

Concentration of Milk and Whey by Membrane Technologies in Alternative Cascade Modes

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Abstract The aim of the present study was the evaluation of a membrane cascade comprised of ultrafiltration (UF) in series with reverse osmosis (RO) or nanofiltration (NF) in comparison to a single-stage process. It was found that the upstream UF accelerated the NF and the RO, whereby the effect was more distinct for the NF. The maximum volume reduction ratio (VRR) during skim milk and sweet whey concentration could be increased by 78 and 96%, respectively, by substituting a single NF by an UF-NF cascade. The replacement of a single RO by a UF-RO cascade during concentration of skim milk slightly increased the VRR by 3%. However, the energy demand could be reduced by approximately 16%. For the concentration of sweet whey, it was found that it is more advantageous to conduct the RO at a higher transmembrane pressure (TMP) instead of applying an UF-RO cascade.

Keywords Skim milk · Sweet whey · Ultrafiltration · Reverse osmosis · Nanofiltration · Membrane cascade

Introduction

The reduction and assessment of the energy demand is an important point in many fields within food processing (see, e.g., Toepfl et al. 2006; Hang et al. 2015; Wojdalski et al. 2013; Peters 2005; Calín-Sánchez et al. 2014). During powder

production from dairy fluids, spray drying is the unit operation with the highest energy consumption (Ramírez et al. 2006). Since the dry matter of those dairy fluids is comparably low (6–9%), a lot of water needs to be removed. In order to save energy during the energy intensive drying stage, the fluids are often pre-concentrated. Evaporation is usually applied as a pre-treatment step due to its better heat transfer capacity in comparison to spray drying. However, it is still a thermal process and the water needs to be converted into the gaseous state for removal. Hence, non-thermal membrane processes, like reverse osmosis (RO), are even more advantageous in terms of energy efficiency as the water can be eliminated without phase conversion. Therefore, there is great interest in a maximum increase of the dry matter by means of RO. However, concentration polarization, the rising osmotic pressure during concentration, and deposit formation of the protein fraction limit the maximum volume reduction ratio (VRR) as well as the permeate flux during RO (see Fig. 1a). Our experimental approach to enhance permeate flux and VRR is a cascade of several membrane systems, as presented in Fig. 1b.

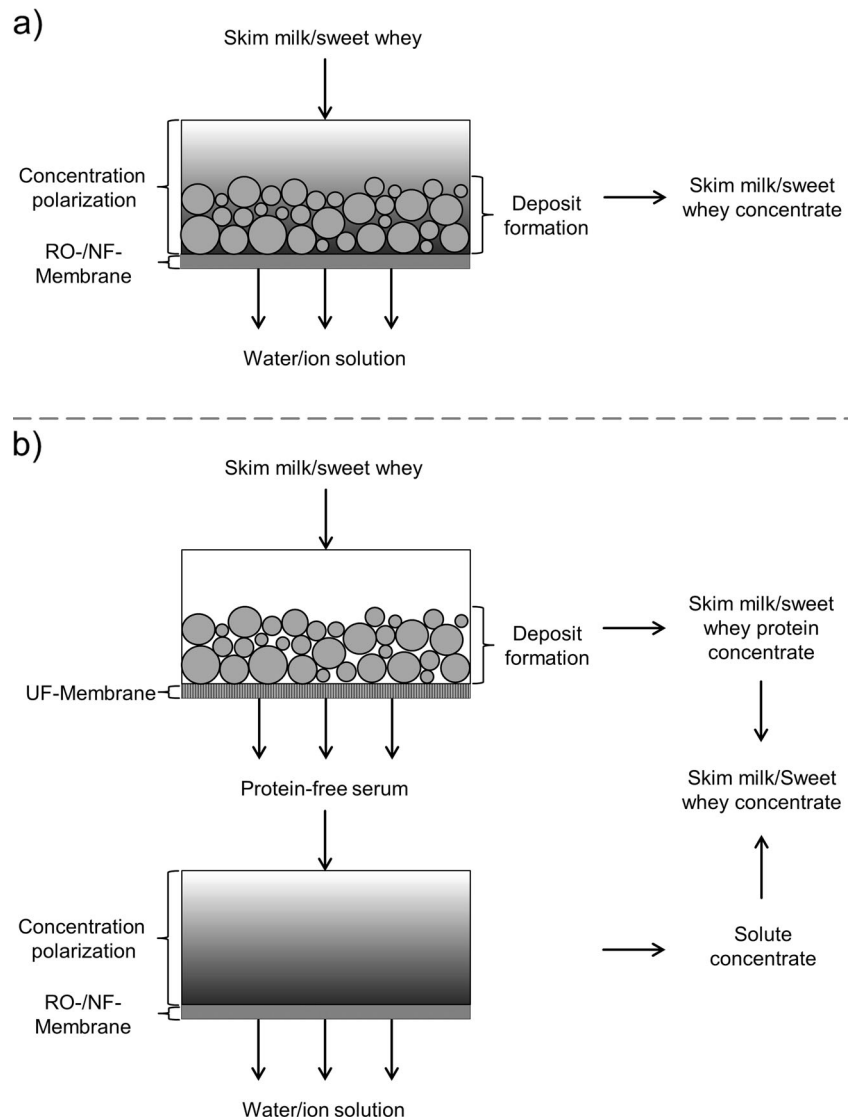
First, the protein fraction is concentrated via ultrafiltration (UF) without being limited by the osmotic pressure. The problem of formation of a deposited layer is shifted to a lower transmembrane pressure (TMP) level, and energy losses through the deposit are thus drastically reduced. Cross-flow ultrafiltration is already applied in the dairy industry for protein concentration. Especially, spiral wound modules (SWM) are frequently used due to their low investment and replacement cost. A major limitation of SWMs is the generation of large pressure drops within the module when processing highly viscous fluids (Lipnizki et al. 2005). Thus, the application of conventional UF modules is limited, as particularly milk protein concentrates show a high viscosity (Solanki and Rizvi 2001). An alternative is

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Fig. 1 a Concentration polarization and deposit formation during reverse osmosis (RO) and nanofiltration (NF) of skim milk and sweet whey. **b** Membrane cascade comprised of ultrafiltration (UF) and RO/NF for the concentration of skim milk and sweet whey



the application of dynamic filtration devices with rotating membranes. In contrast to classical cross-flow filtration systems, the wall shear stress to avoid deposit formation is created through the movement of the membrane itself instead of a high feed volume throughput. Hence, dynamic devices can be operated at much lower feed volume throughputs (Jaffrin 2008), whereby the energy demand can be reduced due to lower pump capacities. Furthermore, the low feed volume throughput facilitates the processing of highly viscous fluids. In a preceding study, the authors could demonstrate that a membrane cascade of a SWM with a dynamic filtration device allows the concentration up to a protein content of approximately 30% (*w/w*) during the UF of skim milk (Meyer et al. 2015). An alternative to SWMs is the utilization of tubular ceramic membranes, which are characterized by more advantageous flow conditions within the module (Goulas and Grandison 2008).

The permeate of the UF contains the low molecular weight substances of milk (e.g., lactose and salts) and can be enriched in a second step via RO. The previous protein removal allows the operation of the RO without being negatively affected by deposit formation of the protein fraction. Therefore, the TMP can be used more efficiently to overcome the rising osmotic pressure during concentration. Besides RO, nanofiltration (NF) is also already applied for the filtration of dairy fluids (see e.g., Román et al. 2011; Rice et al. 2011; Altmann et al. 2016; Cuartas 2004). NF membranes allow the permeation of monovalent ions due to their looser membrane structure and are therefore less limited by the increasing osmotic pressure. During concentration, this may result in higher VRR at a similar TMP or the same VRR can be reached at a lower TMP in comparison to RO. In addition, the application of NF could result in an improvement of the milk powder quality in

terms of heat stability (Syrios et al. 2011) or solubility (Cao et al. 2015) due to the depletion of the monovalent ions.

Finally, the obtained retentates are recombined to obtain a skim milk or sweet whey concentrate that contains all components or is depleted in monovalent ions when NF was applied. It is assumed that such a membrane cascade does not only enhance the maximum VRR and the permeate flux during the RO and NF stage, respectively, but also lowers the energy demand of the pre-concentration step.

Such cascades have already successfully been applied in other research fields like wastewater treatment (Hang et al. 2015) or desalination (Cardona et al. 2005). It was shown that an upstream UF helped to reduce wastewater pollution or cut production costs, respectively. In the dairy sector, only few studies deal with the assessment of data related to the energy demand of UF and RO (Bahnasawy and Shenana 2010; Cheryan and Kuo 1984; Stabile and Roger 1985). In addition, these calculations are often based on small pilot installations and estimations. As far as the authors are aware, no collection and assessment of values for the energy demand of large-scale plants have been done yet.

UF membranes coupled with RO or NF modules are already in use during processing of skim milk and sweet whey (Atra et al. 2005; Butylina et al. 2006; Di Giacomo et al. 1996; Hiddink et al. 1980; Rektor and Vatai 2004; Suárez et al. 2006; Suárez et al. 2009; Meyer and Kulozik 2016). The positive effect of an upstream UF on RO of skim milk and sweet whey was already shown in a previous study (Meyer and Kulozik 2016). The impact of a protein removal on NF of skim milk has not been investigated yet, and the results regarding sweet whey are partly contradictory (Rektor and Vatai 2004; Suárez et al. 2009). Furthermore, the studies of other research groups focus on the separate use of the retentates and provide no information on the maximum VRR after a recombination of the retentates.

This paper aims at evaluating the cascade of different UF modules in combination with RO and NF membranes for the high enrichment of skim milk and sweet whey. Therefore, different ultrafiltration modules (SWM, dynamic device, and tubular ceramic membrane) were compared in terms of their suitability to concentrate the protein fraction. Moreover, it was investigated whether the findings concerning the impact of an upstream protein removal on the RO of skim milk and sweet whey can be transferred to the NF of these fluids. Furthermore, it was evaluated to what extent the maximum VRR can be increased when applying a membrane cascade compared to a single RO or NF. In addition, data about the energy demand of industry-scale membrane units were assessed and used to evaluate the membrane cascade in terms of energy efficiency. Finally, possible connections of the single membrane modules in series were derived.

Material and Methods

Pilot Plant Units and Experimental Procedure

The pilot plant system used for the UF experiments has already been described in detail elsewhere (Kühnl et al. 2010; Piry et al. 2008). The filtration fluid was pumped via a centrifugal pump (Grundfos GmbH, Erkrath, Germany) into the filtration module. The TMP was adjusted by a throttle valve at the membrane outlet. A tubular heat exchanger was used to maintain a constant filtration temperature. Pressure transmitters (WIKA SE & Co. KG, Klingenberg, Germany) and flowmeters (Endress+Hauser Messtechnik GmbH & Co. KG, München, Germany) were used to measure the TMP and the retentate and permeate flow rates. A conditioning step was carried out prior to each experiment. Therefore, a 0.5% Ultrasil 14 solution (Ecolab, Monheim am Rhein, Germany) was circulated in the filtration rig at a temperature of 55 °C for 20 min. Afterwards, the membrane was rinsed with demineralized water and the pure water flux of the membrane was determined. The water flux was used to calculate the membrane resistance according to Darcy's law:

$$J = \frac{\text{TMP}}{\eta \cdot R_T} = \frac{\text{TMP}}{\eta \cdot (R_M + R_F)} \quad (1)$$

where J is the permeate flux in $\text{L m}^{-2} \text{h}^{-1}$, η is the dynamic permeate viscosity in Pa s, R_T is the total and R_M and R_F are the membrane and fouling resistances in m^{-1} , respectively. The membrane resistance is the hydraulic resistance of the membrane; the fouling resistance is the additional flow resistance due to membrane fouling; and the total membrane resistance describes the overall hydraulic resistance of the membrane and the fouling layer. The mean value of the membrane resistance was $1.57 \times 10^{12} (\pm 9.83 \times 10^{10}) \text{ m}^{-1}$. Filtrations in the dairy sector are usually carried out at temperatures around 10 or 50–55 °C to ensure microbiological stability of the product. To benefit from the flux-enhancing effect of a lower viscosity (see Eq. (1)), in the industry, the filtration of milk is commonly conducted at a temperature of 50–55 °C. In order to reduce whey protein fouling, which is more pronounced at elevated temperatures, the filtration of whey is usually carried out at 10 °C. In order to provide data with a practical meaning, 50 and 10 °C were chosen for the filtration of skim milk and sweet whey, respectively. Hence, before filtration, the sweet whey and the sweet whey concentrates were brought to a temperature of 10 °C and the pH was adjusted to pH 6.5. The skim milk and the skim milk concentrates were heated to a temperature of 50 °C prior to adjusting the pH to 6.8. Then, 25 L of the feed solution were filled into the thermostated feed tank and the filtration experiment was performed ($t = 90 \text{ min}$). The retentate and permeate were completely recycled to the feed tank. All filtrations were conducted at a

wall shear stress τ_w of 100 Pa. The wall shear stress in Pa can be calculated from the pressure drop along the membrane (Δp_L) in Pa, the channel diameter (d) in m, and the membrane length (L) in m according to

$$\tau_w = \frac{\Delta p_L \times d}{4 \times L} \quad (2)$$

The impact of the TMP on the UF of sweet whey and skim milk was examined by varying the TMP from 50 to 400 and 500 kPa, respectively. The experiments were carried out in duplicate. The mean transmembrane pressure was calculated according to

$$\text{TMP} = \frac{p_1 + p_2}{2} \quad (3)$$

where p_1 is the pressure at the module inlet and p_2 is the pressure at the module outlet, both in Pa. In order to evaluate the effect of the protein concentration, the sweet whey and skim milk protein concentrates were filtrated at a TMP of 300 kPa.

For the NF experiments, a piston diaphragm pump (Hydra-Cell G10-X, Verder Deutschland GmbH, Haan, Germany) was used to pump the filtration fluid into the filtration module. A throttle valve at the module outlet was used to adjust the TMP. The filtration temperature was kept at a constant value by a tubular heat exchanger. The pressure at the membrane inlet and outlet was measured by pressure transducers (Schneider Electric GmbH, Ratingen, Germany). Magnetic-inductive flowmeters were used to measure the permeate (Endress+Hauser Messtechnik GmbH & Co. KG, München, Germany) and retentate flow rates (Danfoss GmbH, Offenbach am Main, Germany). At the beginning of each experiment, the membrane was conditioned by circulating a 0.1% Ultrasil 69 solution (Ecolab, Monheim am Rhein, Germany) for 20 min at a temperature of 45 °C in the filtration unit. Then, the pure water flux was determined after flushing the system with demineralized water. The feed solutions ($V = 100$ L) were heated to the filtration temperature of 10 °C, and the pH was adjusted to 6.8 before filling them into the feed container. All experiments were carried out in concentration mode at a feed flow rate of 1100 L h⁻¹. Thus, the retentate was completely recycled to the feed tank, whereas the permeate was collected in a separate container. The impact of the TMP on the concentration of skim milk, sweet whey, and protein-free serum was investigated by performing experiments at 2 and 4 MPa. The filtration was stopped when the flux dropped below 2 L m⁻² h⁻¹ or the pressure drop along the membrane was higher than 300 kPa.

Test Fluids

The pasteurized skim milk and sweet whey were purchased from a local dairy. The average composition of the skim milk

was as follows: total protein content 3.47% (± 0.47) (w/w), dry matter (DM) 9.65% (± 0.25), lactose 44.2 g L⁻¹ (± 5.7), calcium 1.1 g L⁻¹ (± 0.4), and sodium 453.7 mg L⁻¹ (± 18.7). The skim milk protein concentrates with protein contents ranging from 6.6 to 14.7% (w/w) were prepared by suspending skim milk protein powder (86.6% (w/w) protein in the DM (Privatmolkerei Naarmann GmbH, Neuenkirchen, Germany) in skim milk at a temperature of 50 °C for 2 h under constant stirring. Afterwards, the protein concentrates were stored overnight at 4 °C to ensure a complete rehydration. In order to perform a curd removal, the sweet whey was clarified at a centrifugation factor of approximately 8000×g using a disk separator (GEA Westfalia Separator AG, Oelde, Germany). The average composition of the clarified sweet whey was as follows: total protein content 0.71% (± 0.07) (w/w), DM 6.3% (± 0.5), lactose 42.0 g L⁻¹ (± 6.2), calcium 404.5 mg L⁻¹ (± 32.0), and sodium 440.2 mg L⁻¹ (± 48.5). To prepare whey protein concentrates with protein contents from 2.9 to 16.9% (w/w), whey protein isolate powder (WPI 895, Fonterra, Auckland City, New Zealand) was suspended in clarified sweet whey at a temperature of 4 °C for at least 12 h in an agitated container. The protein-free serum (UF permeate) was made from pasteurized skim milk using a spiral wound module (GR81PE 6338/30, Alfa Laval, Glinde, Germany) with a nominal cutoff of 10 kDa. The filtration was conducted at a temperature of 50 °C, a TMP of 200 kPa, and a feed flow rate of 15,000 L h⁻¹. The average composition of the ultrafiltration permeate was as follows: DM 6.4% (± 0.46), lactose 53.9 g L⁻¹ (± 3.7), calcium 347 mg L⁻¹ (± 28.7), and sodium 465 mg L⁻¹ (± 27.5). The protein concentrates were prepared 1 day before use. The skim milk, the sweet whey, and the protein-free serum were stored at a maximum 4 days at 4 °C before use.

Membranes and Cleaning Procedure

The details about the membranes used are listed in Table 1. The membranes were cleaned after each experiment following a fixed protocol. First, the filtration system was flushed with demineralized water until the rising water came out clear. Then, a two-step cleaning procedure was carried out. The ceramic membrane was cleaned circulating an alkaline 1% Ultrasil 14 cleaning solution (Ecolab, Monheim am Rhein, Germany) for 40 min at a temperature of 65 °C. The subsequent rinsing with demineralized water was followed by an acid cleaning. Therefore, a 0.5% nitric acid solution (BHM Chemikalienhandel GmbH, Ostrach, Germany) was recycled in the filtration rig for 20 min at 50 °C. For the alkaline cleaning of the SWM, a combination of 0.3% enzymatic (Ultrasil 67, Ecolab, Monheim am Rhein, Germany) and 0.2% alkaline (Ultrasil 69, Ecolab, Monheim am Rhein, Germany) cleaning agents was used (30 min at 45 °C). The system was flushed with demineralized water, and the acid

Table 1 Membrane type and properties

Membrane	Type	Manufacturer	Molecular weight cutoff [Da]	Area [m ²]
UF	Ceramic tubular multichannel membrane, Type (7/6), ZrO ₂	Atech (Gladbeck, Germany)	20,000	0.16
NF	Spiral wound module; DK 2540F1072/30	GE Power & Water (Fairfield, USA)	150–300	2.6

cleaning was carried out (0.2% Ultrasil 75 solution (Ecolab), 30 min, 45 °C). After the acid cleaning, the filtration units were rinsed with demineralized water until a pH of 7 was reached.

Retentate and Permeate Analysis

The DM of the samples was measured by determining the difference between the weight of the wet and dried sample using a SMART Turbo (CEM GmbH, Kamp-Lintfort, Germany). The total nitrogen content was determined by the combustion method of Dumas (Wiles et al. 1998) using a nitrogen analyzing system vario MAX cube (Elementar Analysensysteme GmbH, Hanau, Germany). The total protein content of the samples was calculated from the total nitrogen content by multiplying with a factor of 6.38. The concentration of the major whey protein β -Lactoglobulin, representing about 80% of the whole whey proteins, was analyzed by RP-HPLC according to the method described by Toro-Sierra et al. (2013). The lactose content was measured via HPLC according to a method described by Schmitz-Schug (2014). The content of sodium and calcium was quantified by means of a flame photometer (ELEX 6361, Eppendorf AG, Hamburg, Germany). The measurement was conducted at a wavelength of 589 nm for sodium and 622 nm for calcium. The burnable gas was an acetylene-air mixture. A one-point calibration with a standard containing sodium and calcium, each at a concentration of 150 mg L⁻¹, enabled the quantification of the cations. The rejection coefficient RC of the components in percent was calculated according to

$$RC = \left(1 - \frac{c_{Per}}{c_{Ret}}\right) \times 100\% \quad (4)$$

where c_{Per} and c_{Ret} are the concentrations of the components in the permeate and retentate in g L⁻¹, respectively.

The volume reduction ratio (VRR) at a certain time i was calculated according to

$$VRR_i = \frac{DM_{Ret,i}}{DM_{Ret,0}} \quad (5)$$

where $DM_{Ret,i}$ is the dry matter of the retentate at a certain time i and $DM_{Ret,0}$ is the dry matter of the retentate at the beginning of the concentration in percent.

Energy Calculations

Three different companies provided data about the installed capacities of the feed and recirculation pumps, the permeate volume, and the dry matter of the obtained product for 12 different UF and RO installations in the dairy industry. In order to enable a meaningful comparison of the different membrane systems, the specific energy demand was calculated. Taking into account that the actual energy demand of the pumps is approximately 80% of the installed capacity, the specific energy demand of the membrane process E_m in kJ kg⁻¹ was calculated from the installed capacities according to

$$E_m = \frac{0.8 \times (E_F + E_R)}{m_{Per}} \quad (6)$$

where E_F and E_R are the installed capacity of the feed and recirculation pumps in kJ, respectively, and m_{Per} is the permeate mass in kg.

The specific primary energy demand $E_{m,P}$ in kJ kg⁻¹, which takes into account the efficiency of the power generation ($\eta_P = 0.33$) (Koros et al. 2009), was calculated according to

$$E_{m,P} = \frac{E_m}{\eta_P} \quad (7)$$

Results and Discussion

Concentration of the Protein Fraction

The results of the filtrations with skim milk and sweet whey at different TMP and $\tau_w = 100$ Pa are represented in Fig. 2. The steady state flux at the end of the filtration is plotted against the TMP. It can be seen from the deviation of the curves from the water flux that deposit formation already took place at the lowest applied TMP = 50 kPa. Furthermore, a linear relationship between permeate flux and TMP could be observed at low TMP. Hence, according to Darcy's law (Eq. (1)), the total filtration resistance remained constant in this pressure range and an increase in TMP was directly converted into an increasing flux. At higher TMP, the curves leveled off. This indicates

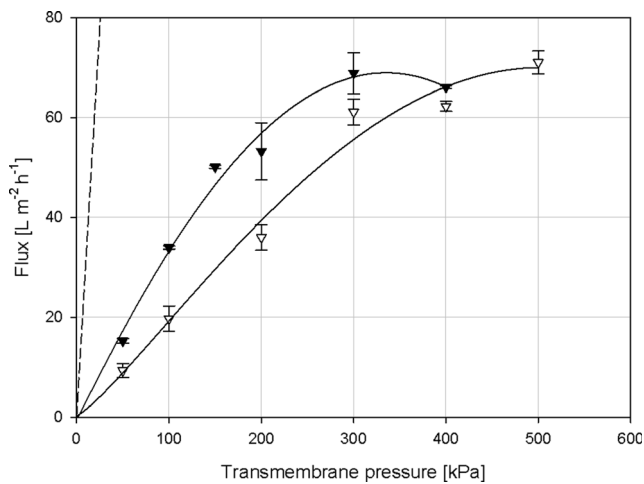


Fig. 2 Permeate flux of skim milk (*inverted empty triangles*, $c_p = 3.47\%$ (w/w), filtration temperature $50\text{ }^\circ\text{C}$) and sweet whey (*inverted filled triangles*, $c_p = 0.71\%$ (w/w), filtration temperature $10\text{ }^\circ\text{C}$) as a function of transmembrane pressure at a wall shear stress of $\tau_w = 100\text{ Pa}$. The pure water flux (*dashed line*) is also plotted as a reference. *Error bars* represent standard deviation of mean data from duplicate experiments

that the threshold flux was reached (Field and Pearce 2011). The deviation from the linear relationship can be explained by a partial compensation of the pressure increase by a more intense deposit formation, or rearrangement of deposited particles, that induces a higher pressure loss across the deposit layer (Kulozik and Kessler 1988). A further pressure increase can lead to a complete compensation, and the pressure-independent, limiting flux is reached (Porter 1972). From an economical point of view, filtrations should be conducted at or below the threshold flux. Hence, the filtration of sweet whey and skim milk should not be conducted above a TMP of 300 kPa. The impact of increasing concentration was investigated by conducting filtrations with several skim milk and sweet whey protein concentrates. The concentration process was simulated by plotting the steady state flux values as a function of the protein concentration. According to the results from Fig. 2, the experiments were conducted at TMP = 300 kPa. Figure 3 shows the findings of the filtration of skim milk concentrates with the tubular ceramic membrane (TMP = 300 kPa; $\tau_w = 100\text{ Pa}$) in relation to the results from concentration experiments using a SWM (TMP = 200 kPa) and a dynamic system (TMP = 100 kPa; $N = 1000\text{ min}^{-1}$) at $50\text{ }^\circ\text{C}$ (data for the SWM and dynamic system are taken from Meyer et al. 2015). The different operating conditions result from the fact that for a meaningful comparison of the various filtration systems, each system was run at its optimal operation point. The concentration with the tubular ceramic membrane could only be simulated up to a protein content of approximately 15% (w/w). At higher protein concentrations, a strong increase in retentate viscosity occurs (Meyer et al. 2015). Such high viscosities could not be processed by the pump used. However, it can be estimated from the slope of the curve that

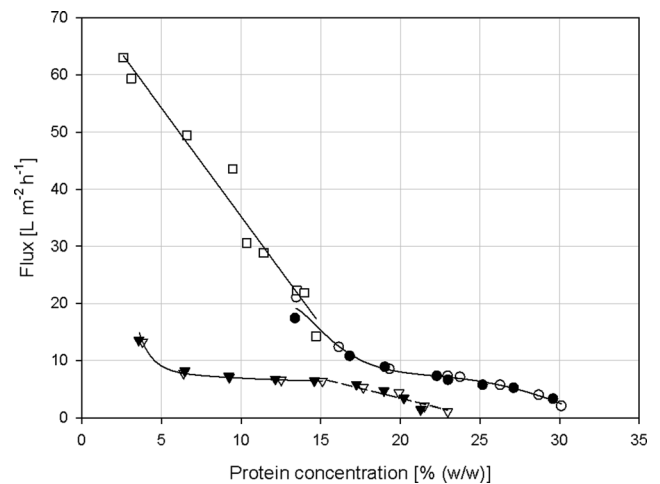


Fig. 3 Permeate flux of skim milk as a function of protein concentration for the spiral wound module (*inverted empty triangles* trial 1, *inverted filled triangles* trial 2, transmembrane pressure (TMP) = 200 kPa), the rotating membrane module (*empty circles* trial 1, *filled circles* trial 2, TMP = 100 kPa; $N = 1000\text{ min}^{-1}$) (data taken from Meyer et al. 2015) and the ceramic tubular membrane (*empty squares*, TMP = 300 kPa; $\tau_w = 100\text{ Pa}$) at a filtration temperature of $50\text{ }^\circ\text{C}$

a protein content of maximum 20% (w/w) can be reached by applying the tubular membrane. A strong flux decline could be observed above a protein content of 15% (w/w) for the SWM, whereas such high viscosities could still be processed with the dynamic system and a protein content of approximately 30% (w/w) could be reached (Meyer et al. 2015).

The dynamic system was used to overcome the limitation of the conventional systems by the high viscosities of the skim milk concentrates. The high viscosity can mainly be attributed to the casein micelles. One reason is the high water binding capacity of the micelles of approximately 4 g per gram of protein (McMahon and Oommen 2013). In addition, the interactions between the casein micelles are intensified and the hairy layers of the micelles overlap with increasing concentration as soon as a certain volume fraction is achieved. This results in a strong viscosity increase (Bouchoux et al. 2009; Dahbi et al. 2010). In contrast, sweet whey protein concentrates do not show such a strong increase in viscosity. This can mainly be attributed to the absence of the casein micelles. Furthermore, whey proteins show a lower water binding capacity (e.g., 0.46 g per gram of protein for the major whey protein β -Lactoglobulin (Sawyer 2013)) as compared to casein micelles. Hence, the application of the dynamic system is not required for the concentration of the protein fraction of sweet whey. The results of the UF of sweet whey applying SWM (data taken from Meyer and Kulozik 2016) and tubular ceramic membrane ($\tau_w = 100\text{ Pa}$) at a TMP of 300 kPa and a temperature of $10\text{ }^\circ\text{C}$ are depicted in Fig. 4. It can be seen that the flux decreased with increasing protein concentration independent of the membrane system and that the permeate flux of the tubular ceramic membrane is higher than the flux of the SWM. Up to a protein content of 10% (w/w), the ceramic

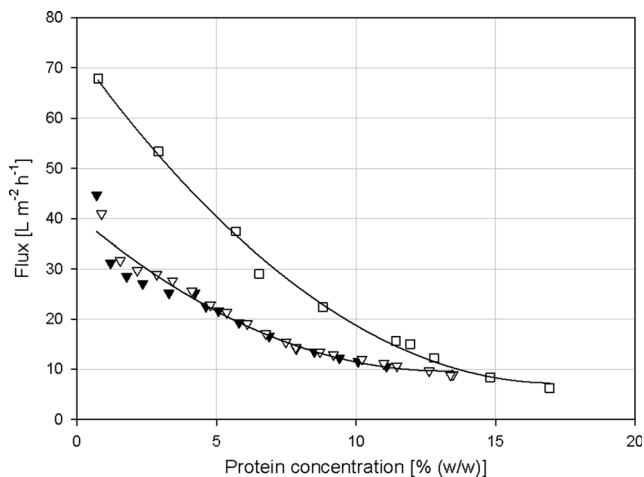


Fig. 4 Permeate flux of sweet whey as a function of protein concentration for the spiral wound module (*inverted empty triangles* trial 1, *inverted filled triangles* trial 2, transmembrane pressure (TMP) = 300 kPa) (data taken from Meyer and Kulozik 2016) and the ceramic tubular membrane (*empty squares*, TMP = 300 kPa; $\tau_w = 100$ Pa) at a filtration temperature of 10 °C

tubular membrane gave 1.5 to three times higher flux values. At higher protein concentrations, the difference was less distinctive. The filtration with the SWM had to be stopped at a protein concentration of 14% (w/w) due to the low quantity of remaining whey concentrate in the feed tank. The filtrations with the tubular ceramic membrane were carried out up to 17% (w/w). However, it can be estimated from the slope of both curves that higher protein concentrations of at least 20% (w/w) could be reached.

Regarding the retention of the protein fraction of skim milk, an almost complete retention was reported for the concentration with the SWM and dynamic device (Meyer et al. 2015). The same accounts for the protein fraction of skim milk using the tubular ceramic membrane. For the filtration of sweet whey and its concentrates, the major whey protein β -Lactoglobulin was used as a measure for the whey protein retention. During concentration using the SWM, β -Lactoglobulin could not be detected in the permeate. For the ceramic tubular membrane, it was found that the retention coefficient of β -Lactoglobulin slightly decreased with increasing concentration. However, it was always >95%.

Impact of the Previous Protein Removal on Nanofiltration and Reverse Osmosis

In Fig. 5, the NF flux of skim milk, sweet whey, and the protein-free milk serum is plotted against the VRR at a TMP = 2 MPa and a temperature of 10 °C. It is obvious that the permeate flux could clearly be increased by an upstream UF and that the effect was more pronounced for skim milk as compared to sweet whey. The permeate flux of the protein-free serum was higher by a factor of two to three compared to

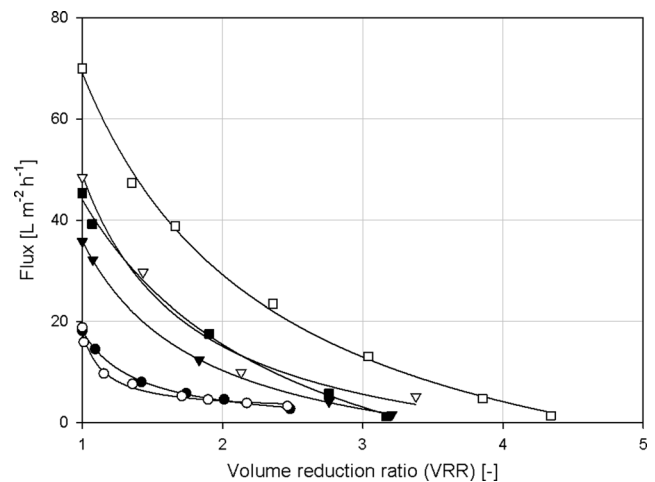


Fig. 5 Permeate flux as a function of VRR during the concentration of skim milk (*filled circles* TMP = 2 MPa, *○* TMP = 4 MPa), sweet whey (*inverted filled triangles* TMP = 2 MPa, *inverted empty triangles* TMP = 4 MPa), and protein-free serum (*filled squares* TMP = 2 MPa, *empty squares* TMP = 4 MPa) via nanofiltration at a filtration temperature of 10 °C

skim milk. Compared to sweet whey, the flux could be enhanced by approximately 30% by removing the protein fraction. The VRR could not (compared to sweet whey) or only slightly (compared to skim milk) be enhanced. The bigger impact of the protein removal on skim milk compared to sweet whey can be attributed to the five times higher protein content of skim milk. The higher protein content induced a stronger deposit formation, and, in turn, a bigger impact of a protein removal could be observed for skim milk compared to sweet whey. The influence of an upstream UF on the NF flux of sweet whey was already studied in the literature. Suárez et al. (2009) compared the NF of sweet whey and protein-free serum at a TMP = 2 MPa and a temperature of 15 °C and only found a small difference in flux. In contrast, Rektor and Vatai (2004) reported a three (at a VRR = 1) to 4.5 times (at a VRR = 3) higher permeate flux of the protein-free serum compared to sweet whey at a temperature of 40 °C and a TMP = 3 MPa. One possible explanation for the bigger impact of the protein removal reported by Rektor and Vatai (2004) as compared to the present study might be the composition of the whey. The whey used for the present study was clarified. Hence, some fouling active substances like, e.g., cheese fines were previously removed. Rektor and Vatai (2004) used untreated whey. This may result in a more intense fouling layer and, in turn, in a bigger impact of a previous protein removal. Another possible explanation for the bigger effect might be the fact that Rektor and Vatai (2004) conducted the filtration at a higher TMP = 3 MPa as compared to the TMP of 2 MPa used in the present study. Suárez et al. (2006) investigated the impact of TMP on the permeate flux of sweet whey and its ultrafiltration permeate in non-concentrating mode. Suárez et al. (2006) found almost no difference in permeate flux up

to $TMP = 2$ MPa, whereas the difference increased with increasing TMP up to a pressure of 4 MPa. Hence, the higher TMP of 3 MPa applied in the experiments conducted by Rektor and Vatai (2004) may be another meaningful explanation for the bigger influence of the protein removal compared to the results of the present study. As a consequence, it was investigated whether the effect of the protein removal on permeate flux and VRR can be enhanced by applying a higher pressure of 4 MPa (Fig. 5). The application of a higher TMP clearly enhanced permeate flux and VRR during the concentration of protein-free serum, whereas the effect was less pronounced for sweet whey and no impact of the pressure increase on skim milk could be observed. The maximum VRR of protein-free serum increased from 3.2 to 4.3, and, compared to skim milk, the flux could be enhanced by approximately 500%. The filtration of skim milk was probably already in the limiting region, where a pressure increase is compensated by a more intense deposit formation or a rearrangement within the deposit layer. As a consequence, neither the permeate flux nor the VRR could be enhanced by the pressure increase. In the case of sweet whey, the permeate flux could be increased, although the effect was not as distinct as for protein-free serum. This indicates that the NF of sweet whey was in the region where a pressure increase is partly compensated by a more intense deposit formation.

An enhancement of the flux through a protein removal means that the NF stage can be accelerated. In order to quantify the acceleration of the whole process when substituting a single NF by an UF/NF cascade, it was calculated how long it takes to reach a certain VRR starting from an initial volume of 100 L. Figure 6 shows the VRR plotted against the concentration time for skim milk and sweet whey at a typical operation pressure of 2 MPa that will be substituted in a potential

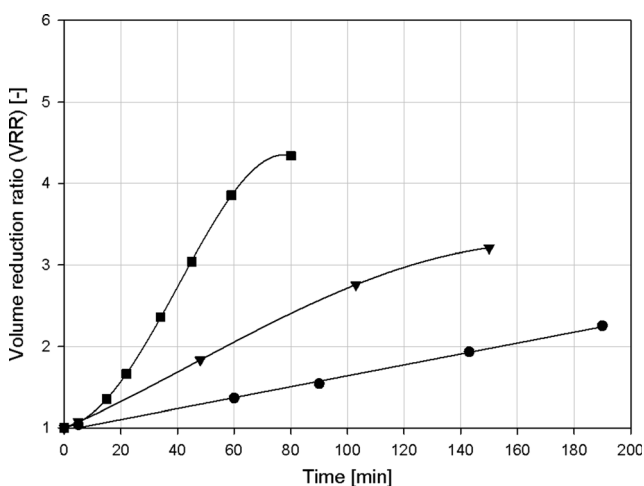


Fig. 6 VRR plotted against the calculated time to concentrate a starting volume of 100 L of skim milk (filled circles) and sweet whey (inverted filled triangles) at a TMP of 2 MPa and of protein-free serum (filled squares) at a TMP of 4 MPa at a filtration temperature of 10 °C via nanofiltration

future process by the concentration of protein-free serum at a TMP of 4 MPa, which gave the best filtration results. It can be seen that a higher VRR could be reached in a shorter amount of time when removing the protein fraction. It took, e.g., approximately 150 min to reach a $VRR = 2$ during the concentration of skim milk, whereas it only took approximately 30 min when concentrating protein-free serum. This means that the NF stage could operate approximately five times faster when removing the milk protein fraction. During the concentration of sweet whey, it took approximately 60 min to reach a $VRR = 2$. Hence, the process could be accelerated by a factor of two. $VRR = 3$ could even be reached approximately three times faster.

Besides NF, the protein-free serum can be concentrated by RO, too. The flux-enhancing effect of an upstream UF on RO has already been discussed in a previous work (Meyer and Kulozik 2016). Analogous to Fig. 6, it was calculated how long it takes to concentrate 100 L to a certain VRR. The data were taken from Meyer and Kulozik 2016. The VRR as a function of the concentration time for skim milk and sweet whey (at a typical operation pressure of 4 MPa during RO) and for the protein-free serum (at a TMP of 6 MPa that gave the best filtration results) is shown in Fig. 7. Again, the upstream UF enabled the concentration up to higher VRR in less time. It took, e.g., approximately 130 min to concentrate skim milk up to a $VRR = 3$, whereas the same VRR was reached after 60 min with protein-free serum. This resulted in an acceleration of the process by a factor of approximately two. The concentration of sweet whey up to a $VRR = 3$ took approximately 85 min. Hence, the concentration could take place 1.5 times faster when removing the whey protein fraction.

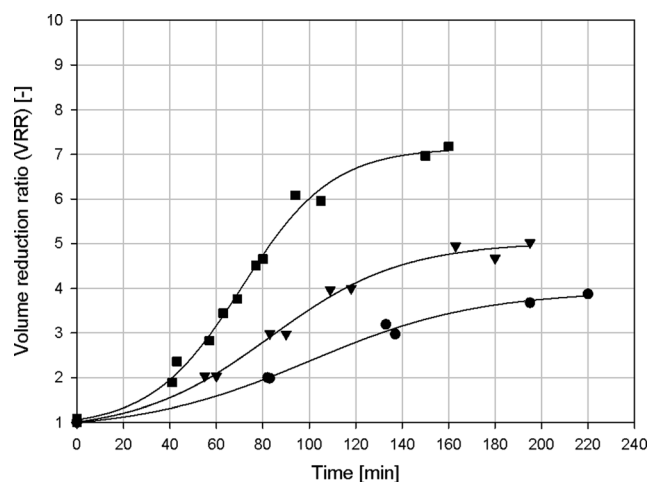


Fig. 7 VRR plotted against the calculated time to concentrate a starting volume of 100 L of skim milk (filled circles) and sweet whey (inverted filled triangles) at a TMP of 4 MPa and of protein-free serum (filled squares) at a TMP of 6 MPa at a filtration temperature of 10 °C via reverse osmosis

Comparison of Cascade and Single-Stage Operation in Terms of Volume Reduction Ratio

It was shown in “Concentration of the Protein Fraction” that the protein fraction of skim milk and sweet whey can be concentrated up to a protein content of 30 and 17% (w/w), respectively, by UF. Furthermore, it was demonstrated in “Impact of the Previous Protein Removal on Nanofiltration and Reverse Osmosis” for the NF and in a previous work for the RO (Meyer and Kulozik 2016) that the VRR could be increased by an upstream UF and that the application of a higher TMP (4 MPa during NF and 6 MPa during RO) is advantageous when concentrating protein-free serum. The question whether the VRR of the recombined concentrate is higher than the VRR achieved by a single NF and RO, respectively, will be answered in the following. As soon as the permeate flux drops below a certain level, the filtration becomes inefficient. Therefore, a minimum flux is often defined as stop criterion. The following considerations are based on a stop criterion during NF and RO of $5 \text{ L m}^{-2} \text{ h}^{-1}$. Table 2 lists the VRR obtained by a single NF and RO at a typical operating pressure (2 and 4 MPa, respectively) and the VRR achieved by applying UF and NF or RO (at 4 and 6 MPa, respectively) in cascade mode. The VRR that can be achieved by applying a higher TMP during NF or RO of skim milk and sweet whey (4 and 6 MPa, respectively) is also included in the consideration. It is obvious that the application of a higher TMP could not increase the VRR during NF of skim milk, whereas the VRR could be increased by approximately 78% from VRR = 1.8 to VRR = 3.2 by application of a UF-NF cascade. The VRR of sweet whey can be increased by an upstream UF (by approximately 96%) as well as by applying a higher TMP of 4 MPa during NF (by approximately 28%). Furthermore, it can be seen that the application of a higher TMP does not enhance the VRR during RO of skim milk. Moreover, the use of an UF-RO cascade connection only slightly increases the VRR by 3% (from VRR = 3 to VRR = 3.1) for skim milk and by 5% (from VRR = 4.4 to VRR = 4.6) for sweet whey. However, in the case of sweet whey, the application of a

higher TMP during RO of 6 MPa significantly enhances the VRR by 27% to VRR = 5.6. The higher VRR obtained by a single RO as compared to an UF-RO cascade can be explained by the comparably low effect of the prior protein removal on the RO of sweet whey. It can be seen from Fig. 5 that the final VRR after the concentration (TMP = 6 MPa) of protein-free serum and sweet whey does not differ as much as, e.g., the maximum VRR of protein-free serum and skim milk at the same TMP. The recombination of the UF and RO retentates results in a dilution of both concentrates. For sweet whey, this results in a lower overall VRR of the recombined concentrate as compared to the concentrate obtained via single RO.

Comparison Between Nanofiltration and Reverse Osmosis

Based on the results from “Impact of the Previous Protein Removal on Nanofiltration and Reverse Osmosis” and “Comparison of Cascade and Single-Stage Operation in Terms of Volume Reduction Ratio,” it can be concluded that the flux-enhancing effect by an upstream UF as well as the impact on final VRR were more pronounced for NF as compared to RO. A possible explanation is the different membrane structures. RO membranes are homogenous, whereas NF membranes have a looser structure and the homogenous areas are interrupted by pores. Hence, the fluid cannot only be transported diffusively through the homogenous parts of the NF membrane but also flows convectively through the pores. As a consequence, the permeate flux through the NF membrane is relatively high if there are no colloidal particles present in the feed solution. During RO, the mass transport through the membrane is exclusively diffusive. This leads to lower permeate flux values as compared to NF. As soon as there are colloidal particles like proteins present in the feed solution, the formation of a deposit layer is induced. The deposit layer acts as an additional flow resistance and reduces the permeate flux through the NF as well as the RO membrane. In NF, the colloidal particles can additionally block the pores of the membrane. This pore blockage may result in a tremendous flux decline as compared to a clean membrane, as especially the convective transport through the pores, which accounts for the larger part of the permeate flow, is diminished. In contrast, the impact of a deposit layer on the permeate flux through a RO membrane may be lower, as no pores are present that can be blocked. Conversely, this means that the removal of the protein fraction can have a higher impact on the NF than on the RO.

Table 2 Volume reduction ratio achieved during the concentration of skim milk and sweet whey with a single nanofiltration (NF) or reverse osmosis (RO) at different transmembrane pressures (TMP) as well as with an upstream ultrafiltration (UF) in cascade mode

	Skim milk VRR	Sweet whey VRR
NF (TMP = 2 MPa)	1.8	2.5
NF (TMP = 4 MPa)	1.8	3.2
UF + NF (TMP = 4 MPa)	3.2	4.9
RO (TMP = 4 MPa)	3	4.4
RO (TMP = 6 MPa)	3	5.6
UF + RO (TMP = 6 MPa)	3.1	4.6

Energy Considerations

The data for the specific primary energy demand of the membrane processes that were calculated from the capacities of the

installed pumps according to Eqs. (6) and (7) are listed in Table 3.

It can be derived that the average specific primary energy demand for the RO of skim milk and whey is 110.6 (± 29.6) and 36.5 (± 6.0) kJ kg^{-1} , respectively. The UF has an average specific primary energy demand of 41.9 (± 3.3) kJ kg^{-1} . The difference in energy demand for the RO of skim milk and whey can be attributed to the composition of the fluids. The RO plants for skim milk and whey have a similar design, i.e., the capacities of the installed pumps are comparable. However, the higher protein content of skim milk results in lower flux values as compared to whey. Hence, a lower permeate volume can be gained in a certain time that results in a higher specific energy demand of skim milk compared to whey, according to Eq. (7). The collected data correspond well with values that were estimated before for large-scale production plants. Stabile (1983) based his calculations on estimations for medium-size to larger RO plants. He calculated a specific primary energy demand for the RO of skim milk of 80–110 kJ kg^{-1} (for DM from 8 to 25%). The economic evaluation of Peters (2005) was based on realistic data for different whey processing options. He reported a specific primary energy demand of 38.2 kJ kg^{-1} for the RO of sweet whey (to DM of 18–20%) as well as for the UF of sweet whey to produce a whey protein concentrate (WPC34). Hence, the assumptions and estimations that were made before by other research groups could be verified by the collection and evaluation of real data for the energy demand of membrane processes in the dairy industry.

To compare the energy demand of the single-stage with the cascade operation, it was calculated how much energy is needed for the concentration of 100 kg skim milk to a VRR of three using only RO. With an average energy demand during RO of skim milk of 110.6 kJ kg^{-1} , the energy demand was found to

be 7377 kJ. In addition, the energy consumed by concentrating the same amount of skim milk via UF in series with RO to a total VRR of 3.1 was determined. The exact energy demand of the RO of protein-free serum is not known. However, it can be estimated that it is in the same range as the energy demand of sweet whey or even below. Therefore, an energy demand of 36.5 kJ kg^{-1} for the RO of protein-free serum was used for the calculation. This resulted in a total energy demand for the concentration via an UF-RO cascade of 6160 kJ. Hence, the substitution of the energy-consuming RO of skim milk by two less energy-consuming processes (UF of skim milk and RO of protein-free serum) results in energy savings of approximately 16%. The same calculations were carried out for the concentration of 100 kg sweet whey via a single RO (VRR = 4.4) and an UF-RO cascade (VRR = 4.6). In this case, the concentration via UF-RO cascade consumes approximately 2.4 times more energy as compared to the concentration via a single RO (6864 compared to 2821.5 kJ). This is due to the fact that the RO of sweet whey is not as energy consuming as the RO of skim milk. Hence, the substitution of this unit operation by two processes that have a similar energy demand is not advantageous in terms of energy efficiency.

Possible Membrane Cascades

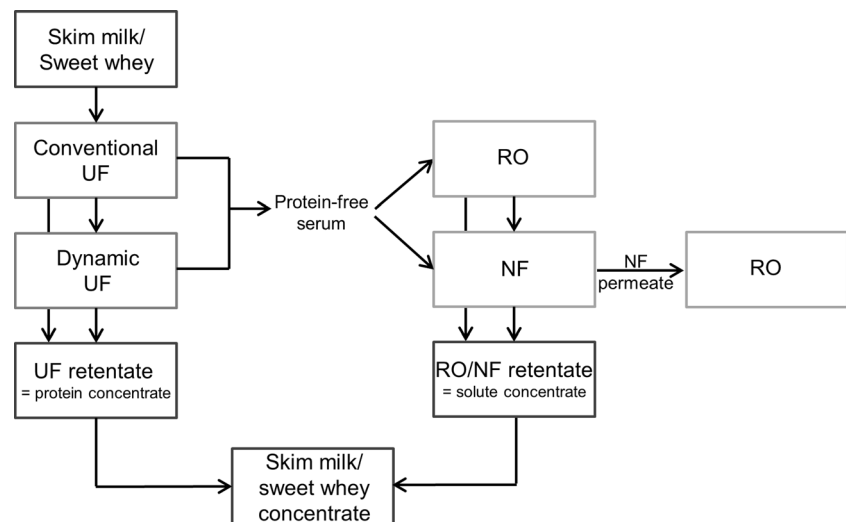
A flowchart of possible membrane cascades for the concentration of skim milk and sweet whey via UF, NF, and RO is shown in Fig. 8. Based on the results from Fig. 3, several cascades of the conventional (SWM and tubular membranes) and dynamic UF devices can be deduced to concentrate the protein fraction of skim milk. It is possible to concentrate up to a protein content of 15% (w/w) with conventional systems before switching to the dynamic unit and reach a high final protein concentration of 30% (w/w). Regarding the conventional systems, the ceramic tubular membrane is preferable because of the higher permeate flux. As ceramic membranes are more expensive than SWM, it is also conceivable to use a combination of both to reach a protein content of 15% (w/w). The skim milk can for example be concentrated to a VRR of two or three using a SWM, before switching to the ceramic tubular membrane. At this stage, the retentate volume is already drastically reduced and the ceramic membrane unit can be designed with lower dimensions and consequently at lower cost. An alternative is the concentration up to a protein content of approximately 22% (w/w) using a SWM, before switching to the dynamic system. However, in this case, a stronger flux decline during the enrichment with the SWM needs to be accepted above a protein concentration of 15% (w/w).

Different approaches are also conceivable for the concentration of sweet whey. Tubular membranes are favorable compared to SWM in terms of permeate flux values (see Fig. 4). When including investment costs, it is also imaginable to use SWM for the concentration. Another conceivable procedure is

Table 3 Final dry matter (DM) and specific, primary energy demand per kilogram of water removed ($E_{m,P}$) for different reverse osmosis (RO) and ultrafiltration (UF) installations and various starting products

Process	Starting product	DM _{concentrate} [%]	$E_{m,P}$ [kJ kg^{-1}]
RO	Skim milk	25	70.9
RO	Skim milk	29	139.1
RO	Skim milk	36	106.4
RO	Skim milk	36	125.8
RO	Sweet whey	12	29.7
RO	Sweet whey	18	35.5
RO	Sweet whey	20	32.1
RO	Acid whey	18	41.8
RO	Acid whey	25	43.6
UF	Sweet whey	17	41.2
UF	Acid whey	18	39.1
UF	Skim milk	20	45.5

Fig. 8 Possible membrane cascades for the concentration of skim milk and sweet whey via ultrafiltration (UF), nanofiltration (NF), and reverse osmosis (RO)



the use of a combination, i.e., SWM can be used, e.g., to concentrate up to a VRR = 5, before switching to the ceramic tubular membrane, and increase the permeate flux by approximately 80%. Again, the ceramic membrane unit can then be designed with lower dimension and at lower cost.

The protein-free serum can be concentrated by means of NF or RO. When choosing NF for the concentration of the solute fraction, this can result in an improvement of the powder quality. However, it has to be noted that the resulting permeate contains monovalent ions. Hence, the permeate has to be further processed, like, e.g., by means of RO. Another possible scenario is the application of RO and NF in series. First, as long as the VRR and hence the osmotic pressure are still comparably low, RO can be used for the concentration. Then, in the late stages of the process, NF can be applied. Due to more open membrane structure, the process is then less limited by the osmotic pressure and the applied pressure can be used more effectively to reach high final dry matter contents.

Conclusion

The substitution of a NF by an UF-NF cascade resulted in an increase in VRR of the skim milk and sweet whey concentrates. Hence, less water needs to be eliminated in the subsequent thermal processes. The application of an UF-RO cascade only slightly increased the VRR. However, the substitution of the RO of skim milk by two less energy-consuming processes markedly reduces the energy demand of the non-thermal concentration step itself. The energy demand of the concentration of sweet whey cannot be reduced by application of the UF-RO cascade. However, the VRR could be markedly increased by applying a higher TMP during the single-stage

RO, resulting in a reduced energy demand for evaporation. The proposed cascades can easily be integrated in already existing membrane processes. Regarding the application of large-scale dynamic devices, further research has to be done to investigate whether the high retentate viscosity causes practical problems, like, e.g., during discharging of the product. Depending on the desired product and permeate quality, it has to be decided whether NF or RO is applied. The achievement of higher VRR may clearly cut production costs. Furthermore, the output of climate-damaging greenhouse gases can be reduced, resulting in a reduction of the carbon footprints of the products.

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