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ORIGINAL PAPER

Cheese whey tangential filtration using tubular mineral membranes

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Membrane separation techniques are extensively used in dairy industry both for milk and cheese whey processing. However, cheese whey might still be considered as a problematic waste despite its high content of many valuable substances, such as proteins, lactose or minerals, which can be further used, e.g. in human nutrition, pharmacy or biotechnologies. Another problem, which food technologists have to face, is variable quality, composition and properties of food materials bringing high demands on manufacturing industry. In this paper, filtration kinetics and separation efficiency during purification and fractionation of cheese whey (sweet and salty) from Czech dairies by pilot-plant filtration (Bollene, France) was studied using tubular membranes (Membralox, USA). Various mineral membranes' cut-offs were tested and all experiments ran in the retentate recycling mode. The obtained mass concentration factors were between 1.9 and 16.5. Steady state fluxes were calculated from the experimental data using a mathematical model. Fine ultrafiltration on a 5 kDa membrane gave steady state fluxes of 14–19 L m^{-2} h^{-1}. The coarse pre-filtration on 100 nm, 200 nm or 500 nm membranes showed various permeate fluxes between 22 L m^{-2} h^{-1} and 153 L m^{-2} h^{-1}. Despite the high pore sizes of the used membranes, lactose was partially rejected by all membranes tested.
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Keywords: microfiltration, ultrafiltration, nanofiltration, inorganic membranes, proteins, lactose

Introduction

Whey contains more than a half of the solids of original whole milk, including whey protein and most of the content of lactose, minerals and water soluble vitamins. Even if some applications of whey already exist, it is still often considered as a waste product of the cheese industry and the utilisation of minor whey components represents a big challenge for whey processing nowadays.

According to the methods used in cheese manufacturing, different types of whey can be obtained. Sweet whey is obtained during the production of rennet types (enzyme action) of hard cheese, acid whey is obtained during acidification of cheese, and salty whey is drained from the cheese vat post curd salting. Salty whey is currently underutilised in the dairy industry because of its high salt content and increased processing and disposal cost (Blaschek et al., 2007). Whey composition depends on the production path chosen and on the initial milk properties but typically, the total solids content of whey is 5.0–6.0 %, of which approximately 80 % are lactose and 10–15 % are proteins (Räsänen et al., 2002). Whey proteins represent approximately 90 % of all present milk proteins, the remaining 10 % is represented by casein or casein macropeptide (CMP), respectively.

Various membrane separation processes have a long-standing tradition in milk and cheese whey processing. Microfiltration (MF) is used for clarification, as well as for fat and microorganism removal. Fractionation and concentration of whey protein from cheese whey are among the most successful industrial applications of ultrafiltration UF (Cheryan & Kuo,

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Table 1. Membrane properties

Parameter	Value
membrane filtration area/m^2	0.24
pore sizes (cut-offs)/nm	500, 200, 100, 20, 5[a], 1[a]
membrane length/mm	1020
material	α-alumina (0.1–12 mm), zirconia (20–100 nm), TiO_2 (1–5 kDa)
number of channels:	19

a) In kDa.

1984; Hanemaaijer et al., 1989; Atra et al., 2005). Nanofiltration (NF) and reverse osmosis (RO) are used for concentration of lactose and whey demineralisation (Räsänen et al., 2002; Suárez et al., 2006).

Generally, two kinds of problems must be faced in ultrafiltration: decline in flux with time and partial solute rejection. Concentration polarisation and membrane fouling are mainly responsible for the reduction of UF efficiency in whey processing (Brans et al., 2004; Rao, 2002). Cheryan (1998) suggested that pH should be far away from the isoelectric point of the proteins to minimise the fouling effect. A recent study on membrane fouling confirmed that the main fouling mechanisms both on polymeric flat-sheet and ceramic tubular membranes are complete pore blocking and cake formation (Corbatón-Báguena et al., 2015).

The choice of a UF membrane is also very important. The membrane material and molecular conformation of the whey protein are of considerable influence on the flux decline (Marshall et al., 1993; Rao, 2002; Merin & Cheryan, 1980). Many papers on membrane filtration of cheese whey can be found; however, mostly using polymeric membranes, such as poly(ethersulphone) (PES) tested by Yorgun et al. (2008) and Konrad et al. (2012), or poly(vinylidene difluoride) and polyamide (Atra et al., 2005). The polymeric polysulfone membrane remains to be the most widely used type of membranes in whey UF primarily because of its low cost, good thermal stability, and mechanical properties (Brans et al., 2004; Qin et al., 2003). It is commonly believed that, in comparison with ceramic and hydrophilic polymeric membranes, hydrophobic polysulfone membranes suffer from lower fluxes and more severe fouling (Marshall et al., 1993). However, Doyen et al. (1996) showed that in whey UF, practically, the same flux/concentration factor and whey permeability coefficient were obtained as for polysulfone and ceramic membranes. Aimar et al. (1988) showed that, in UF of sweet whey at pH 6.3 using a tubular ceramic membrane, there was no considerable difference in the flux plateau values at cross flow velocities from 1.8 m s^{-1} to 4.0 m s^{-1} at 50 °C and the trans-membrane pressure (TMP) of 300 kPa.

Since UF covers a wide range of molecular weights, it is important to know the molecular weight of the targeted protein in order to ensure good separation. Due to the proteins' large range of sizes and shapes, it is also difficult to recover all proteins (and only the proteins) in a single-step filtration. Even though the recovery of proteins can be very effective, it is not the case of minerals and lactose, which are equally distributed in different phases.

Although whey filtration is the topic of a large number of papers, no data comparing permeate fluxes on a wide range of ceramic membranes with various cut-offs are available. Also, the cut-offs declared by membrane producers do not always express the real rejection of components in a complex solution such as whey. This work focused on the separation of lactose and proteins from bovine cheese whey using commercially available inorganic tubular membranes in pilot-plant experiments. Separation efficiency and permeate fluxes were measured with various membrane cut-offs comprising a whole range of pressure driven separation processes from micro- to nanofiltration. Filtration kinetics during purification and fractionation of different types of cheese whey (sweet and salty) from Czech dairies were determined using six tubular inorganic membranes with various pore sizes ranging from 1 kDa to 500 nm. Experimental values of permeate fluxes were fitted with a mathematical model to calculate the steady state fluxes and the rejections of lactose and proteins were also measured.

Experimental

Filtration experiments were carried out using inorganic tubular membranes Membralox (Pall, USA) with cut-offs: 1 kDa, 5 kDa, 20 nm, 100 nm, 200 nm and 500 nm. Properties of the ceramic membranes are summarised in Table 1.

The experiments were carried out using a pilot-plant filtration unit T.I.A. Bollene (France; Table 2).

All filtrations were carried out using different kinds of cheese whey; composition of whey is presented in Table 3.

A dried sweet whey solution (DW-sweet) was prepared from dried sweet whey (Moravia Lacto, Czech Republic) diluted with demineralised water to the final concentration of dry solids of 30 g L^{-1}.

Natural sweet whey (NW-sweet) from the dairy in Jihlava (Czech Republic) was processed immediately after its delivery from the dairy; it was not stored nor modified before the UF.

Concentrated natural sweet whey (CNW-sweet) was provided by the dairy Polabské mlékárny, Milko

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Table 2. Technical specification of the T.I.A filtration unit for cross-flow ultra- and microfiltration (Techniques Industrielles Appliquées (Bollene, France))

Parameter	Value
pump output (Hyginox SC20, Italy)/(m^3 h^{-1})	100
cross-flow velocity/(m s^{-1})	1–7
operating pressure/kPa	100–500
capacity (feed tank)/dm^3	50

Table 3. Whey composition

Whey type	Dairy	pH	Dry solid content/%	Lactose content/(g L^{-1})	Total protein content/(g L^{-1})
dried sweet	Jihlava	7.13	2.80	82.00	n.a.
natural salty	Příšovice	6.10	7.09	35.59	n.a.
natural sweet	Jihlava	5.85	4.50	27.98	n.a.
natural salty	Dolní Přím	6.10	9.40	75.72	6.04
concentrated sweet	Poděbrady	6.00	17.20	108.00	22.00

n.a. – Not available.

Table 4. Experimental conditions of filtration processes

Membrane cut-off/nm	Experiment	Whey used	Dairy	Driving pressure/kPa	Temperature/ °C	Duration of filtration/min
500	F 500 D-sw	dried sweet	Jihlava	200	20–21	90
500	F 500 N-sa	natural salty	Dolní Přím	100 and 200	20–22	170–527
200	F 200 D-sw	dried sweet	Jihlava	100	20–25	100
100	F-100 D-sw	dried sweet	Jihlava	100 and 200	18–20	20–70
100	F-100 N-sa	natural salty	Příšovice	200	20	100
100	F-100 N-sw	natural sweet	Jihlava	200	20	100
20	F-20 N-sw-con	concentrated sweet	Poděbrady	100	16–24	160
5[a]	F-5 D-sw	dried sweet	Jihlava	200	18–21	31–130
5[a]	F-5 N-sa	natural salty	Příšovice	200	20	100
5[a]	F-5 N-sw	natural sweet	Jihlava	200	20	100
1[a]	F-1 N-sw-con	concentrated sweet	Poděbrady	100	22–28	80

a) In kDa; sw – sweet whey, sa – salty whey, con – concentrated whey.

(Poděbrady, Czech Republic) after being concentrated by reverse osmosis on a membrane GE Osmonics, type AF3840C-30D (Lenntech, The Netherlands).

Natural salty whey (NW-salty) was supplied either by the dairy in Příšovice (Czech Republic) or by the Dolní Přím dairy (Czech Republic). The first one was used directly for UF, the latter was filtered through a mesh filter before the ultrafiltration. The sieve aperture size was 0.2 mm.

UF conditions are shown in Table 4. Different types of whey and different membrane cut-offs were combined and the filtration experiments were carried out at least in duplicates. All filtrations were performed in the retentate recycling mode and the temperature was mostly held constant, unless the temperature range shown in Table 4 indicates otherwise. The trans-membrane pressures were also constant during the filtration, either 100 kPa or 200 kPa. Filtrations of natural salty whey on the 500 nm membrane and of dried sweet whey on the 10 nm membrane were performed at both TMPs, i.e. 100 kPa and 200 kPa. Table 4 also shows the duration of the filtration experiments.

During the cleaning procedure, the module and membranes were flushed several times with cold tap water which was filtered through a cartridge filter to remove iron traces. Then, the membrane was washed using 3 vol. % NaClO at 60 °C for 60–80 min and then rinsed with water twice.

Lactose content was measured by anion-exchange chromatography with amperometric detection (electrochemical detector ED50, Dionex, USA), column CarboPac PA1 (2 × 250 mm, Dionex), at the flow rate of 0.25 mL min^{-1}, temperature of 25 °C, and the mobile phase composition: 50 mM NaOH; isocratic elution was followed by 20 min of column regeneration in 200 mM NaOH.

Total protein content was measured using the Kjeldahl method with the multiplication factor to calculate the protein content of 5.7 (International Association for Cereal Science and Technology, 1996). The content of individual proteins was analysed by an

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Table 5. Steady state permeate fluxes (J_{SS}), MCF and pure water fluxes before filtration (J_{wi})

Membrane cut-off/nm	Experiment	MCF	J_{SS}/(L m^{-2} h^{-1})	J_{wi}/(L m^{-2} h^{-1})
500	F 500 D-sw	4.39	31.5	948
500	F 500 N-sa	7.5–16.5	31.2–58.8	1267–1747
200	F 200 D-sw	3.35	33.6	972
100	F-100 D-sw	2.72–3.29	21.5–58.4	742–863
100	F-100 N-sa	4.43	65.6	987
100	F-100 N-sw	6.97	54.0	1046
20	F-20 D-sw	3.04	153.4	678
5[a]	F-5 D-sw	2.17–2.66	11.8–18.3	261–863
5[a]	F-5 N-sa	2.30	13.8	916
5[a]	F-5 N-sw	4.56	18.7	963
1[a]	F-1 N-sw-con	1.85	3.2	34

a) In kDa; sw – sweet whey, sa – salty whey, con – concentrated whey.

ion chromatography HPLC system Series 1100 (Agilent Technologies, Germany) with a UV-VIS detector (TSP Spectra System UV 200, Germany). Dry solid content was determined by drying the sample at 105 ℃ for 2 h.

Pure water flux (J_w) was measured before the filtration and after the membrane cleaning and calculated according to Eq. (1):

$$J_w = J_P k_T / S \tag{1}$$

where J_P is the permeate flux (L h^{-1}) of distilled water; S is the filtration area (m^2); k_T is the viscosity coefficient for conversion at the temperature of 20 ℃.

Feed and permeate masses were measured before and after the filtration, however the amount of retentate could not be precisely measured due to the losses of the solution inside the filtration units. That is why the amount of retentate was calculated from the mass balance expressed in Eq. (2):

$$m_R = m_F - m_P \tag{2}$$

where m_R, m_P and m_F are masses of retentate, permeate and feed (kg), respectively.

Mass concentration factor (MCF) was then calculated according to Eq. (3):

$$\mathrm{MCF} = m_F / m_R \tag{3}$$

Rejection factor R_i was expressed by Eq. (4):

$$R_i = 1 - \frac{c_{iP}}{c_{iF}} \tag{4}$$

where c_{iP} and c_{iF} are the concentrations (g L^{-1}) of component i in the permeate and in feed, respectively.

To study the filtration kinetics, permeate fluxes were measured during the filtration. Since the filtrations were carried out at two different pressures (100 kPa and 200 kPa), all permeate fluxes were calculated for the pressure of 100 kPa assuming that the dependence of the permeate flux on the pressure applied is almost linear. From the data obtained, the steady state permeate fluxes (J_s) were calculated using the mathematical described by Eq. (5) suggested by Cheryan et al. (1998):

$$J_i = J_s + ae^{(-bt)} \tag{5}$$

where J_i is the permeate flux at time t (min), and a and b are constants calculated from the experimental data using the MS Solver.

Results and discussion

Table 5 shows the steady state permeate flow rates, achieved MCFs, as well as pure water fluxes (J_{wi}) before filtration. The course of filtration of salty whey on the 500 nm cut-off membrane and concentrated sweet whey on the 1 kDa membrane are shown in Figs. 1 and 2, respectively. Due to high permeability, microfiltration on the 500 nm membrane was one of the fastest processes where high steady state fluxes between 31–59 L m^{-2} h^{-1} and MCFs ranging between 1.9 and 17 were achieved. These values are suitable for industrial applications. On the other hand, nanofiltration on the open 1 kDa membrane was the slowest process with the steady state flux of 3 L m^{-2} h^{-1} (Figs. 2 and 3). Here, the low flux was caused by two factors: the pre-concentration of filtered whey using reverse osmosis, and the low trans-membrane pressure applied (100 kPa) to enable the comparison of the performance of all membranes under the same conditions.

High concentration of feed (MCF 2.7–7) and high steady state permeate fluxes were also achieved using the 100 nm cut-off membrane (21–58 L m^{-2} h^{-1}). The 5 kDa membrane provided steady state fluxes between 12–19 L m^{-2} h^{-1}. In order to obtain a whey protein concentrate, Atra et al. (2005) achieved a permeate flux varying from 33 L m^{-2} to 42 L m^{-2} on a 6–8 kDa cut-off poly(vinylidene difluoride) membrane at the of 800 kPa. In the same work, the permeate

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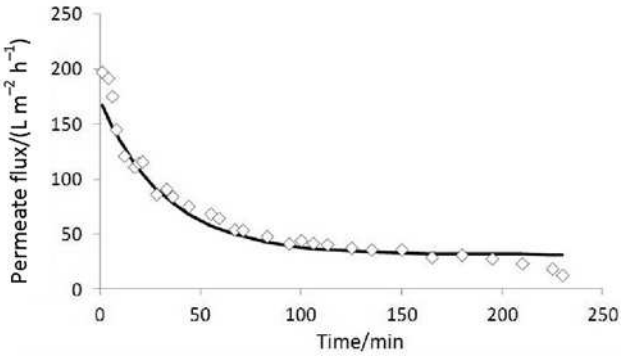

Fig. 1. Permeate flow rate in time during the filtration of natural salty whey on the 500 nm cut-off membrane including the flux calculated using the Cheryan model; conditions: temperature of 20–22 °C; ◊ – experimental data, — – mathematical model.

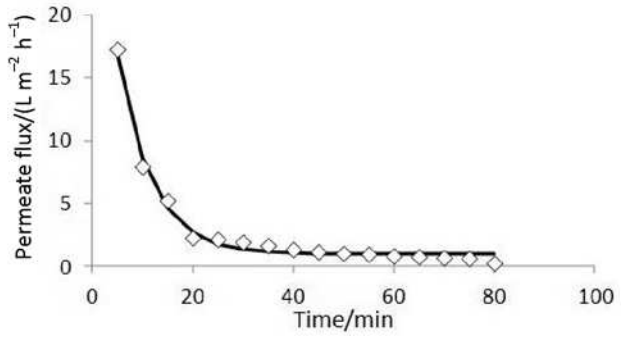

Fig. 2. Permeate flow rate in time during the filtration of concentrated sweet whey on the 1 kDa cut-off membrane including the flux calculated using the Cheryan model; conditions: temperature of 20–28 °C; ◊ – experimental data, — – mathematical model.

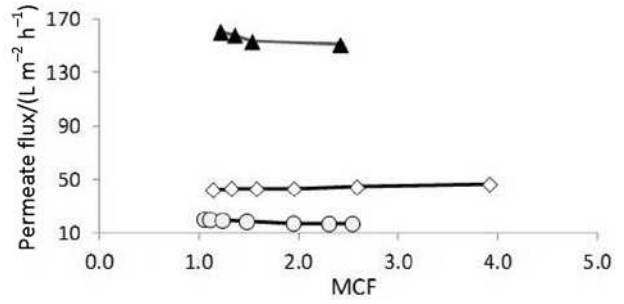

Fig. 3. Dependence of permeate flux on the mass concentration factor (MCF) during the filtration of solution prepared from dried sweet whey from the dairy in Jihlava; conditions: temperature of 18–21 °C; ▲ – 20 nm, ○ – 5 kDa, ◊ – 100 nm.

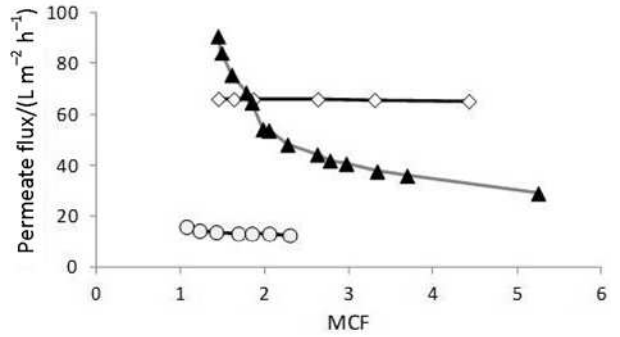

Fig. 4. Dependence of permeate flux on the mass concentration factor (MCF) during the filtration of salty whey from the dairy in Příšovice and in Dolní Přím; conditions: temperature of 20–22 °C; ▲ – 20 nm, ○ – 5 kDa, ◊ – 100 nm.

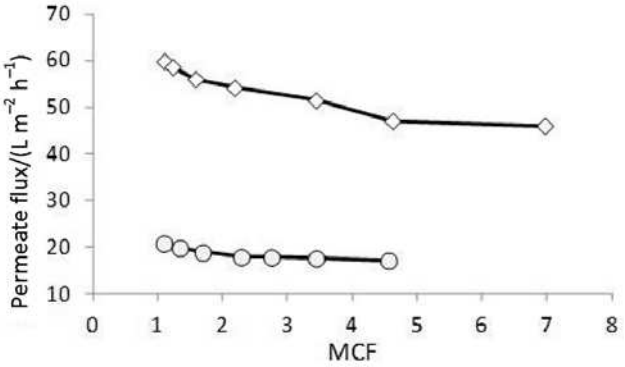

Fig. 5. Dependence of permeate flux on the mass concentration factor (MCF) during the filtration of natural sweet whey from the dairy in Jihlava; conditions: temperature of 20 °C; ○ – 5 kDa, ◊ – 100 nm.

flux increased with the temperature and pressure but decreased as the concentration factor increased since the concentration polarisation layer may precipitate. Optimum temperature and flow rate (linked with the tangential velocity and shear rate) providing higher flux were found at around 50 °C and 30 L m^{-2} h^{-1} for a plate membrane of the 6–8 kDa cut-off at the applied pressure of 350 kPa. A further increase in temperature would cause the denaturation of proteins (Atra et al., 2005).

The dependence of permeate flux on MCF for different membranes and cheese whey used is shown in Figs. 3–6. In general, the results confirm the findings of Atra et al. (2005) on a poly(vinylidene difluoride) membrane; permeate flux decreases with increasing whey concentration. In some cases, such as the 500 nm membrane in Fig. 4, the flux decline at MCF of 5.3 was 67 % of the initial flux. On the other hand, the 5 kDa membrane did not show any significant dependence of the permeate flux on MCF (Figs. 3 and 5) probably due to low TMPs, which were far from the critical pressure.

From the data it is apparent that the flux is affected by membrane permeability which is expressed by pure water flux. The flux on the 5 kDa membrane (Fig. 6) seems to be lower in salty whey than in sweet whey, however, the initial pure water flux was lower

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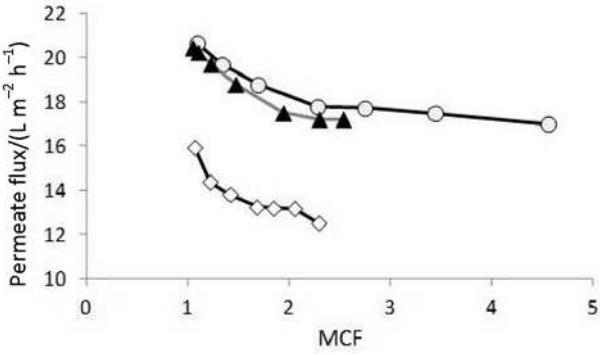

Fig. 6. Dependence of permeate flux on the mass concentration factor (MCF) during the filtration of various types of whey; conditions: membrane cut-off of 5 kDa; temperature of 18–21 °C; ◊ – salty whey, ○ – sweet whey, ▲ – dried sweet whey.

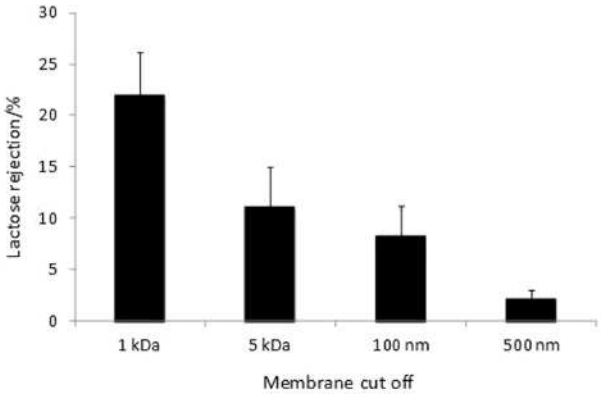

Fig. 7. Average lactose rejection on ceramic membranes of various cut-offs.

Table 6. Average lactose rejection on membranes with different cut-off

Membrane cut-off/nm	$R_{\mathrm{Lac}} \pm$ SD/%
1[a]	22.0 ± 4.1
5[a]	11.1 ± 3.9
100	8.3 ± 2.9
500	2.1 ± 0.9

a) In kDa; R_{Lac} – rejection of lactose, SD – standard deviation.

by nearly 50 L m^{-2} h^{-1} during the filtration of salty whey (Table 5) causing lower permeate flux.

Pure water flux values before the filtration (see J_{wi}, Table 5) were very variable and showed strong membrane fouling and insufficient membrane cleaning by 3 vol. % NaClO. Further optimisation of the cleaning procedure is necessary especially because natural whey composition is unstable; hence, higher variability of results can be expected.

Rejection during ultrafiltration

To study the rejection of individual components, rejection factors of lactose and individual proteins were calculated. The results are summarised in Tables 6–7.

Lactose rejection was in the range of 2–22 % on average and depended on the membrane cut-off. The smaller pore size the higher lactose rejection (Fig. 7). Lactose rejection on the 500 nm cut-off membrane was 2.1 %, therefore lactose losses during MF are minimal when using the 500 nm membrane for pre-filtration. On the other hand, lactose rejection on the 100 nm and 5 kDa ultrafiltration membranes, which are intended mostly for protein concentration, was high. Considering the sieving effect, which is the main mechanism of separation during ultrafiltration, lactose should not be retained by these membranes; however, high rejection is probably caused by the formation of a polarisation layer acting as a secondary membrane and retaining lactose.

The average rejection of whey proteins (Table 7, Fig. 8) was calculated for 5 kDa, 100 nm and 500 nm membranes. The highest average protein rejection (95–98 %) was observed for the 5 kDa membrane, the 500 nm membrane protein rejection varied between 16 % and 26 % and that on the 100 nm membrane was in the range of 70–87 %. The 5 kDa membrane removes almost all present proteins and retentate can

Table 7. Average protein rejection on ceramic membranes

Protein	Protein size[a]/kDa	Rejection factor ± SD/%		
		Membrane cut-off		
		5 kDa	100 nm	500 nm
CMP	7	94.6 ± 7.6	70.2 ± 7.6	16.3 ± 2.3
α−La	14.2	96.7 ± 5.7	69.7 ± 1.5	16.3 ± 2.8
β−Lg B	18.4	97.5 ± 4.3	81.8 ± 2.7	17.8 ± 2.1
β−Lg A	18.4	96.3 ± 6.5	87.2 ± 3.1	26.3 ± 0.9

a) Maubois and Ollivier (1997); R_{CMP} – rejection of casein macropeptide, $R_{\alpha-\mathrm{La}}$ – rejection of α-lactalbumin, $R_{\beta-\mathrm{Lg\ A}}$ – rejection of β-lactoglobulin B, $R_{\beta-\mathrm{Lg\ A}}$ – rejection of β-lactoglobulin A, SD – standard deviation.

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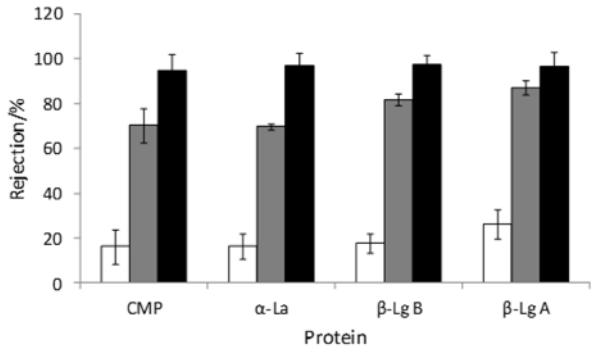

Fig. 8. Average protein rejection on ceramic membranes of various cut-offs: casein macropeptide (CMP), α-La (α-lactalbumin), β-Lg B (β-lactoglobulin B), β-Lg A (β-lactoglobulin A); □ – 500 nm, ■ – 100 nm, ■ – 5 kDa.

be further used in the production of protein isolates or concentrates.

Our results show a reasonable rejection of whey proteins in a single step separation in comparison with other results from literature. For example, Cheang and Zydney (2004) examined the use of a two-stage tangential flow filtration system for the purification of α-La (α-lactalbumin) and β-Lg (β-lactoglobulin) from whey protein isolate. Separation was achieved employing 100 kDa and 30 kDa membranes in series. α-La was obtained in the 90 % yield; however, the recovery of β-Lg was more challenging. Almécija et al. (2007) investigated the potential of membrane ultrafiltration for the fractionation of clarified whey. A 300 kDa tubular ceramic membrane was used in a continuous diafiltration mode, and the effect of pH was evaluated. The highest permeate yields for α-La and β-Lg of 56 % and 33 %, respectively, were obtained at pH 9 while bovine serum albumin (BSA), immunoglobulins IgG and lactoferrin (LF) were mostly retained at the working pH values. Muller et al. (1999) investigated the separation of α-La using different ultrafiltration modes of operation. They developed model equations for the continuous, discontinuous concentration or diafiltration modes (single or combined). They showed that continuous concentration up to a high volume reduction ratio (11–15) or combined continuous concentration- diafiltration helped to obtain a fraction with both enhanced purity and satisfactory yield of α-La, of up to 90 %, in the permeate.

Atra et al. (2005) found that the rejection of proteins increased when the pressure decreased and that it can reach 98 % at the pressure of 100 kPa. Typical initial protein content was 10–12 % and UF could increase it from 35 % up to 80 % (decreasing at the same time the content of lactose and some salts passing through the membrane to the permeate). Nevertheless, the rejection of proteins was higher at low pressures, so the best compromise between flux (30 L m^{-2} h^{-1}) and rejection (96 %) was found at an intermediate pressure of 300 kPa. When UF was followed by DF (diafiltration), the yield of the final spray-dried form was about 1.5 kg of 35 % protein in whey protein concentrate (WPC) per 100 kg of whey.

Conclusions

A wide range of tubular inorganic membranes with various pore sizes were tested for whey separation using tangential pilot plant filtration. Microfiltration using a 500 nm membrane was one of the fastest processes with high steady state fluxes (31–59 L m^{-2} h^{-1}) and high concentration factors achieved (1.9–17). Only 2 % losses of lactose were observed using this type of membrane. The 100 nm ultrafiltration membrane provided high steady state permeate fluxes (22–66 L m^{-2} h^{-1}) as well as high rejection of proteins (70–87 %). The steady state permeate fluxes on the 5 kDa membrane (12–19 L m^{-2} h^{-1}) are sufficient for industrial applications; the membrane also retained 95–98 % of proteins.

Lactose rejection on the 100 nm and 5 kDa ultrafiltration membranes were high, probably due to the formation of a polarisation layer acting as a secondary membrane.

The main problem was to restore the initial pure water flux by membrane cleaning. Therefore, pure water fluxes were very fluctuating. Further optimisation of the cleaning procedure is necessary.

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