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O. Akoum[a]; R. Chotard-Ghodsnia[a]; L. H. Ding[a]; M. Y. Jaffrin[a]

[a] Department of Biological Engineering, Technological University of Compiègne, Compiègne, France

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Ultrafiltration of Low-Heat and UHT Skim Milks with a Shear-Enhanced Vibrating Filtration System

O. Akoum, R. Chotard-Ghodsnia, L. H. Ding, and M. Y. Jaffrin*

Department of Biological Engineering, Technological University of Compiègne, Compiègne, France

ABSTRACT

A vibratory shear-enhanced filtration system (VSEP) was used for ultrafiltration of UHT skim milk and a skim milk reconstituted from low-heat powder, which has a similar protein content as fresh milk. Two polyethersulfone membranes of 10 and 50 kDa cutoff were used, respectively, for total protein concentration and for α-lactalbumin (α-LA) separation from β-lactoglobulin (β-LG). With the 50 kDa membrane, casein micelles were completely rejected after 40 min of filtration while α-LA transmission rate (ratio of permeate to retentate concentrations) remained between 22% and 28%. The β-LG transmission rate was around 1% and the stabilized permeate flux at initial concentration (Volume Reduction Ratio, VRR = 1) and a transmembrane pressure of 250 kPa was between 60 and 70 $L\,h^{-1}\,m^{-2}$. Permeate flux data on this membrane were found to be very close to those obtained under the same conditions

*Correspondence: M. Y. Jaffrin, UMR 6600, Department of Biological Engineering, Technological University of Compiègne, BP 20529, 60205 Compiègne, France; Fax: 33-3-44-20-48-13; E-mail: michel.jaffrin@utc.fr.

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with UHT skim milk until a VRR of 3. At higher concentrations, the reconstituted milk yielded higher permeate fluxes, $29\,L\,h^{-1}\,m^{-2}$ at a VRR of 6 vs. $15\,L\,h^{-1}\,m^{-2}$ for UHT milk. The 10 kDa membrane retained whey proteins completely and yielded higher fluxes—up to $82\,L\,h^{-1}\,m^{-2}$ at initial concentration and a transmembrane pressure (TMP) of 1500 kPa—than data reported for conventional ultrafiltration. The maximum VRR reached in concentration tests was 13 with a final flux of $8\,L\,h^{-1}\,m^{-2}$, corresponding to a maximum theoretical VRR by extrapolation to zero flux of 17. The critical shear stress at which a steady TMP could be maintained for a constant flux of $30\,L\,h^{-1}\,m^{-2}$ was found to be 13.7 Pa.

Key Words: Skim milk; Ultrafiltration; Shear-enhanced filtration; Filtration stability; Vibrating membrane.

INTRODUCTION

Ultrafiltration (UF) has been used extensively in the dairy industry for concentrating total proteins in the production of soft cheese,[1–4] for the recovery of soluble proteins from whey,[5,6] and for the standardization of milk protein concentration throughout the year in order to reduce enzyme consumption and labor.[7] More recently, it has been proposed for protein fractionation, separating α-lactalbumin (α-LA) from β-lactoglobulin (β-LG).[8,9] An excellent review of current and emerging applications of UF in the dairy industry may be found in Ref.[10]. The advantages of incorporating whey proteins to casein in the cheese-making industry were recently reviewed by Hinrichs.[11] Since the smallest whey protein, α-LA, has a molecular weight of 14.2 kDa vs. 18 kDa for β-LG or 36 kDa in dimer form, membranes with cutoff from 5 to 25 kDa are generally used for total protein concentration of whey protein recovery,[12] while a cut-off between 30 and 100 kDa is necessary for separating α-LA from β-LG.

We review briefly here the literature concerning these two applications. Clarke and Heath[13] have ultrafiltered skim milk using spiral-wound modules equipped with 5 kDa polysulfone membranes. They found that the permeate flux increased linearly with crossflow velocity but remained below $14\,L\,h^{-1}\,m^{-2}$ at 225 kPa and a velocity of $0.3\,m\,s^{-1}$. Makardij et al.,[14] who used a polysulfone membrane of 3.5 kDa, observed an important flux decline during the first 40 min and reported stabilized fluxes of $10\,L\,h^{-1}\,m^{-2}$ at 45°C and 200 kPa. They attributed this fouling, using atomic-force microscopy observations, to pore blockage. Ultrafiltration at 20 kDa followed by a diafiltration was carried out by Alvarez et al.[15] to produce concentrated milks with lower lactose-to-protein ratios. During a concentration test with a Pellicon cassette, their permeate flux dropped from $18\,L\,h^{-1}\,m^{-2}$ at initial concentration to $10\,L\,h^{-1}\,m^{-2}$ at a volume reduction ratio (VRR) of 3.

Membrane fouling during UF of whey using 20 kDa zirconium oxide membranes on carbon support (Carbosep, Techsep, Miribel, France) was investigated by Labbe et al.[16] While permeate fluxes were higher than for skim milk, they decayed significantly during the first hour of filtration. From infrared spectroscopy of deposits on the membrane, these authors attributed the fouling to protein–ZrO_2 interactions. Another investigation of membrane fouling in UF of whey was reported by Koutake et al.[17] using a 20 kDa polyacrilonitrile membrane. They concluded that, in UF of whey, the filtration resistance was mainly due to surface fouling while, in UF of skim milk, it was mainly caused by adsorption in the pores. They reported a maximum permeate flux of $14\,L\,h^{-1}\,m^{-2}$ at 30 kPa with skim milk at a temperature of 25°C.

Extraction of α-LA, which is used in infant formula preparation, by UF of whey protein concentrate was investigated by Lucas et al.[8] using Carbosep zirconium membranes of 10, 15, 50, and 150 kDa. These membranes were modified with a positively charged polyethyleneimine coating in order to increase the α-LA/β-LG separation selectivity, $S = Tr_{\alpha-LA}/Tr_{\beta-LG}$, where Tr is the transmission rate. This selectivity was increased from 3 for the unmodified 150 kDa membrane to 10 with the modified one at the expense of α-LA transmission rate, which dropped from 40% to 10%.

Because of the high fouling observed in UF of skim milk by these various investigators, resulting in low permeate fluxes, we think that it is appropriate to examine the performance of a vibration shear-enhanced filtration system for this application. In a recent study[18] we had obtained promising results in microfiltration (MF) at 0.1 μm and UF at 50 kDa of UHT skim milk using a VSEP laboratory pilot. The present study was performed using a skim milk reconstituted from low-heat powder,[19] which is more representative of fresh milk, together with UHT milk for comparison. In this reconstituted milk, whey proteins are not denatured as in UHT milk.

Our goal was to compare filtration data for this reconstituted milk in UF at 50 kDa with those for UHT milk using the same equipment and conditions in order to check whether the UHT skim milk could be a good model of fresh skim milk from a filtration point of view. Secondly, we wanted to investigate the VSEP performance in UF at 10 kDa for the purpose of total protein concentration with this reconstituted milk.

MATERIALS AND METHODS

Experimental Set-up and Operating Parameters

The filtration module depicted in Fig. 1 was a VSEP Series L (New Logic International, Emeryville CA). It was equipped with a single 13.5-cm outer radius (R_2), and a 4.7-cm inner radius (R_1) circular membrane with an effective area of 503 cm^2. The shaft supporting the membrane housing acts as a torsion spring that transmits the oscillations of a lower plate in the base, which is vibrated by an eccentric drive motor. As a result, the membrane and its housing oscillate azimuthally with a displacement amplitude "d", depending upon frequency, and has been checked[20] to be 32 mm on the outer rim at the resonant frequency of 60.75 Hz. This frequency can be adjusted by an electronic controller. The module was fed from a thermostated and stirred 10-L tank by a volumetric pump at a flow rate of 2 L min^{-1} in all tests.

The local membrane shear rate varies sinusoidally with time and proportionally to radius. On the basis of earlier experiments, we felt that the representative shear rate for the VSEP is the maximum with time averaged

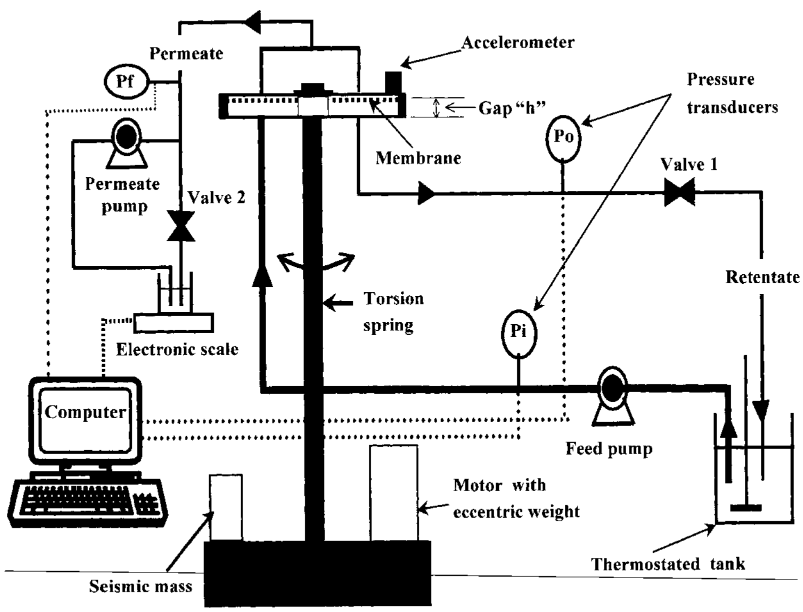

Figure 1. Schematic of experimental set-up.

over the membrane area. This mean shear rate was calculated in[20] to be

$$\bar{\gamma}_w = \frac{2d(R_2^3 - R_1^3)(\pi F)^{3/2}}{3R_2(R_2^2 - R_1^2)\nu^{1/2}} \tag{1}$$

where F is the oscillations frequency and ν the retentate kinematic viscosity. The permeate viscosity μ at 45°C necessary to compute the filtration resistance R_t was taken to be that of water, 0.6×10^{-3} Pa s. The milk viscosity was measured by a Rheostress Rheometer (HAAKE, Karlsruhe, Germany) with coaxial cylinders at 5000 s^{-1}, which was the highest shear rate possible. The kinematic and dynamic viscosities at 45°C are given as a function of volume reduction ratio in Table 1. The permeate was checked for casein micelles using a HACH turbidimeter (Loveland, CO) to ±1 NTU. It was collected into a beaker placed on an electronic scale (Sartorius B3100 P, Gottingen, Germany) connected to a computer in order to measure the permeate flux by calculating the derivative of weight signal and dividing it by membrane area. Inlet, outlet, and permeate pressures were measured by VALIDYNE DP 15 (Validyne Corp, Northridge, CA) pressure transducers in order to determine the transmembrane pressure (TMP) as mean of inlet and outlet pressures minus permeate one. The TMP was adjusted by a clamp (valve 1, Fig. 1) on the outlet tubing. In constant permeate flux experiment, valve 2 was shut and the permeate flow rate was regulated by a volumetric peristaltic pump (Masterflex 7518-12, Barrington, IL). The milk temperature was monitored in the tank by a Digitron platinum resistance thermometer (SIFAM Ltd, Torquay, Devon, UK). Tests were mostly carried out at initial concentration (VRR = 1) and at VRR = 1.8 or 2, which are the standard operating concentrations for this process in the dairy industry. But concentration tests up to VRR = 10 were also carried out for testing the limits of our system.

Table 1. Density, dynamic, and kinematic viscosities of reconstituted milk at T = 45°C as function of VRR.

	Powder milk at T = 45°C		
VRR	ρ (kg m^3)	μ (mPa s)	ν ($m^2 s^{-1}$)
1	1016.0	1.0	0.98×10^{-6}
2	1042.0	1.3	1.25×10^{-6}
3	1062.0	2.0	1.88×10^{-6}
4	1083.5	2.9	2.68×10^{-6}
5	1106.5	4.2	3.80×10^{-6}

The total filtration resistance R_t was calculated from permeate flux J, permeate viscosity μ, and TMP from

$$R_t = TMP/(\mu J) \tag{2}$$

since osmotic pressure can be regarded to be negligible in comparison with TMP.

Membranes and Cleaning Procedure

Membranes were permanently hydrophilic PES (polyethersulfone, Nadir Filtration, Wesbaden, Germany) of 10 and 50 kDa nominal cutoff. A new membrane was used in each test.

The circuit and module were rinsed with demineralized water, then washed with 5 L of Ultrasil P3-25F solution at 0.5% and 50°C for 15 min and rinsed again with demineralized water before and after each test.

Milk Characteristics and Sample Analysis

The milks used in the test were a commercial UHT skim milk (Printiligne, Paturages de France), sterilized during 3 sec at 140°C, and a milk reconstituted from low-heat powder milk, which will be referred to in the text as powder milk. This powder was prepared in the LRTL laboratory of INRA, Rennes, France,[19] by drying skim milk heated at 63°C for 15 sec to avoid whey protein denaturation. The reconstituted milk was obtained by dissolving 500 g of powder in 5 kg of pure water at 20°C while stirring for 20 min. Then the solution was placed in a tank thermostated at 45°C for 2 hr before starting the experiment.

Permeate and retentate samples collected during microfiltration or ultrafiltration were analyzed for α-LA and β-LG contents by reversed-phase high-pressure liquid chromatography with a Vydac C4 column (150 × 4.6 mm, 5 μm, 300 A°, Touzart et Matignon, France) according to the method described by Le Berre and Daufin.[21] Eluent A was composed of milli-Q water with 0.1% trifluoracetic acid and eluent B of 20% water and 80% acetonitrile with 0.096% trifluoracetic acid. The gradient applied (expressed in % of eluent B) was: 0–15 min: 48%–61%; 15–17 min: 61%–100%; 17–22 min: 100%; 22–24 min: 100%–48%; and 24–34 min: 48%. The flow-rate was 1 ml/min. The detection was carried out at 280 nm. The accuracy for concentration determination was estimated to be 2.5%. Since the determination of transmission rate requires two concentration measurements, its accuracy was taken to be 5%. A maximum error of 10% was assumed to account for other experimental errors during sampling. The minimum measurable concentration was 0.02 gl^{-1}. Chromatographic spectra of α-LA and β-LG for powder milk and UHT milk with the same solid contents are displayed in Fig. 2a and 2b, respectively. The α-LA an β-LG peaks are smaller for UHT milk than for powder milk, indicating lower whey-proteins concentrations for UHT milk, which could be explained by their denaturation during UHT process.

At initial concentration (VRR = 1), powder milk composition was: casein: 25 gl^{-1}, α-LA: 0.43 gl^{-1}, and β-LG: 3.00 gl^{-1}. UHT milk composition was: casein: 25.6 gl^{-1}, α-LA: 0.16 gl^{-1}, and β-LG: 0.2 gl^{-1}, confirming partial denaturation of whey proteins.

Initial pH was, in most cases, equal to 6.4 ± 0.1 and decreased by less than 0.2 throughout the experiment. Tests were carried out at a temperature of

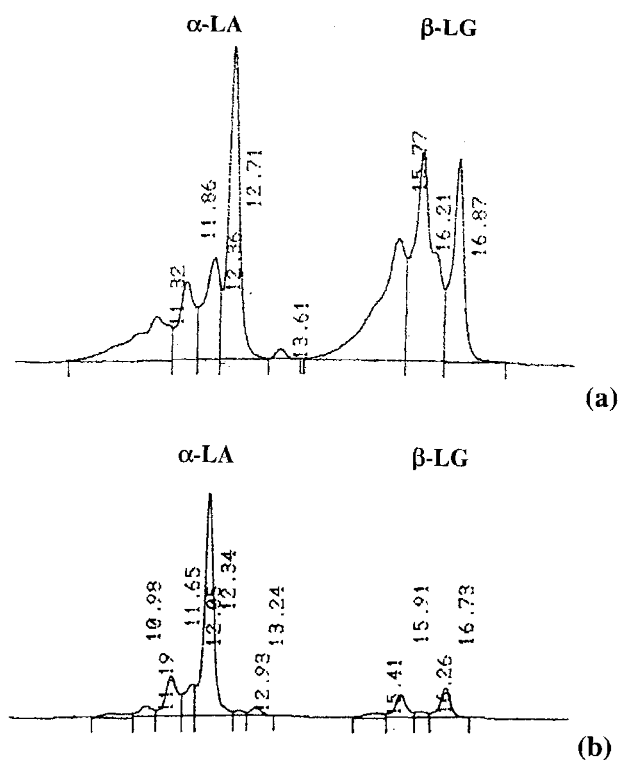

Figure 2. Chromatographic spectra of α-LA and β-LG for powder milk (a) and UHT milk (b).

45°C to avoid risks of protein denaturation[22] since our reconstituted milk was considered to be more heat sensitive than fresh milk.

RESULTS

Ultrafiltration at 50 kD. Comparison Between Powder and UHT Skim Milks

Variation of Permeate Flux and Protein Transmission with TMP

Figure 3 depicts the variation of permeate flux and turbidity with TMP at a frequency of 60.75 Hz and while recycling the permeate and retentate to retain initial concentration. The initial hydraulic permeability of membrane was 260 L h^{-1} m^{-2} bar^{-1}. The permeate flux rises rapidly until a TMP of 200 kPa, then more slowly up to 850 kPa. The permeate turbidity drops from 24 NTU at 150 kPa to zero when the TMP reached 550 kPa, which corresponded to an elapsed time of 40 min since the start of filtration. This permeate turbidity is proportional to casein concentration in the permeate. As TMP increases,

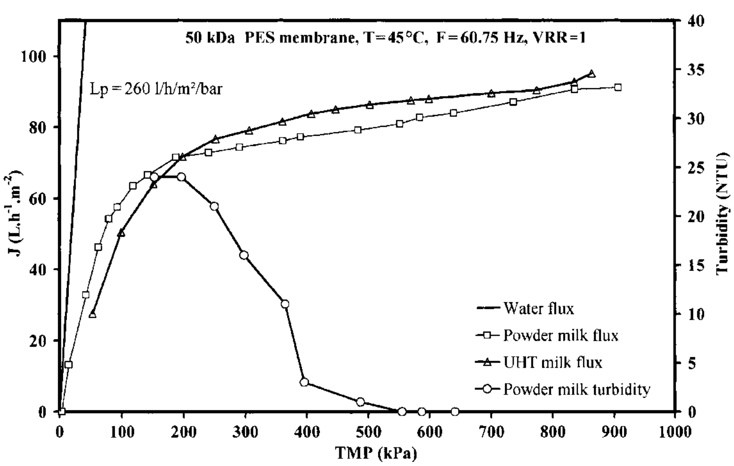

Figure 3. Variation of permeate flux and permeate turbidity for powder milk with TMP for a 50 kDa PES membrane at VRR = 1 and 60.75 Hz. Comparison of permeate flux with UHT milk.

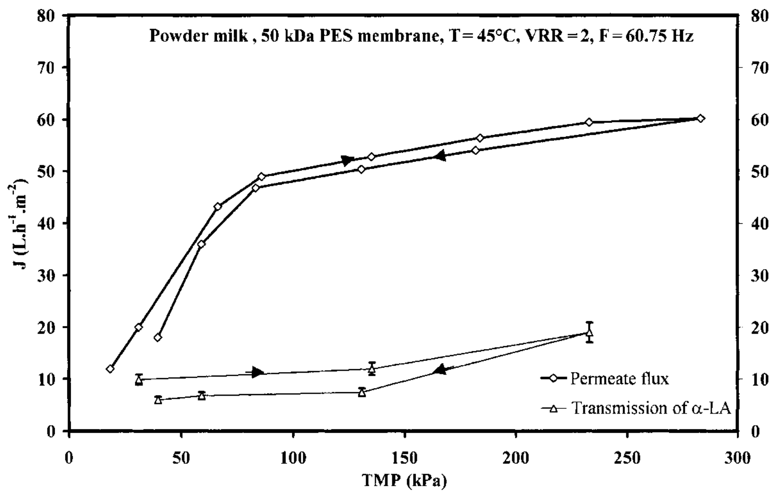

Figure 4. Variation of permeate flux and transmission of α-LA with TMP for powder milk at a VRR of 2 during a TMP cycle from 25 to 275 kPa and back to 30 kPa.

the casein layer on the membrane gets more and more packed and its permeability decreases. As a result, the permeate flux increases more slowly at high TMP and casein transmission decreases. The variation of permeate flux of UHT milk with TMP using the same membrane, shown in the same graph, is very close to that of powder milk, so that, from the point of view of filtration, UHT milk can be considered to behave similarly as powder milk.

The reversibility of flux and α-LA transmission during a TMP cycle at a VRR of 2 is displayed in Fig. 4. This cycle was started after 2 hr of filtration at 200 kPa, which was necessary to reach the desired concentration, and lasted another 2 hr. The flux reversibility is very good, indicating that fouling is minimal, perhaps because the maximum TMP of 280 kPa was not very high. However, α-LA transmission rate was reduced by 30%–35% when the TMP was decreased.

Variation of Permeate Flux, Membrane Displacement, and Protein Transmission with Frequency and Shear Rate

Figure 5 represents the variation with frequency of permeate flux for both types of milk at a VRR of 1.8 and a TMP of 400 kPa. Both membrane

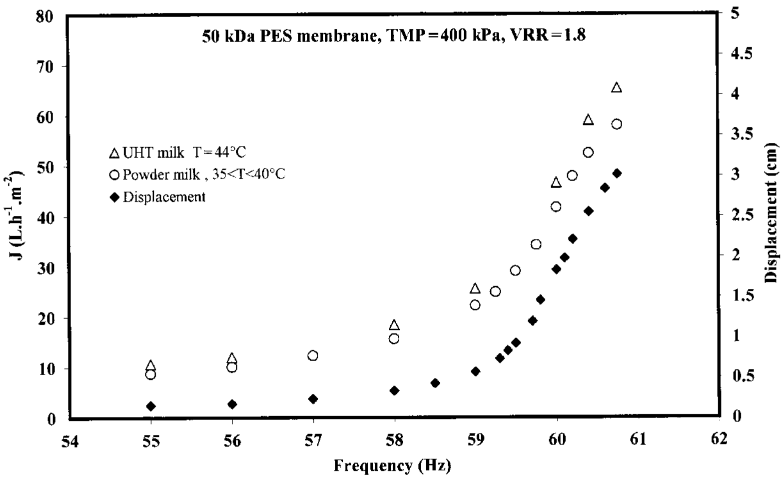

Figure 5. Variation of permeate flux with frequency for powder and UHT milks at a TMP of 400 kPa and a VRR of 1.8.

displacement and shear rate at membrane decrease when frequency is reduced. As a result, the permeate flux drops rapidly when frequency is reduced from 60.75 Hz to 59 Hz, and more slowly afterwards. The permeate flux for powder milk is slightly below that of UHT milk because of lower temperature due to insufficient temperature regulation in this test. This explanation of the difference is confirmed in Fig. 6, which represents the variation of total filtration resistance defined by Eq. (2) with mean membrane shear rate in log–log coordinates for the test of Fig. 5. Here the calculation of the resistance eliminates the effect of different temperatures in the two tests, and, as expected, the regression lines for the two milks are the same, which confirms that the filtration performances of the two milks are identical. The resistance varies as $\bar{\gamma}_w^{-0.56}$ which, since the TMP was constant, corresponds to a variation of permeate flux as $\bar{\gamma}_w^{0.56}$ according to Eq. (2).

The variation of permeate flux and temperature during a frequency cycle from 60.75 to 55 Hz and back to 60.75 Hz at a VRR of 1.8 and a TMP of 400 kPa is displayed in Fig. 7a. The duration of the test was 90 min. The apparent hysteresis for the permeate flux close to resonant frequency can be explained by the lower temperature when the frequency was raised, as confirmed by Fig. 7b, since the filtration resistance, which is insensitive to temperature changes, does not present any hysteresis. The α-LA transmission increases slightly from 22% to 28% as frequency was reduced to 59 Hz and

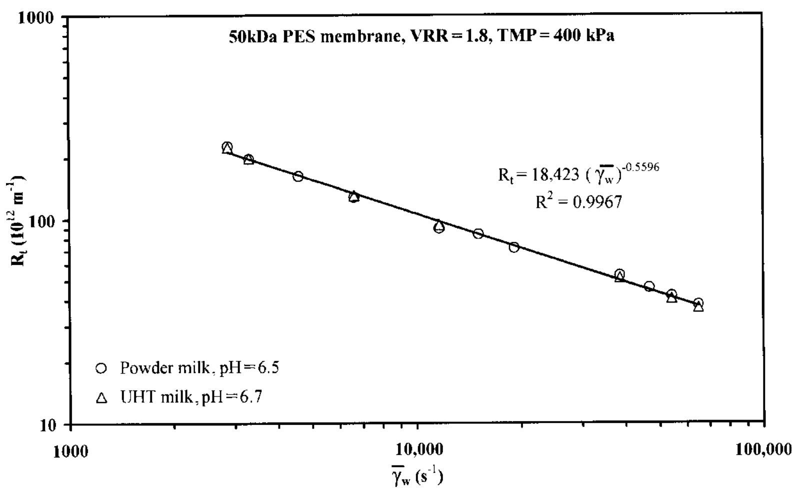

Figure 6. Variation of total filtration resistance, R_t, with mean membrane shear rate in log–log coordinates for powder and UHT milks at a TMP of 400 kPa and a VRR of 1.8.

dropped to 18% when resonant frequency was reached again at the end of the test. These variations are probably not significant in view of experimental errors, but the mean value of 22% agrees well with the value obtained from Ferry's sieving model. The β-LG rejection was nearly complete throughout the test, confirming that they were mostly dimers at this pH.

Variation of Permeate Flux During Concentration Tests

The comparison of concentration tests performed at resonant frequency with powder milk at 360 kPa and UHT milk at 400 kPa is depicted in Fig. 8. The permeate flux is significantly higher at the same concentration for powder milk, even though the TMP was slightly lower. The highest VRR value of 9 was also obtained with the powder milk vs. 5.8 for UHT milk. Both milks obey reasonably well the logarithmic dependence with concentration with a higher wall-limiting concentration for the powder milk (15 instead of 8.5), but with a smaller-mass transfer coefficient (32 vs. 41 $L\,h^{-1}\,m^{-2}$). The α-LA transmission in the permeate can be seen to increase with VRR, probably due to the increase in shear stress caused by the rise in viscosity while β-LG rejection remained nearly complete. Also shown is β-LG concentration in

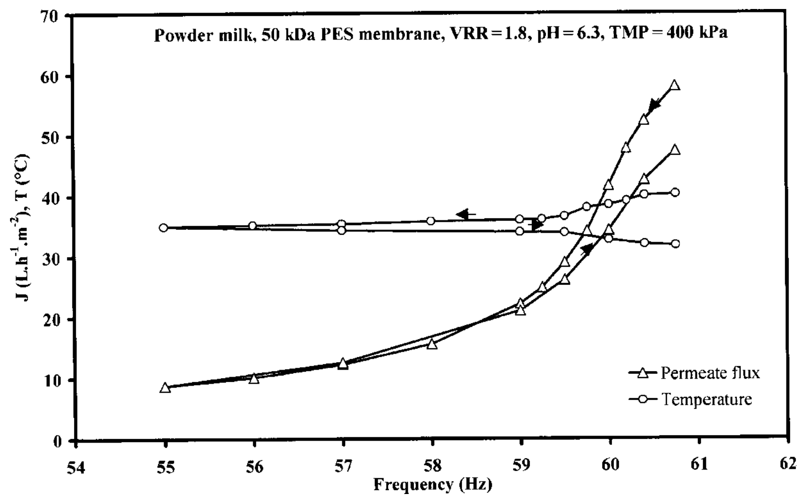

Figure 7a. Variation of permeate flux and temperature with frequency for powder milk at a TMP of 400 kPa and a VRR of 1.8.

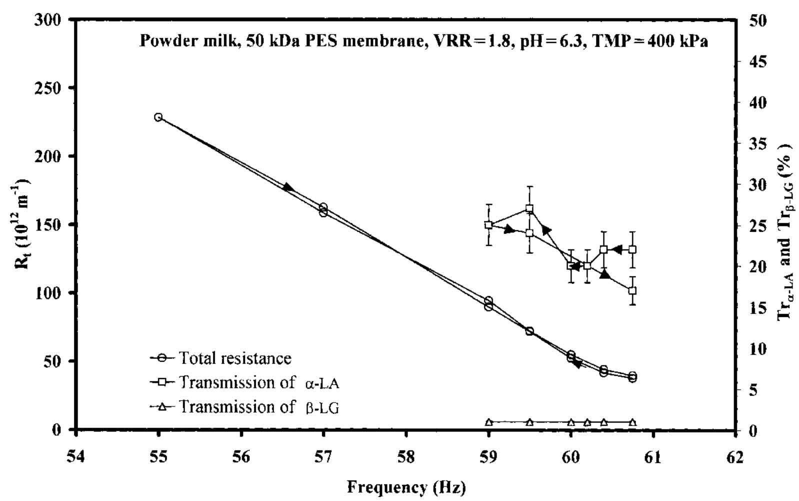

Figure 7b. Variation of filtration resistance and transmission of α-LA and β-LG with frequency for powder milk at a TMP of 400 kPa and a VRR of 1.8.

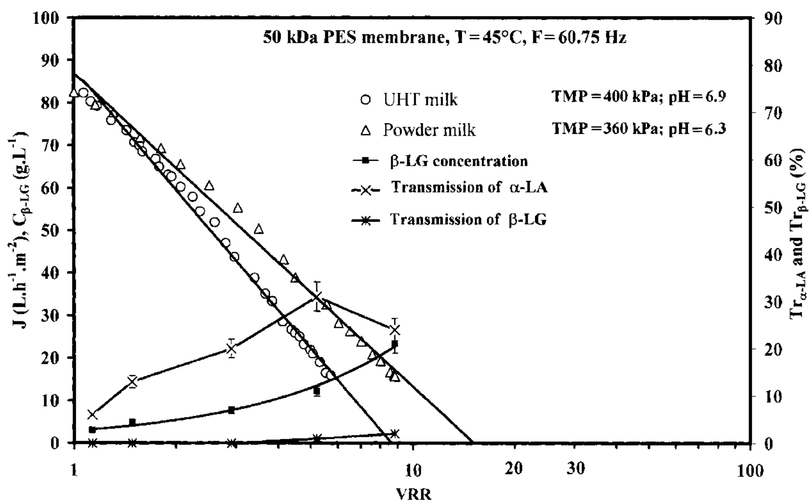

Figure 8. Variation of permeate flux with VRR for powder and UHT milks in semilog coordinates at 60.75 Hz using a PES 50 kDa membrane.

retentate, which increases with VRR up to 23 $g l^{-1}$ at VRR = 9, in accordance with mass conservation.

Ultrafiltration of Powder Milk with a PES 10 kD Membrane

Variation of Permeate Flux with TMP and Frequency

This variation is displayed in Fig. 9 for VRR = 1.8. The flux was measured first as a function of TMP at the maximum frequency of 60.75 Hz. Then, this experiment was repeated at various frequencies starting from 60.4 Hz down to 59.5 Hz at a temperature of 42°C. The permeate fluxes at TMP, less than about 350 kPa, are practically independent of frequency while, at larger pressure, the representative curves for each frequency separate. It can be observed that the maximum flux increases with frequency as well as the TMP at which this maximum occurs. At VRR = 1 (not shown here), the maximum permeate flux was 82 $L\,h^{-1}\,m^{-2}$ at 1500 kPa. The variation of maximum permeate flux with mean membrane shear rate at a VRR of 1.8 is represented in Fig. 10. This flux is found to vary with almost the same power of shear rate (0.52 instead of 0.56) as for the 50 kDa membrane.

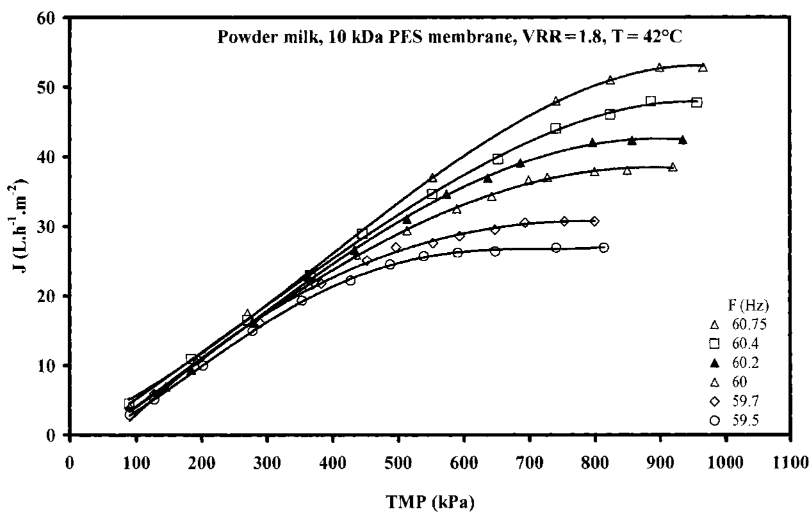

Figure 9. Variation of permeate flux with TMP for powder milk at various frequencies using a PES 10 kDa membrane at a VRR of 1.8.

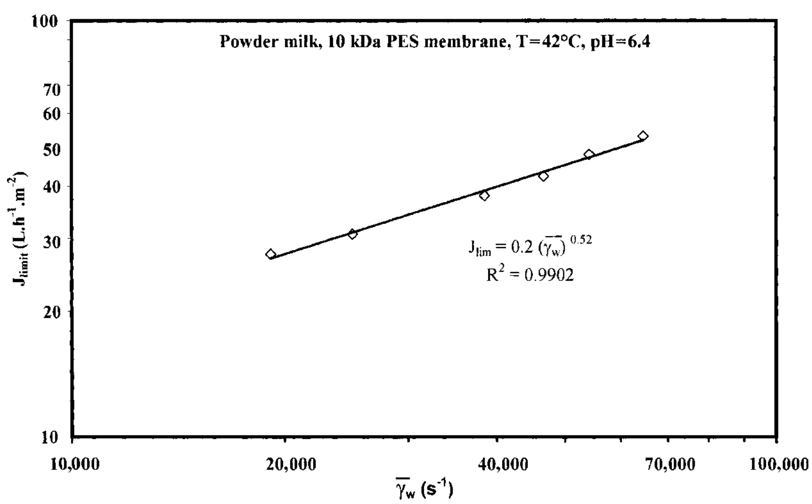

Figure 10. Variation of maximum permeate flux J_{limit} with mean shear rate at membrane for powder milk using a PES 10 kDa membrane at a VRR of 1.8.

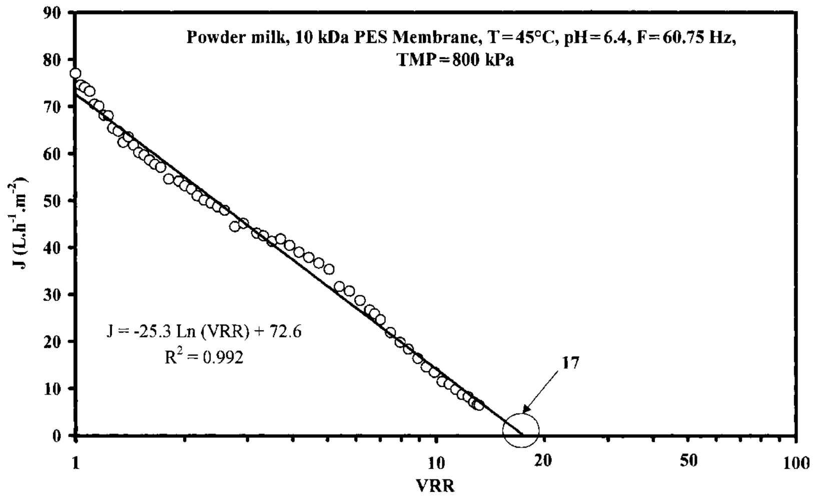

Figure 11. Variation of permeate flux with VRR for powder milk in semi-log coordinates at 60.75 Hz using a PES 10 kDa membrane.

Variation of Permeate Flux with Concentration

This variation is displayed in Fig. 11 for a test performed at 800 kPa. The maximum VRR reached in the test was 13, while the limiting concentration obtained by extrapolation to zero flux was 17—higher than what has been reported in the literature, at least for crossflow filtration. The permeate flux obeys the logarithmic dependence with VRR very well with a mass transfer coefficient of 25 $L\,h^{-1}\,m^{-2}$, slightly smaller than that found for the 50 kDa membrane (32 $L\,h^{-1}\,m^{-2}$).

Determination of Critical Shear Stress for Flux Stability

In order to determine the critical shear stress above which a steady filtration can be maintained (constant permeate flux and constant or slowly increasing TMP), we have decreased the oscillation frequency in small steps while maintaining a constant permeate flux and monitoring TMP. The results are illustrated in Fig. 12. It can be seen that the TMP, which remained constant as long as the frequency was above 59.7 Hz, started to increase rapidly with time when the frequency reached 59.3 Hz. Since the permeate flux could not be maintained completely constant due to a non truly volumetric pump, we

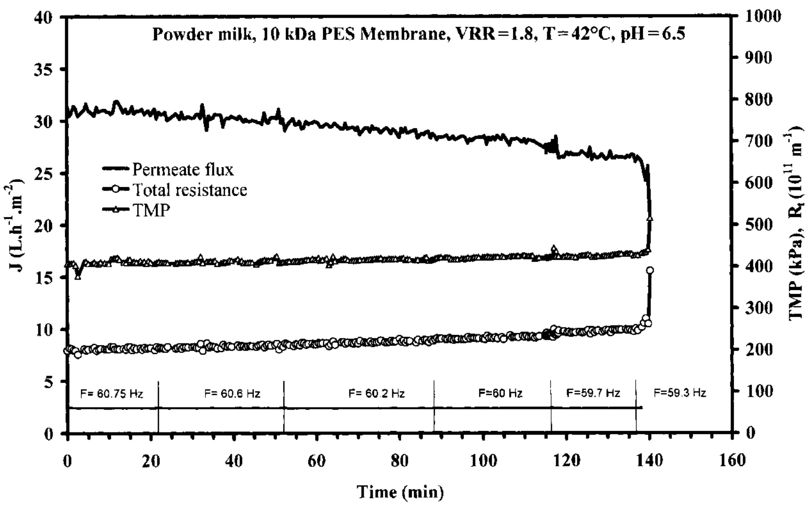

Figure 12. Determination of critical shear stress for powder milk by lowering frequency at constant flux, using a PES 10 kDa membrane at a VRR of 1.8.

have computed the total filtration resistance, R_t, which eliminates the artifact caused by the variation in permeate flux. The critical frequency can be seen from these data to be about 59.5 Hz, which corresponds to a critical shear stress of 13.7 Pa.

DISCUSSION AND CONCLUSION

The permeate fluxes obtained both with UHT and powder milks are significantly higher than those reported in the literature with conventional crossflow ultrafiltration, especially for the 10 kDa membrane, which has yielded a flux of $82\,L\,h^{-1}\,m^{-2}$ at initial concentration and $53\,L\,h^{-1}\,m^{-2}$ at a VRR of 1.8. Another interesting observation is that, with the 10 kDa membrane, the permeate flux continues to rise until a TMP of 1500 kPa, probably because the high shear rates retard the occurrence of concentration polarization. However, the comparison with other published data is not very precise because their authors generally use TMP below 400 kPa and do not indicate at which pressure the flux reaches its maximum. The VSEP has also proved to be able to yield very high-volume reduction ratios, 13 in our case, with a permeate flux of $8\,L\,h^{-1}\,m^{-2}$.

With the 10 kDa membrane, complete rejection of α-LA and β-LG was observed. With the 50 kDa membrane, we obtained an average α-LA transmission rate of 22% with a β-LG transmission rate of about 1%. This represents a selectivity of 22 and, therefore, a better performance than the selectivity of 10 with an α-LA transmission rate of 10% obtained by Lucas et al.[8] with modified inorganic membranes. Thus, this 50 kDa membrane may be a good choice for whey-protein fractionation.

Finally, up to a VRR of 2, the permeate fluxes obtained with the 50 kDa membrane under the same conditions of TMP and temperature were very close for both types of milks. Only when the VRR exceeded 4 did the powder milk show higher permeate fluxes than UHT milk. The same powder milk has been previously shown by Gésan-Guiziou et al.[23] to yield the same filtration performance as fresh skim milk during microfiltration at 0.1 μm. Therefore, we expect that UHT milk, which is readily available, could be substituted for fresh skim milk for investigating the effect of hydrodynamic parameters on permeate flux in ultrafiltration tests.

ACKNOWLEDGMENTS

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NOMENCLATURE

d	displacement at rim (m)
F	oscillation frequency (Hz)
J	permeate flux ($L\,h^{-1}\,m^{-2}$)
J_{limit}	maximum permeate flux ($L\,h^{-1}\,m^{-2}$)
L_p	membrane hydraulic permeability ($L\,h^{-1}\,m^{-2}$/bar)
R_1 (R_2)	inner (outer) membrane radius (m)
R_t	total filtration resistance (m^{-1})
S	selectivity
TMP	transmembrane pressure (Pa)
Tr	transmission rate
$\bar{\gamma}_w$	mean shear rate on membrane

μ	fluid dynamic viscosity (Pa s)
ν	fluid kinematic viscosity ($m^2 s^{-1}$)
τ_w	shear stress averaged over the membrane (Pa)

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