Synder Filtration Inc., Vacaville, CA) with a MWCO of 10,000 Da. The module (model ST-3b-2540M) was 1.00 m long, with an internal diameter of 1.91 cm, a feed spacer thickness of 1.17 mm, and a membrane area of 2.04 m². The same module and UF membrane were used for all UF experiments.

Operating Conditions

Before skim milk UF, pure water flux was measured using tap water (10 or 50°C) at a TMP of 620 kPa. Skim milk (60 kg batch size) was ultrafiltered at 2 different temperatures (10 or 50°C) and 3 different TMP, including 2 constant TMP of 672 kPa (**T1**, maximum TMP recommended by the membrane supplier) and 465 kPa (**T2**, minimum TMP to provide separation), and one dynamic TMP ranging from 276 to 758 kPa (**T3**, TMP required to maintain a constant flux of 12.6 ± 4.00 kg/m²·h due to manual TMP adjustments by the operator). A tubular heat exchanger connected to

an ice bath was placed into the UF tank to maintain a constant filtration temperature of 10°C during UF. Before UF was performed at high temperature, the skim milk was warmed to 50°C, transferred to a stainless tank, and maintained at this temperature using a tubular heat exchanger. During UF concentration, the retentate was recycled back and the permeate was continuously removed from the system until a mean concentration factor (**CF**) of 3.6×, as determined by the weight (kg) of permeate collected, was reached for all TMP and temperature conditions. Samples of permeate and retentate were collected at the beginning (1×) and at the end (3.6×) of UF and stored at −20°C for subsequent analyses. The pH of all retentates was measured at the end of the process and was in the range of 6.5 to 6.7.

Cleaning Procedure

Before and after each UF experiment, the membrane was rinsed and the water flux was measured using the same parameters as described for the initial clean water flux measurement. Immediately after rinsing, a first cleaning step was performed at pH 10.5 with liquid alkaline membrane cleaner (0.1 mol/L of NaOH; Ultrasil 25, Ecolab Inc., Laval, QC, Canada) followed by cleaning with citric acid solution at 0.1% (vol/vol; Ultrasil 76, Ecolab Inc.). The final cleaning stage was performed with Ultrasil 25 solution. Between all steps, the system was rinsed with tap water to reach a neutral pH. At the end of the membrane cleaning procedure, the membrane water flux was always restored. Finally, the membrane was stored at 4°C in a 0.5% (vol/vol) acid solution (Ultrasil MP, Ecolab Inc.).

Determining Ultrafiltration Performance

Permeation Flux. During UF experiment, permeation flux (**J**; Equation [1]) was measured every 10 min by weighing permeate collected in a flask during 1 min until a final volume concentration ratio (**VCR**) of 3.6×. Permeation flux was expressed as a relative permeation flux (J_r) by using Equation [2].

$$J = \frac{F_p}{S}, \quad [1]$$

$$J_r = \frac{J}{\text{TMP}}, \quad [2]$$

where J is the permeation flux (kg/h·m²), F_p is the permeation flow rate (kg/h), S is the surface area (m²), J_r

is the relative permeation flux (kg/m²·h·Pa), and TMP is the transmembrane pressure (Pa).

Mean permeation flux J_{av} (kg/h·m²) was also calculated for each condition by determining the arithmetical mean value of all flux values between FCV 1 to 3.6×.

Rejection Coefficient. The rejection coefficients (σ) of the 10-kDa PES UF membrane for total nitrogen and calcium were determined using Equation [3].

$$\sigma = 1 - \frac{C_p}{C_r}, \quad [3]$$

where C_p and C_r are the protein or calcium concentration in the permeate and retentate, respectively.

Membrane Resistance. Membrane fouling was characterized using the resistance-in-series model according to Equation [4] (Purkait et al., 2004).

$$R_t = R_m + R_f = R_m + R_{irr} + R_{rev}, \quad [4]$$

where R_t is the total resistance (m^{−1}), R_m is the membrane resistance (m^{−1}), and R_f is the fouling resistance (m^{−1}), which is composed of the irreversible resistance (m^{−1}, R_{irr}) and the reversible resistance (m^{−1}, R_{rev}). Membrane resistance (R_m), as well as irreversible (R_{irr}) and reversible (R_{rev}) fouling, can be determined using Equations [5], [6], and [7], respectively (Piry et al., 2012).

$$R_m = \frac{\text{TMP}}{\mu J_w}, \quad [5]$$

$$R_{irr} = \frac{\text{TMP}}{\mu J_R} - R_m, \quad [6]$$

$$R_{rev} = \frac{\text{TMP}}{\mu J_E} - R_m - R_{irr}, \quad [7]$$

where TMP is the transmembrane pressure (Pa), μ is the permeate viscosity (Pa·s^{−1}), J_w is the water flux (m³/m²·s), J_R is the water flux (m³/m²·s) after membrane rinsing, and J_E is the permeation flux (m³/m²·s) at the end of UF (at 3.6×).

Energy Consumption. The total energy consumption (E_{Total} , kWh) during UF experiments included the pumping energy (E_p) and thermal energy (E_T), and was calculated using Equation [8] (Cheryan, 1998):

$$E_{\text{Total}} = E_p + E_T. \quad [8]$$

Total pumping energy (E_P , kWh), calculated in real-time and every minute from the beginning of UF ($1.0\times$; t_0) until final CF ($3.6\times$; t_f ; Equation [9]), was obtained from data recorded by a voltmeter (model Fluke 3000FC, Montréal, QC, Canada) and 3 current clamps (model Fluke A3100FC) directly connected to the induction motor terminals of positive displacement pump:

$$E_P = \int_{t_0}^{t_f} P \, dt = \sum_{i=1}^n P_i \, \Delta t_i. \quad [9]$$

Power (P , kW) consumed by the pump at each minute during skim milk UF was obtained by Equation [10]:

$$P = \sqrt{3} \times U \times I \times \cos(\phi), \quad [10]$$

where U is the voltage (volt), I is the current (A), and $\cos(\phi)$ (value from 0.37 to 0.55) is the power factor calculated as a function of pump load, according to the manufacturer's data.

The theoretical thermal energy (E_T ; kWh) used to obtain the required temperature (10 or 50°C) was estimated using Equation [11] (Rinaldoni et al., 2009):

$$E_T = C_m m \Delta T, \quad [11]$$

where C_m is the specific heat capacity of milk (0.92 kcal/kg°C), m is the mass of skim milk (kg), and ΔT (°C) is the difference between the initial temperature (4°C) and the processing temperature of the milk (10 or 50°C). For thermal energy calculations, we assumed the system had ideal thermal insulation and we did not account for the dissipation of mechanical energy.

The total energy consumption (E_{Total}) was expressed as the specific energy consumption (**SEC**), as proposed by Xu and Flapper (2011). In this study, the SEC is defined as final energy use ($E_P + E_T$, Equation [7]; i.e., end-use energy) divided by the production quantity of a dairy product output (kg of $3.6\times$ concentrate; Xu and Flapper, 2011). Consequently, E_{Total} was expressed in kilowatt hours per kilogram of milk retentate.

Analytical Methods

Total solids of milk samples were measured using AOAC International (2000) method 925.23. The total N and NPN were determined using the Kjeldahl method according to ISO 8968-1 (ISO-IDF, 2001a) and ISO 8968-4 (ISO-IDF, 2001b), respectively. The calcium and phosphorus content was measured by inductively coupled plasma-optical emission photometer (Optima

4300, dual view, Perkin-Elmer, Norwalk, CT) with wavelengths of 327.396 and 178.287 nm, respectively (Bazinet et al., 2000).

Statistical Analysis

All experiments were performed in duplicate. To determine the effect of operating parameters (pressure and temperature) on skim milk composition (TS, true protein, calcium, phosphorus), rejection coefficient, membrane resistance, and energy consumption, we performed a 2-way ANOVA in a complete block design, using the SAS GLIMMIX procedure (SAS version 9.3, SAS Institute Inc., Cary, NC). Protected Fisher's least significant difference tests were used to compare means ($\alpha = 0.05$) using the LSMEANS statement. To assess the effect of operating parameters on permeation flux as a function of CF, a repeated 3-way ANOVA was performed using the SAS MIXED procedure. In case of significant interaction effects, we used the SLICE option of the LSMEANS statement to help interpret the interaction. The normality and homogeneity assumptions were verified.

RESULTS

Effect of TMP and Temperature on Relative Permeation Flux

Figure 1 shows the effect of constant (T_1 , T_2) or dynamic TMP (T_3) at operating temperatures of 10°C (Figure 1A) and 50°C (Figure 1B) on relative permeation flux as a function of CF during UF of skim milk (experimental data available in Supplemental Figure S1; <http://dx.doi.org/10.3168/jds.2016-11504>). Overall, relative permeation flux values were significantly higher ($P < 0.05$) at 50°C compared with 10°C whatever the operational conditions (T_1 to T_3). Permeation flux decreased continuously whatever the filtration temperature and TMP applied, but some differences were observed between treatments and temperatures.

At 10°C, the relative permeation flux decline was similar ($P > 0.05$) for all TMP conditions. Indeed, relative permeation flux decreased from mean values of $2.5 \times 10^{-5} \pm 0.8 \times 10^{-5}$ kg/m²·h·Pa to $1.0 \times 10^{-5} \pm 0.02 \times 10^{-5}$ kg/m²·h·Pa at CF $3.6\times$. No significant differences ($P > 0.05$) were observed between curves for T_1 , T_2 , and T_3 , indicating that pressure adjustment had no effect on relative permeation flux at 10°C. Nevertheless, we noticed that a more constant relative flux was obtained with the low-pressure operation (T_2), suggesting that this condition offers some advantages for longer filtration periods.

When UF was performed at 50°C, marked differences occurred in the changes in relative permeation flux according to the TMP operating mode. Relative

permeation flux slightly decreased from 2.8×10^{-5} kg/m²·h·Pa to 1.5×10^{-5} kg/m²·h·Pa at CF 3.6× for the high-pressure mode (T1), whereas higher flux values

Figure 1. Effect of operating modes on relative permeation flux (J_r ; kg/m²·h·Pa) versus concentration factor (CF) during UF of skim milk at constant transmembrane pressure (TMP; 672 kPa, T1, and 465 kPa, T2) and dynamic TMP (T3) at 10°C and at 50°C. Error bars indicate standard deviation of duplicate determinations.

Table 1. Effect of operating mode on protein and mineral (calcium and phosphorus) content of UF skim milk retentate and permeate

Item	Skim milk	UF retentates ¹		<i>P</i> -value ²	UF permeates		<i>P</i> -value
		10°C	50°C		10°C	50°C	
TS ³ (%)	9.29 ± 0.02	16.92 ± 0.36	18.10 ± 0.21	<0.01	5.78 ± 0.25	5.93 ± 0.26	NS
True protein (%)	34.7 ± 2.85	55.01 ± 0.99	60.74 ± 0.78	<0.01	41.2 ± 1.43	40.30 ± 0.60	NS
Calcium (mM)	31.6 ± 1.56	70.09 ± 2.08	80.22 ± 1.24	<0.01	20.8 ± 0.90	13.20 ± 1.20	<0.01
Phosphorus (mM)	34.2 ± 0.85	62.34 ± 1.75	69.44 ± 1.07	<0.01	30.3 ± 1.00	24.50 ± 2.64	<0.01
σ _{Ca} ⁴		0.67 ± 0.01	0.77 ± 0.00	<0.01			

¹Mean concentration factor (CF) at 10°C = 3.5; mean CF at 50°C = 3.69.

²*P*-value compared retentate and permeate compositions at 10 and 50°C.

³Expressed on dry basis, conversion factor: N × 6.38.

⁴Rejection coefficient (σ) of calcium.

(3.9×10^{-5} kg/m²·h·Pa to 2.5×10^{-5} kg/m²·h·Pa) were obtained for the low-pressure mode (T2). A different trend was observed for constant flux operating mode (T3) where flux sharply decreased from 8.8×10^{-5} kg/m²·h·Pa to 1.8×10^{-5} kg/m²·h·Pa as the CF increased to 2.4×, with a specific decrease in flux of 58% from 1.0 to 1.75×. From 2.4 to 3.6× CF, the relative permeation flux remained constant. Because of the sharp decrease in relative permeation flux under T3 and the lower flux values under T1, we estimate that T2 operating mode offers advantages in performance because relative permeation flux remained constant from 1.0 to 3.6×, was higher than T1 during all UF steps and T3 from 1.5× until the end of filtration.

Effect of TMP and Temperature on Retentate Composition and Milk Component Rejection

Table 1 reports the final retentate composition in terms of TS, true proteins, calcium, and phosphorus content. Because TMP operating modes (T1, T2, and T3) had no significant effect on the composition of UF retentates at each temperature, the data were pooled in 2 groups (10 and 50°C). Significant temperature effects ($P < 0.01$) were observed for all components analyzed in retentates. The TS, true proteins, calcium, and phosphorus values were all higher in retentates prepared at

50°C. The rejection coefficient (σ) for calcium, but not nitrogen, was significantly higher ($P < 0.01$) at 50°C compared with 10°C, whatever the TMP operating mode (T1, T2, or T3).

Effect of TMP and Temperature on Membrane and Fouling Resistances

Table 2 reports the membrane resistance (R_m) as well as reversible (R_r) and irreversible fouling resistances (R_{irr}) for each TMP operating mode (T1, T2, and T3) at 10 and 50°C. Membrane resistance (R_m) and irreversible resistance (R_{irr}) were strongly influenced ($P < 0.0001$) by temperature, whereas TMP operating mode had no significant effect. Membrane resistance was higher at 50°C than at 10°C ($11.12 \pm 0.08 \times 10^{12}$ m⁻¹ vs. $6.57 \pm 0.29 \times 10^{12}$ m⁻¹) but R_{irr} was lower ($0.88 \pm 0.01 \times 10^{12}$ m⁻¹ vs. $1.24 \pm 0.03 \times 10^{12}$ m⁻¹). Reversible resistance (R_{rev}) increased significantly ($P < 0.01$) at 50°C compared with 10°C ($3.31 \pm 0.25 \times 10^{12}$ m⁻¹ vs. $2.02 \pm 0.29 \times 10^{12}$ m⁻¹). Interestingly, the low-pressure operating mode (T2) induced lower R_{rev} values ($P < 0.05$) compared with the 2 other modes at both temperatures.

Overall, total resistance values (R_t) were higher at 50°C ($P < 0.01$) and the T2 mode induced lower (R_t) values ($P < 0.01$) compared with the 2 other modes at

Table 2. Effect of operating mode on membrane resistance (R_m), irreversible resistance (R_{irr}), reversible resistance (R_{rev}), and total resistance (R_t ; mean ± SE) for producing a 3.6× skim milk retentates by UF

Resistance (m ⁻¹)	Temperature			Operating mode			
	10°C	50°C	<i>P</i> -value	T1 ¹	T2 ¹	T3 ¹	<i>P</i> -value
R_m (× 10 ¹²)	6.57 ± 0.29	11.12 ± 0.08	***	8.65 ± 1.34	9.01 ± 1.25	8.89 ± 1.42	NS
R_{irr} (× 10 ¹⁴)	1.24 ± 0.03	0.88 ± 0.01	***	1.09 ± 0.13	1.04 ± 0.10	1.05 ± 0.09	NS
R_{rev} (× 10 ¹⁴)	2.02 ± 0.29	3.31 ± 0.25	**	2.81 ± 0.35 ^a	1.96 ± 0.45 ^b	3.23 ± 0.43 ^a	*
R_t (× 10 ¹⁴)	3.33 ± 0.30	4.30 ± 0.26	**	3.98 ± 0.23 ^a	3.09 ± 0.39 ^b	4.37 ± 0.36 ^a	**

^{a,b}Different superscripts indicate significant differences between applied pressures.

¹T1 = 672 kPa; T2 = 465 kPa; T3 = dynamic flux.

* $P < 0.05$; ** $P < 0.01$; *** $P < 0.0001$.

any temperature. The constant flux mode (T3) induced the highest R_{rev} values for all conditions.

Effect of TMP and Temperature on Energy Consumption

Figure 2 illustrates the requirements in mechanical (E_p) and thermal (E_T) energy (kWh/kg of retentate) for the production of CF 3.6 $\times$ retentate under each TMP operating mode at 10 and 50°C. The TMP operating mode had no significant effect on E_T or E_p values at both temperatures. However, UF temperature resulted in highly significant differences ($P < 0.0001$) for both E_T and E_p . Indeed, E_p was 2.3 times higher at 10°C compared with 50°C with values of 0.28 ± 0.02 and 0.12 ± 0.00 kWh/kg, respectively. Conversely, the thermal energy needed to maintain milk at 50°C was 4.5 times higher compared with 10°C, with values of 0.16 ± 0.01 and 0.035 ± 0.001 kWh/kg, respectively. When changed, only temperature significantly affected ($P < 0.01$) the total energy requirement ($E_T + E_p$), and this was higher at 10°C (0.32 ± 0.02 kWh/kg) than at 50°C (0.28 ± 0.01 kWh/kg).

DISCUSSION

Our results provide evidence that temperature adjustments (10 vs. 50°C) had more significant effects on flux and hydraulic resistance compared with the effects of TMP operating regimens (T1, T2, and T3). The effect of TMP operating regimens has been limited to some flux decline characteristics at the beginning of UF experiments (e.g., under T3 at 50°C).

We systematically obtained higher relative permeation flux values at 50°C. Other studies also reported a significant reduction in permeation flux at low temperature (Pompei et al., 1973; Kapsimalis and Zall, 1981; St-Gelais et al., 1992; Luo et al., 2015).

This effect can result from increased water viscosity as the UF temperature decreases to 10°C. As temperature affects filtration fluid viscosity, according to Equation [1], flux (J) is inversely related to permeate viscosity (μ). Our results also show that at low temperature (10°C), some calcium and phosphate were solubilized in the serum phase because decreases in those mineral contents were observed in the retentate at 10°C compared with 50°C at CF 3.6 $\times$.

Temperature-dependent changes in permeation flux during UF concentration of milk may also be related to changes in soluble-colloidal equilibrium, which, in turn, may affect protein deposition on the UF membrane. Soluble-colloidal equilibrium between casein micelles and milk serum is profoundly affected by temperature (Horne, 2006). Luo et al. (2015) found that casein mi-

Figure 2. Total energy (E_{Total} , kWh/kg of retentate) used as a function of operating conditions (dynamic and constant transmembrane pressure, 10°C and 50°C) for the production of 1 kg at volume concentration ratio of 3.6 $\times$, including the thermal (E_T) and pumping energy (E_p ; T1 = 672 kPa; T2 = 465 kPa; T3 = dynamic flux). Different letters indicate significant differences for E_T and E_p as a function of operational conditions. Error bars indicate standard deviation of duplicate determinations.

celles obtained from UF retentates prepared at 15°C had lower zeta potential compared with those obtained by UF at 50°C.

We also observed that permeation flux decline profiles were similar for all TMP operating modes (T1, T2, T3) at 10°C but not at 50°C. These results agree with recent observations by Luo et al. (2015) who found that the rate of flux decline was more rapid during the initial operation for the UF at 50°C compared with UF at 15°C. These authors related this observation to increased deposition of calcium and protein on the membrane surface at 50°C. The distinctive flux decline observed during the T3 operating mode at 50°C between CF 1.0 to 2.4 $\times$ can be explained by the rapid concentration polarization of solutes at the membrane surface, resulting from the convective flow at high flux values. At CF >2.4 $\times$, the concentration polarization layer is more compact and its permeability gradually decreases.

The retentate compositions agree with published reports that lower temperatures decrease the proportion of colloidal calcium and phosphorus after UF of skim milk with membranes with MWCO ranging from 10 to 50 kDa (Brule et al., 1974; Kapsimalis and Zall, 1981; Luo et al., 2015). Also, studies by Miller et al. (2014) revealed that over the threshold flux, at constant TMP and constant flux, rejection of organic particles increases as the TMP increases because the dynamic cake layer formed acts as another barrier to the particles (Miller et al., 2014). In contrast, we did not observe significant differences between calcium and nitrogen rejection at either constant or dynamic TMP. This can be explained by the fact that Miller et al. (2014) used UF membranes with MWCO of 20 kDa or because we

observed similar fouling at constant and dynamic flux because all the true proteins were retained with the 10-kDa MWCO membranes.

We found that temperature had a strong and significant effect ($P < 0.0001$) on membrane resistance (R_m), which was higher at 50°C. Stade et al. (2015) showed that PES and regenerated cellulose with MWCO of 20 and 30 kDa, respectively, were subjected to compaction at 50°C and R_m increased with temperature due to changes in the viscoelastic properties of the polymeric layer (Stade et al., 2015). These findings suggest that membrane compaction increases the total hydrodynamic resistance, which negatively affects permeability. Increasing the temperature to 50°C decreased R_{irr} ($P < 0.0001$) but increased R_{rev} ($P < 0.001$). Also, at both UF temperatures, the contribution of R_{rev} was more important than that of R_{irr} . These observations agree with those of Grandison et al. (2000), who demonstrated that the contribution of irreversible fouling to total hydraulic resistance was low compared with reversible fouling when skim milk was ultrafiltered at 50°C with a 200-kDa tubular membrane (Grandison et al., 2000). The same conclusion was reached by Muthukumaran et al. (2007) and Macedo et al. (2015), who used different 10-kDa UF membrane materials to concentrate cheese whey. Our data contrasted, however, with the conclusions of Luo et al. (2015), who showed that more fouling occurred at high temperature during UF of skim milk. We must emphasize that none of these published data were obtained using spiral-wound elements, especially for Luo et al. (2015), who used flat-sheet membrane coupons. Such a laboratory-scale membrane system generates different hydrodynamic conditions and directly affects results on propensity for membrane fouling.

The hydraulic resistance values obtained under the TMP operating regimens were not statistically different, except for R_{rev} which was lower ($P < 0.05$) for T2, compared with T1 and T3. Some studies have demonstrated that at high TMP, a more compact and compressed deposit layer structure occurs, which is responsible for the increase in reversible fouling (Gézan et al., 1994; Samuelsson et al., 1997; Carić et al., 2000). Also, Miller et al. (2014) reported that constant permeation flux operation caused the continuous accumulation of fouling on the membrane (i.e., increasing total hydraulic resistance), compared with constant TMP where the fouling deposit remained constant. We were unable to confirm these hypotheses in this study.

Our energy consumption data provides strong evidence for the effect of UF temperature. First, the thermal energy consumption required to produce 1 kg of 3.6× retentate was lower at 10°C than at 50°C, agreeing

with Rinaldoni et al. (2009). However, we must mention that the energy required to maintain skim milk at 10 or 50°C, the dissipation of mechanical work into heat by the feed pump and the energy loss during UF were not included in the calculation. Consequently, we likely underestimated the thermal energy consumption. Second, producing a 3.6× milk retentate at 10°C required 2.3 times more pumping energy (0.28 ± 0.02 kWh/kg) than operating at 50°C (0.12 ± 0.002 kWh/kg), whatever the TMP operating mode. This difference could be explained by the higher fluid viscosity value at lower temperatures compared with 50°C. Third, the mechanical energy data obtained for UF of skim milk at 50°C agree with the results of Xu and Flapper (2011), who reported a reference SEC value, based on global dairy plants, of 0.11 kWh of electricity and 0.36 kWh of fuel to produce 1 kg of concentrated milk (Xu and Flapper, 2011).

Using the total energy consumption as an indicator helps assess the effect of different TMP regimens and temperatures on the efficiency of the UF concentration process. Figure 3 illustrates the relationship between mean permeation flux ($\text{kg}/\text{m}^2\cdot\text{h}$) and total energy consumption (kWh/kg of retentate) for the different TMP (T1, T2, T3) and temperatures (10 and 50°C). The graph has been divided into 4 quadrants to make data interpretation easier. The most efficient conditions in terms of flux and energy consumption is when flux is maximal and energy consumption is minimal. Figure 3 shows that optimal energy efficiency is achieved when the UF is operated at high temperature compared with low temperature. We also observed improved energy efficiency when the TMP was low (i.e., T2 operating mode; 465 kPa at 50 and 10°C), compared with the high and dynamic TMP. However, the high (T1) TMP operating mode has an energy efficiency ratio between the dynamic (T3) and the low (T2) TMP at 10 and 50°C. Overall, the optimal energy efficient operating mode will be at a low constant TMP at 50°C.

CONCLUSIONS

Permeation flux values were increased at 50°C compared with 10°C, whatever the TMP. To save energy, processors should consider ultrafiltering skim milk at 50°C, which requires 2.3 times less pumping energy compared with ultrafiltering at 10°C, even if more thermal energy must be applied. When scaling up the process to higher working volumes, processors should calculate the energy efficiency ratio because operating the UF at low constant TMP is more efficient. This study will help dairy processors to determine the optimal parameters needed to improve UF energy efficiency.

Figure 3. Efficiency ratio as illustrated by the relationship between mean permeation flux (J_{av} ; kg/m²·h) at volume concentration ratio of 3.6× and total energy consumption (E_{Total} ; kWh/kg of retentate) for dynamic and constant transmembrane pressure (T1 = 672 kPa; T2 = 465 kPa; T3 = dynamic flux) at 10°C and 50°C.

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