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Comparison of 3DTA and VSEP systems during the ultrafiltration of sweet whey

Cecilia Hodúr[a]*, Szabolcs Kertèsz[b], József Csanádi[c], Gábor Szabó[a]

[a]*Department of Mechanical and Process Engineering, University of Szeged, H-6725 Szeged, Moszkvai krt 5-7, Hungary*
Tel. +36-62546037; Fax +36-62546549; email: hodur@mk.u-szeged.hu
[b]*School of Environmental Sciences, University of Szeged, H-6720 Szeged, Dugonics tér 13, Hungary*
[c]*Department of Food Engineering, University of Szeged, H-6725 Szeged, Moszkvai krt 5-7, Hungary*

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ABSTRACT

Whey, a side-product of the cheese-making and casein industry, is a nutritious protein source. The nutritional, biological and functional properties of whey proteins make them attractive and explain why an active whey industry has been developing over the last 30 years. The preconcentration of whey at its production site is the major field of application of membrane separation. The high salt content of whey (8–20% dry matter) gives rise to numerous processing difficulties, a low lactose crystallization rate, and fouling in microfiltration (MF) and ultrafiltration (UF) performed to produce whey protein concentrates. In order to improve UF performance, it is advisable to limit fouling of the membranes by selecting an appropriate flux or shear stress ratio [1]. In this study, the performance of a vibratory shear-enhanced processing system (VSEP) for the concentration of cheese whey was assessed and compared with a classical, cross-flow, plate and frame membrane configuration system (3DTA) with the same membrane (i.e. a C30F UF regenerated cellulose UF membrane with a 30 kDa molecular mass cut-off). The temperature and pressure dependences of the permeate flux, the permeate flux reduction ratio, the resistances and the rejection values were investigated. Comparison of the two systems revealed a definite advantage for the VSEP system equipped with the same membrane and operated at the same pressure and temperature. The flux reduction ratio (J/J0) was 0.60 vs. 0.42, and the total resistances 2.87×10^{13} m^{-1} vs. 4.54×10^{13} m^{-1} for the VSEP and 3DTA system, respectively.

Keywords: VSEP; Ultrafiltration; Whey

1. Introduction

Ultrafiltration (UF) in the dairy industry has many applications, including the preconcentration of milk, the fractionation of whey, and micellar casein enrichment for cheese making [2]. Such concentrates are very rich in proteins and have a wide range of uses, such as dietary proteins for functional foods, ice-cream production and pharmaceuticals. UF is also a widely used separation technique for whey processing. Since whey is very rich in proteins, lactose, mineral salts, vitamins and free amino acids, it is worth processing by a membrane technique, but the process performance is diminished due to high osmotic pressure, high retentate viscosity, lactose crystallization and calcium phosphate precipitation. The high salt content of whey (8–20% dry matter) gives rise to

* Corresponding author.

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numerous processing difficulties, a low lactose crystallization rate, and fouling in microfiltration and UF performed for the manufacturing of whey protein concentrates.

The conventional cross-flow filtration of whey suffers from low protein transmission due to cake build-up. The gradual build-up of solute particles (i.e. proteins, micelles, etc.) near the membrane surface affects the permeate flux in various ways. It may lead to the formation of a dense cake or a gel layer. The solute particles may also block the membrane pores and thus alter its sieving characteristics and permeability. Fouling within the membrane pores causes changes in apparent pore size, pore size distribution and pore density of the membrane, which reduce the permeate flux [3,4]. These problems could be solved by circulating the retentate at high velocities and increasing the solution concentration. This not only provides a higher driving force for deposit formation, but also enhances the deposit removal due to the increased viscosity [5,6]. However, the drawback of these techniques is their high operating costs. In order to improve UF performance, it is advisable to limit the cake build-up and fouling of the membranes by selecting an appropriate shear stress ratio [1]. The vibratory shear-enhanced processing (VSEP) system permits the combination of high shear rates with a low trans-membrane pressure (TMP) since the membrane shear rate is created by the inertia of the fluid motion relative to the membrane, and not by the feed flow, which can be set very low.

The aim of this study is to assess and compare the performance of a VSEP system for the concentration of cheese whey with a classical, cross-flow, plate and frame membrane configuration system (3DTA), using the same membrane (i.e. C30F UF, a regenerated cellulose UF membrane with a 30 kDa molecular mass cut-off). The temperature and pressure dependences of the permeate flux, the permeate flux reduction ratio, the resistances and the rejection values were investigated.

2. Materials and methods

Cheese whey provided by Sole-Mizo Hungaria Ltd. Co. (Szeged) was pasteurized at 70°C to prevent pH decrease, frozen for conservation, and thawed at room temperature just prior to UF. The concentration was performed with a VSEP system and the classical, 3DTA cross-flow system. The vibratory shear-enhanced filter series L was a semi-pilot scale module, manufactured by New Logic International (USA).

The filter pack assembly consists of two steel pressure plates and the polypropylene clamshell, which is the housing containing the installation of the membrane. The filtration module consists of an annular membrane with an area of about 503 cm^2, an outer radius (R_1) of 13.5 cm and an inner radius (R_2) of 4.7 cm separated from the permeate by a support screen and a drainage cloth, in a circular housing placed at the top of a 4.5 m vertical shaft. This shaft is mounted on a seismic mass and acts as a torsion spring, which transmits the oscillations created by an eccentric drive motor. The membrane oscillates azimuthally in its own plane, with amplitude dependent upon the frequency of the drive motor. The shear rate that is created at the membrane is produced by the inertia of the fluid. The frequency of the oscillations is adjusted by an electronic controller with 0.01 Hz accuracy, while the resulting amplitude is recorded according to the pattern of appropriate black indicator marks situated on the front of the clamshell. The fluid enters and exits through symmetric radial slots, and circulates along concentric streamlines. On the top of the spring is the permeate tubing that removes the permeate created by the membrane filtration action at atmospheric pressure. The concentrated stream is returned through the 'process out' line, as shown in Fig. 1. The return flow passes through the flow limiter and the control valve, which allows fine adjustment of the outlet pressure. Inlet and outlet pressures were measured by Validyne analog gauges, in order to determine TMP as the mean of the inlet and outlet pressures, since the permeate was collected at atmospheric pressure.

During the VSEP process, the maximum ($\gamma_{w,\max}$) and mean ($\overline{\gamma}_w$) induced shear rates at the membrane surface were calculated via the following equations [7]:

$$\gamma_{w,\max} = 2^{1/2} d \left(\pi F\right)^{3/2} \eta^{-1/2} \tag{1}$$

$$\overline{\gamma}_w = \frac{2^{3/2}\left(R_2{}^3 - R_1{}^3\right)}{3\pi R_2\left(R_2{}^2 - R_1{}^2\right)} \tag{2}$$

where d is the peak to peak vibration amplitude at the periphery of the membrane, m; F is the vibration frequency, Hz, and η is the kinematic viscosity of the fluid, m^2 s^{-1}.

The classical membrane filtration experiments were performed on a Uwatech 3DTA laboratory membrane filter (Uwatech GmbH, Germany) with the use of a flat sheet standard C30F UF regenerated cellulose UF membrane with a 30 kDa molecular mass cut-off with a filtering surface area of 0.0156 m². The retentate stream was recycled back to the feed, the permeate stream was collected in a vessel and its flow rate was measured by using a volumetric cylinder and timer. The pressure applied was 0.4 MPa, the measurements were carried out at 25°C, the feed was thermostated, and the temperature was checked before and after the membrane filter. After each run, the membranes were washed with distilled water until the pure water flux reached the initial value measured after compaction (±2%).

The flux was determined via the equation:

$$J = \frac{dV}{d\tau}\frac{1}{A} = K_M\left(\Delta p - \Delta\pi\right) = \frac{\Delta p - \Delta\pi}{\eta\, R_T} \tag{3}$$

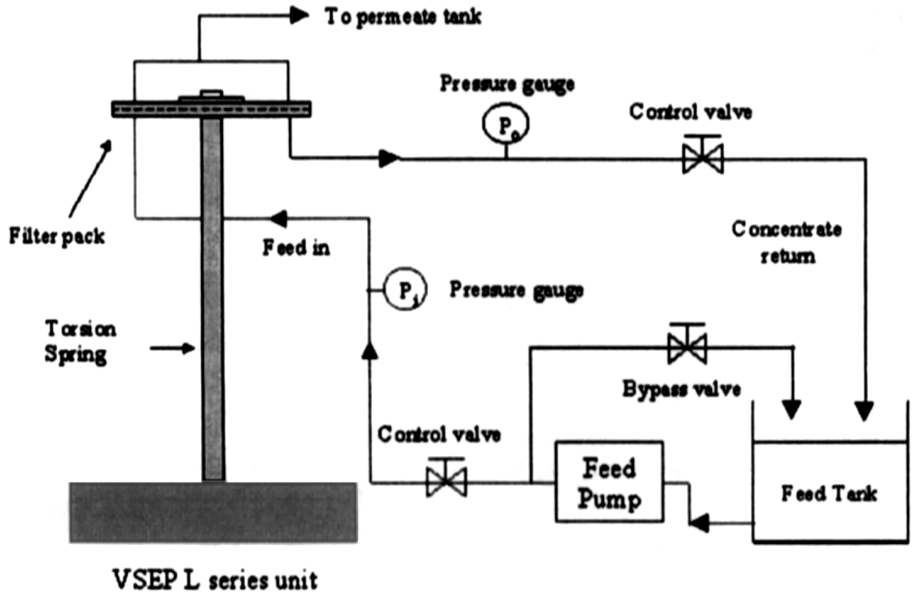

Fig. 1. The vibratory shear-enhanced process L series unit.

$$J = \frac{\Delta p}{\eta R_T} \tag{4}$$

where J is the flux, $m^3m^{-2}s^{-1}$; A is the surface area of the filter, m^2; V is the filtration volume, m^3; τ is time, s; K_M is the permeability coefficient, $m^3m^{-2}s^{-1}Pa^{-1}$; Δp is the pressure difference between the two sides of the membrane, Pa, and $\Delta\pi$ is the osmotic pressure, Pa.

The rate and extent of membrane fouling and its effect on permeate flux for any given system depend on various parameters:

- Specific interactions between the membrane surface and various fouling species
- Hydrodynamic forces exerted by the flowing process fluid
- Process parameters such as cross-flow velocity, TMP, feed concentration, pore size and temperature.

$$R_T = R_M + R_F + R_G \quad (m^{-1}) \tag{5}$$

The membrane resistance (R_M) was calculated as

$$R_M = \frac{\Delta p}{J_W \cdot \eta} \quad (m^{-1}) \tag{6}$$

where J_W is the flux of clear water, $m^3m^{-2}h^{-1}$, and η is the water viscosity at 25°C. The fouling resistance (R_F) of the membrane can be measured by washing the gel layer from the membrane. R_F and the resistance of the gel layer (R_G) can be calculated as

$$R_F = \frac{\Delta p}{J_W \cdot \eta} - R_M \quad (m^{-1}) \tag{7}$$

and

$$R_G = \frac{\Delta p}{J_W \cdot \eta} - R_M - R_F \quad (m^{-1}) \tag{8}$$

where η is the viscosity of the filtered solution at 25°C.

The selectivity of a membrane for a given solute was expressed by the average retention (R):

$$R\% = \left(1 - \frac{c}{c_0}\right) \cdot 100\% \tag{9}$$

where c is the average concentration of the solute in the permeate phase, m/m %, and c_0 is the concentration of the solute in the bulk solution, m/m %. The data were analysed by two-way analysis of variance (ANOVA).

The conductivity and the turbidity of the permeate and the concentrate were determined with a Consort C535 conductivity meter and an HACH00N turbidimeter, respectively. Crude protein, fat, total N and total soluble solid (TSS) content were measured with a Bentley IR 150 Instrument (Bentley Instruments, Chaska, MN, USA), expressed in m/m %.

3. Results

3.1. Fluxes

A comparison of the VSEP and 3DTA systems during concentration is illustrated in Fig. 2. Concentration tests were conducted at 25°C and 0.4 MPa with both modules. The membrane was the same C30UF/cut-off 30 kDa. It is clear that, during concentration, the VSEP system yields

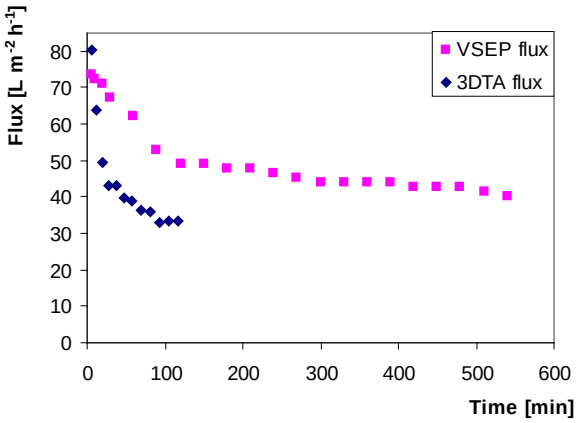

Fig. 2. Flux of permeate vs. time during concentration of whey with the VSEP and 3DTA systems (pressure: 0.4 MPa, temperature: 25°C).

a higher flux than the 3DTA system together with a lower permeate turbidity due to proteins. The initial flux values, the steady-state flux values, and the decline in the flux ratio differed considerably. The permeate flux decreased very rapidly in the first few seconds for the 3DTA system, followed by a slower fall, finally bevelling out at a lower value (33 L $m^{-2}h^{-1}$) than for the VSEP system (50 L $m^{-2}h^{-1}$). In VSEP system, not only yielded much higher permeate fluxes than cross-flow filtration, but also increased the lactose and ion rejections as their concentrations at the membrane were reduced, lowering their diffusive transfer through the membrane [8]. The initial fall was caused in both cases by concentration polarization, which is generally unavoidable in membrane processes. The gradual build-up of solute particles near the membrane surface affects the permeate flux in various ways. It may lead to the formation of a dense cake or a gel layer. The solute particles may also block the membrane pores and thus alter its sieving characteristics and permeability.

The difference was much more characteristic when the flux reduction ratio (J/J_0) was plotted vs. the volume reduction ratio (VRR) (Fig. 3). The difference between the curves started in the first minute of processing (when VRR reached 1.03); this initial difference between the fluxes was 2%. After the first 10 min, the gap had increased to 13%, after 20 min — to 31%, after 180 min — to 25%, and after 270 min — to 27%.

The initial rapid decreases in the flux (Fig. 2) and in VRR (Fig. 3) were greatly enhanced for the 3DTA system.

This was mainly due to concentration polarization. In the second stage, the flux continued to decline, but because of deposit formation. In the third stage, also called the quasi-steady-state period, the flux settled to a steady-state value [4]. The much milder VRR for the VSEP system means much milder concentration polarization and deposit (gel layer) formation due to the higher shear stress.

3.2. Retentions

With the C30F regenerated cellulose UF membrane with a 30 kDa molecular mass cut-off for both systems, the measured retentions of the systems differed. The retention was much higher (ca. 23%) in all cases for the VSEP system (Fig. 4).

When the concentration process was conducted at 45°C while TMP was increased stepwise from 2 bar to 8 bar, the fat, the protein, the lactose, the TSS and the total N in the permeate progressively decreased (Fig. 5), while the retentions of lactose, protein and total nitrogen progressively increased (Fig. 6). Similar phenomena were observed by Gresan-Guiziou et al. [9] and Al-Akoum et al. [10], who attributed these findings to an increase in TSS (i.e. lactose, fat, protein, etc.) retention resulting from the deposited protein layer on the membrane, which acts as a barrier to lactose and dissolved matter and thickens as TMP increases.

The contents of TSS and lactose in the permeate phase increased continuously during the 3DTA processing whereas the change during the VSEP procedure was just the opposite, progressively decreasing (Fig. 7). These

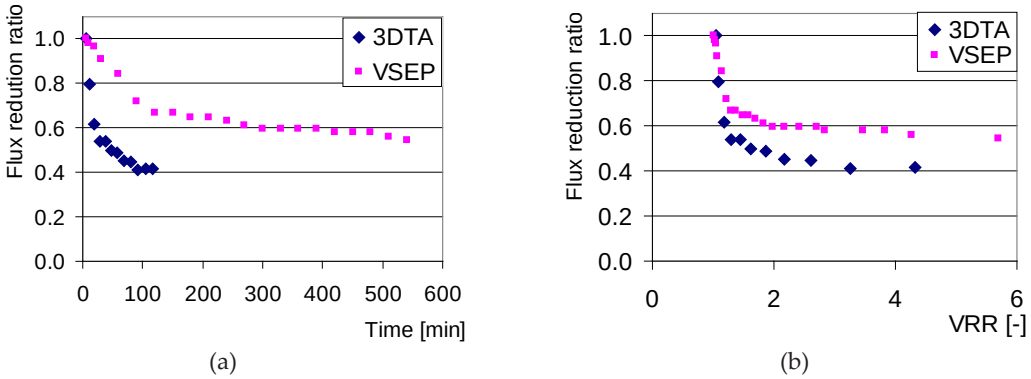

Fig. 3. Flux reduction ratio vs. time (a) and vs. volume reduction ratio (VRR) (b) during concentration of whey with the VSEP and 3DTA systems (pressure: 0.4 MPa, temperature: 25°C).

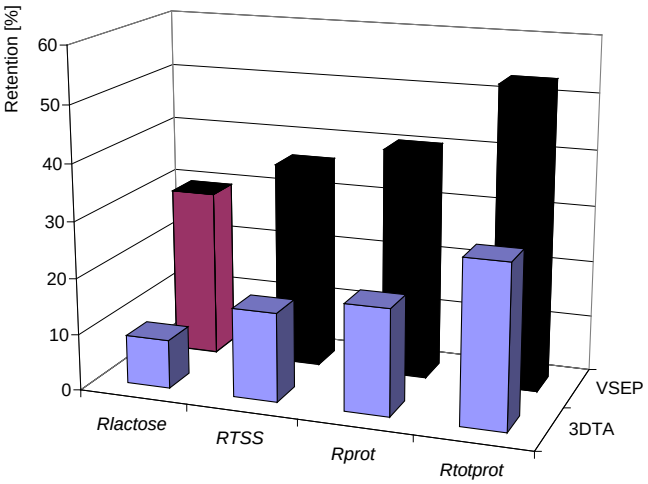

Fig. 4. Comparison of lactose, TSS, protein and total N retentions with the VSEP and 3DTA systems (pressure: 0.4 MPa, temperature: 25°C).

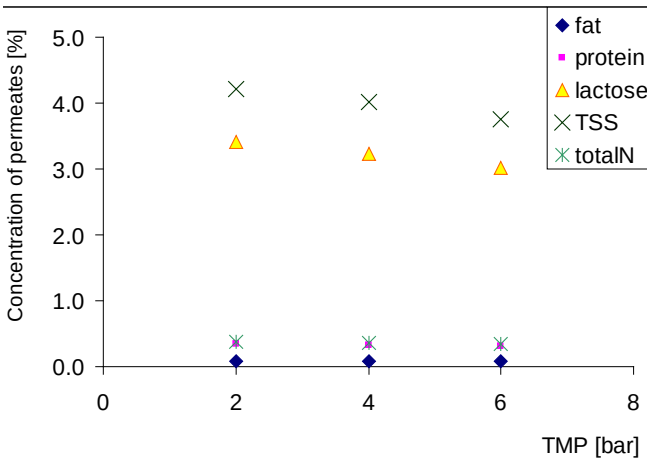

Fig. 5. Effects of the transmembrane pressure (TMP) on the compounds in the permeate.

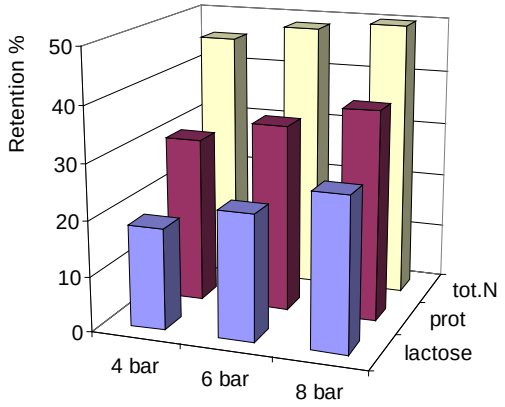

Fig. 6. Comparison of lactose, protein and total N retention for the VSEP system at different transmembrane pressures.

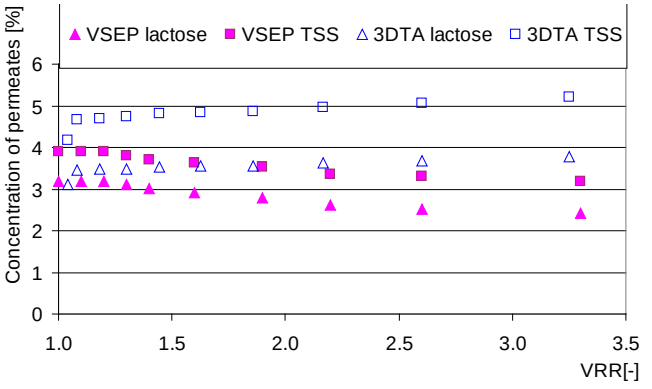

Fig. 7. Changes in lactose and TSS content of permeate during concentration of cheese whey with the VSEP and 3DTA systems.

phenomena could also be explained by the protein layer that developed on the top of the membrane in the 3DTA method; for the VSEP system, the data rather show that the solute particles may block the membrane pores thereby altering its sieving characteristics and the pore distribution.

This is confirmed in Fig. 8, which depicts the changes in lactose and TSS retention with time.

3.3. Resistances

The total resistance of membrane filtration is made up of the membrane resistance (R_M) itself and the resistances of the membrane transport phenomena, i.e. fouling and concentration polarization/gel layer formation. Gel layer resistance (R_G) refers to the concentration polarization, the formation of a gel layer on the surface of the membrane. This leads to blockage of the membrane, thereby reducing its throughput or flux.

Fouling within the membrane pores causes changes in the apparent pore size, the pore size distribution and the pore density of the membrane, which reduce the permeate flux [4]. During cheese whey ultrafiltration with the 3DTA system, R_G is higher than R_F [5]. The concentration polarization can be minimized by adequate selection of TMP or/and the whey feed tangential velocity, i.e. the shear stress. When the VSEP system was used for the concentration of cheese whey, the total resistance was reduced as low as 63% and R_F was larger than R_G; the measured R_G was 31.7% and R_F was 133% of that measured with the 3DTA system.

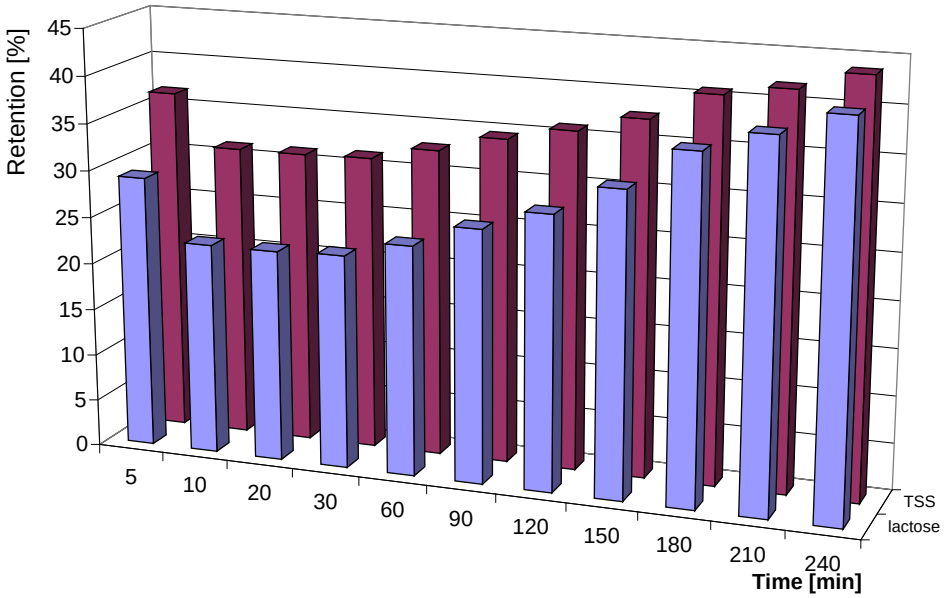

Fig. 8. Changes in lactose and TSS retention with time during concentration of cheese whey by VSEP.

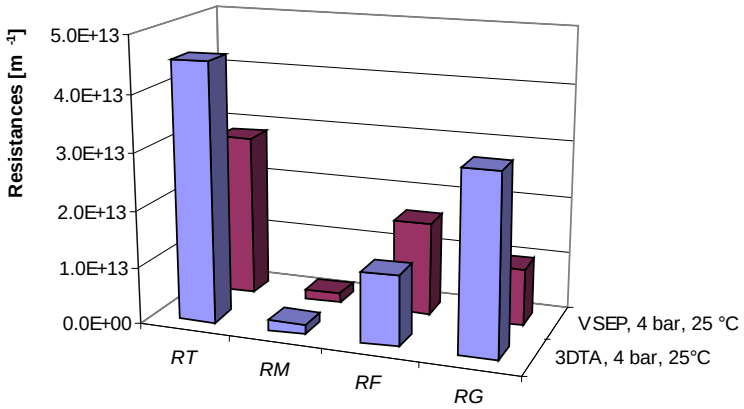

Fig. 9. Resistances in VSEP and 3DTA systems during concentration of cheese whey.

4. Discussion

The performance of the 30 kDa ultrafiltration membrane (C30F) was investigated during the processing of whey protein concentrate solution. With the 3DTA system, the membrane suffered significant fouling and the permeate flux was reduced by up to 40%. The VSEP system underwent a milder reduction in flux: 55%, due to the higher shear rate.

The permeate flux in the VSEP system is mostly controlled by the vibration frequency, and not by the inlet flow-rate [7]. Using the same laboratory pilot as ours, Takata et al. [11] observed a 50% rise in permeate flux during the UF of humic substances with a 100 kDa membrane when the displacement was increased from almost 0 to 2.5 cm at 60 Hz. For the same displacement increase, our data for the UF of cheese whey revealed a 33% flux increase for a 30 kDa membrane. As the test fluids and the membranes were different, we consider that our data are coherent with those of Takata et al. The total resistance was lower with the VSEP system, and the proportions of R_G and R_F also differed. R_G was much lower than in the 3DTA system, and lower than R_F (Fig. 9). High concentration polarization increases the fouling, which is not reversible by modification of the process parameters. A comparison of the data measured with the two systems demonstrated a definite advantage for the VSEP system equipped with the same membrane and operated at the same pressure and temperature. The VSEP system yielded a permeate protein retention of 99.7 NTU vs. 74.5 NTU

for the 3DTA system, together with a higher flux: 64 L $m^{-2} h^{-1}$ vs. 44.2 L $m^{-2} h^{-1}$. The flux reduction ratio (J/J_0) was 0.60 vs. 0.42, and the total resistance 2.87×10^{13} m^{-1} vs. 4.54×10^{13} m^{-1} for the VSEP and 3DTA system, respectively.

5. Conclusions

Our data show that the VSEP system outperforms the conventional 3DTA system in UF in terms of both permeate flux and permeate turbidity reduction due to reduced protein transmission through the membrane. The higher permeate flux of the VSEP system stems from its higher membrane shear rate, which allows increasing retention of protein and lactose with TMP. A rather unexpected result was observed during the concentration: the retention rate of lactose and TSS increased with the VSEP system on reduction of their concentration at the membrane, while the retention ratio decreased for the 3DTA system. In view of its good performances and lower energy consumption, we conclude that the VSEP system could be a viable alternative for the concentration of cheese whey.

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