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Flux and energy requirement during ultrafiltration of a complex industrial process stream

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ABSTRACT

Membrane filtration is a separation process already used in many industrial applications. However, optimization of the design and operating conditions is necessary to lower the investment and operating costs. To achieve this for industrial (multi-component) process streams, a combination of experiments and calculations is needed. The permeate flux and energy requirement are important design parameters for ultrafiltration plants. The influence of the operating conditions on these factors should therefore be investigated. In this work, bench-scale filtration experiments were performed on kraft black liquor using a ceramic membrane. The experimental data were used to calculate the average flux and energy requirement for different transmembrane pressures and cross-flow velocities. The optimal flux at different inlet cross-flow velocities was found to vary depending on feed concentration and transmembrane pressure.

Keywords: Ultrafiltration; Kraft black liquor; Lignin; Ceramic membrane; Operating conditions

1. Introduction

Separation accounts for 60 to 80% of the process cost of most mature chemical processes [1]. Development of cost-efficient separation processes is therefore of the utmost importance. Membrane processes are already used in many applications such as the treatment of food and beverages, electrodeposition paint and oily wastewater, and the desalination of water. The potential for membrane processes is enormous as many more industries could benefit from this technology. However, to ensure that membrane filtration gains its well-earned place in industrial processes, investment and operating costs must be reduced. To achieve this, the design and operating conditions must be optimized.

Today, the design and optimization of ultrafiltration (UF) plants is based on extensive experimental work. Membrane manufacturers and engineering companies specializing in membrane applications use experimental data and rely on experience and rules of thumb to identify suitable plant configuration and operating conditions. Membrane filtration models that consider the variation in transmembrane pressure, cross-flow velocity and concentration along the flow channel when predicting membrane performance in full-scale modules usually deal with well-defined solutions (model solutions). In most industrial applications, however, the solutions treated are complex mixtures of substances. For such mixtures a complete description would require an unrealistic amount of information since each solute would have to be defined separately [2]. By using a previously developed method [3–5] combining experiments and calculations, the influence of operating parameters can be evaluated and optimal conditions can be found to maximize the flux and minimize the energy requirement.

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The aim of this work was to study the influence of transmembrane pressure (TMP) and cross-flow velocity on membrane performance during UF of kraft black liquor. A ceramic membrane with a cut-off of 15 kDa was used in the investigation. A small number of experiments on bench scale were used to estimate the average flux and energy requirement for a unit with two modules in series.

2. Experiments

2.1. Material

Experiments were performed with hardwood black liquor from a Swedish kraft pulp mill. The black liquor was withdrawn from the evaporation unit. The total solids (TS) content of the evaporated black liquor (EBL) was 31% and the pH was 13–14. The liquor was used without adjustment of pH or prefiltration.

2.2. Equipment and membranes

A parametric study was performed in a bench-scale unit equipped with a K01 membrane module and a ceramic Kerasep membrane from Novasep, France, with a cut-off of 15 kDa. The membrane was made of Al_2O_3-TiO_2, and had seven parallel flow channels, each with a diameter of 6 mm. The total area of the membrane was 0.155 m^2.

The experimental set-up consisted of the membrane module, a feed tank and a centrifugal pump, as shown in Fig. 1. The TMP was controlled by the manual valves on the retentate line and the permeate line, and the cross-flow velocity by a frequency converter (Lust CDA3000, Lust Antriebstechnik, Germany) connected to the pump (NB32/25-20, ABS Pump Production, Sweden).

Pressure was measured with pressure transmitters (dTrans p02 and dTrans p30, JUMO Mätoch Reglerteknik, Sweden). The analogue signals from the pressure transmitters were transformed with an analogue-to-digital converter (Intab AAC-2, Intab Interface-Teknik, Sweden) before being transferred to a PC. The TMP was calculated as:

$$TMP = \frac{P_1 + P_2}{2} - P_3 \quad (1)$$

The frictional pressure drop in the membrane (length 1.2 m) was defined as the difference between the inlet and outlet pressures, $P_1 - P_2$.

Two 200-L feed tanks were connected to the plant. One was used as the feed tank during the experiments and the other was used when cleaning the membrane. The feed tank was heated with steam which condensed inside a coil

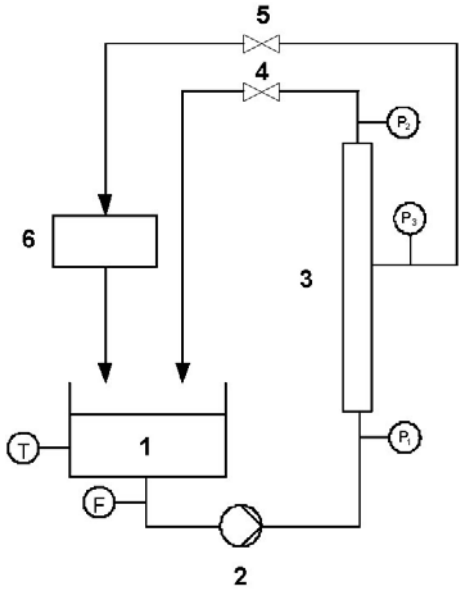

Fig. 1. Schematic illustration of the equipment used in the UF experiments. 1 feed tank, 2 pump, 3 membrane module, 4 retentate valve, 5 permeate valve, 6 balance. T: temperature regulator, F: flow meter, P_1, P_2, P_3: pressure transmitters.

in the feed tank. When the feed solution had reached the desired temperature this was maintained by a regulated electrical heater inside the tank.

The flux was determined by measuring the permeate flow with an electronic balance (PL6001-S, Mettler-Toledo, Switzerland) connected to the PC. The data were recorded and analysed with LabVIEW 6.0 software (National Instruments, Austin, TX, USA).

2.3. Experimental procedure

The membrane was cleaned at 50 kPa with the alkaline cleaning agent Ultrasil 11 (Henkel Chemicals, UK) for 60 min at a concentration of 0.25% and a temperature of 50°C. The system was thoroughly rinsed with deionized water. The pure water flux was measured at 50°C and 50 kPa.

The parametric study was performed at 90°C and three different concentrations, as shown in Table 1. The black liquor was circulated in the plant, while being heated to the temperature required for the experiment (90°C). During the heating period the permeate valve was closed. When the solution had reached 90°C the permeate valve was opened and the retentate and permeate were recirculated at 50 kPa for 16 h. To minimize the risk of cake formation at the membrane surface during the parametric study, the experiment was begun at the highest cross-flow velocity (6 m/s) and lowest pressure. The TMP at the inlet was increased step-wise from 100 to

400 kPa. The TMP was then decreased to 100 kPa and the cross-flow velocity reduced to 4 m/s. The TMP was increased step-wise again. The same procedure was repeated at 2 m/s. For the higher TS levels and cross-flow velocities, the frictional pressure drop was so high that it was not possible to achieve a TMP of 100 kPa and the parametric study was therefore started at a higher TMP (see Table 1). Steady-state was reached almost immediately after alteration of the operating conditions. Measurements were carried out for 30 min under each set of experimental conditions. Both retentate and permeate were returned to the feed tank during the experiment.

The concentration was increased by withdrawal of permeate and the liquor was circulated in the plant at the new concentration for at least 16 h before the parametric study. As the temperature of the black liquor was close to its boiling point, evaporation from the tank must be kept to a minimum. This was achieved by using a sealed lid with only a small hole for a temperature probe. However, in spite of these precautions part of the increase of concentration is attributed to evaporation of liquor during the experiment.

The membrane was not cleaned during the experiment.

2.4. Analysis

The TS content was determined by drying duplicate weighed samples at 105 °C and determining the weight of the residue. The ash content was measured by heating the residue from the TS measurement to 575 °C, maintaining the sample at this temperature for 24 h, and then weighing the sample. The ash content is the ratio between the weight of residue after and before heating to 575 °C.

The lignin concentration was measured by light absorption at a wavelength of 280 nm. The samples were diluted with 0.1 M NaOH and the UV light absorption was measured using a Shimadzu UV-160 spectrophotometer (Shimadzu, Japan). An absorption constant for hardwood lignin of 21.2 L/(g cm) was used.

Table 1
Conditions used in the parametric study

Total solids (%)	Cross-flow velocity (m/s)	TMP at the inlet (kPa)
31	2	100; 200; 300; 400
	4	100; 200; 300; 400
	6	100; 200; 300; 400
35	2	100; 200; 300; 400
	4	100; 200; 300; 400
	6	150; 200; 300; 400
44	2	100; 200; 300; 400
	4	150; 200; 300; 400
	6	200; 300; 400

The hemicellulose content was analysed using high-performance anion-exchange chromatography coupled with amperometric detection using an electrochemical detector (Dionex, Sunnyvale, CA, USA) after acid hydrolysis of the sample. Hemicelluloses were hydrolysed by adding 72% (w/w) H_2SO_4 to the sample to pH <1. The acid hydrolysate was autoclaved at 121 °C for 60 min. The sample was filtered through a 0.2 µm filter prior to analysis. The amount of monomeric sugar in the black liquors studied was low. The sugars detected in the liquor are therefore assumed to originate from hemicelluloses.

The viscosity was measured at 90 °C using a StressTech rheometer (ReoLogica, Sweden) with a 25 mm concentric cylinder. Measurements were performed in the range 10–100 s^{-1}.

The density was determined by weighing samples in measuring cylinders at 90 °C. An analytical balance, accurate to ±0.1 mg, was used.

3. Results and discussion

3.1. Parametric study

A parametric study was performed at three concentrations of black liquor. The characteristics of the three liquors are given in Table 2. The density of the liquors was around 1200 kg/m^3 at 90 °C. The liquor with TS 44% showed non-Newtonian behaviour and the viscosity is thus given at a constant shear rate.

The flux measured in the parametric study is shown in Fig. 2. The flux decreases and the frictional pressure drop increases with increasing concentration. The flux increases with increasing cross-flow velocity and TMP for all three concentrations.

The highest inlet TMP in the parametric study was 400 kPa. Hence, the maximum average transmembrane pressure decreases as the frictional pressure drop increases with concentration and cross-flow velocity.

3.2. Variation of pressure, velocity and concentration along the feed flow channel

In full-scale installations with ceramic membranes, the modules are usually connected two in series with a 180°

Table 2
Characteristics of the three liquors used in the parametric study

Liquor	TS (%)	Ash (% of TS)	Lignin (g/l)	Hemi-celluloses (g/l)	Viscosity[a] (mPa s)
1	31	50	138	14	2.2
2	35	48	155	18	3.3
3	44	47	193	30	12.6

[a]At 90 °C and shear rate 45 s^{-1}.

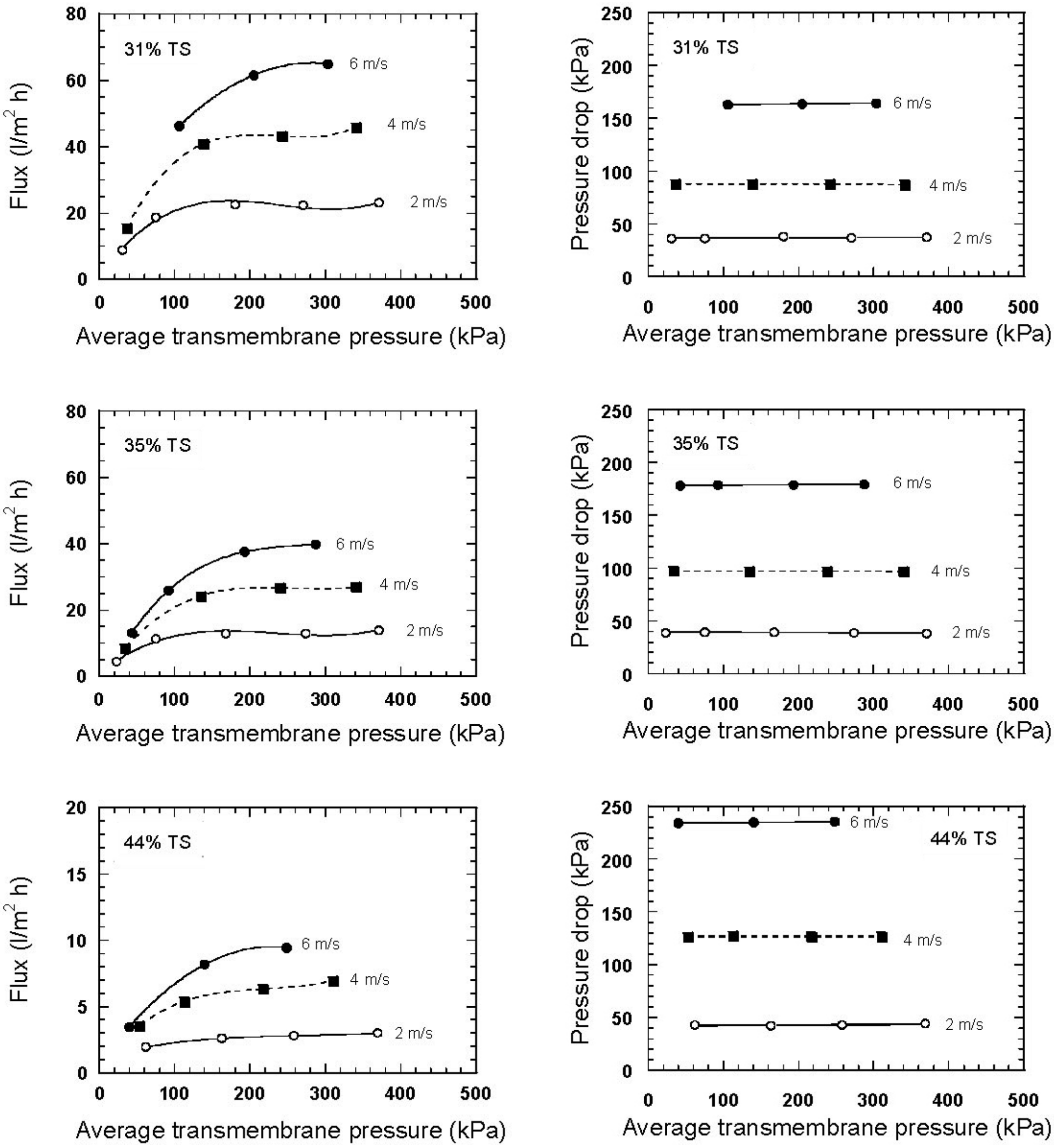

Fig. 2. Influence of TMP and cross-flow velocity on flux (left) and frictional pressure drop (right) during ultrafiltration of hardwood black liquor with three different TS contents. Temperature was 90°C during all experiments. Symbols represent experimental data and lines second and third-degree polynomial (flux) and linear (pressure drop) fits to experimental data.

bend, as shown in Fig. 3. The flux for two membrane elements connected with a 180° bend was therefore predicted with the simulation tool. The pressure drop in the bend was calculated using the correlation:

$$\Delta p_f = \xi \cdot \frac{\rho \cdot v^2}{2} \quad (2)$$

where ξ is the friction loss coefficient for the bend, ρ is the density and v is the cross-flow velocity. A resistance coefficient of 0.35 was used. This is valid for 2-inch flanged or welded regular bends according to Murdock [6].

A calculation tool [3–5] was used to simulate variations in flux and operating parameters along the feed flow channel. The concentration, cross-flow velocity and transmembrane pressure along the feed flow channel in the unit with two modules in series are shown in Fig. 4. The retention of TS was assumed to be 100% to obtain the maximum possible TS. In reality, the increase in the TS content is markedly lower, as the cooking chemicals making up about half of the solids in black liquor (NaOH and Na_2S), pass almost freely through the membrane [7].

The ratio between the permeate flow and the feed flow is small. Hence, the difference in concentration and cross-flow velocity between inlet and outlet is marginal and the

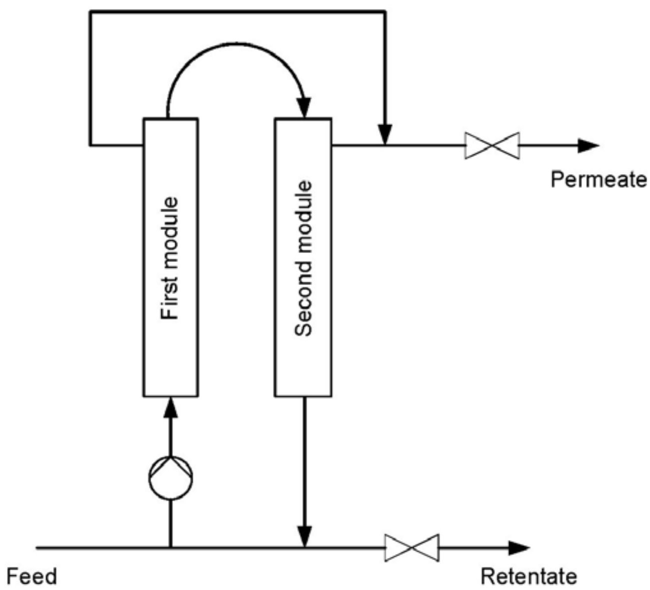

Fig. 3. Unit with two membrane modules in series.

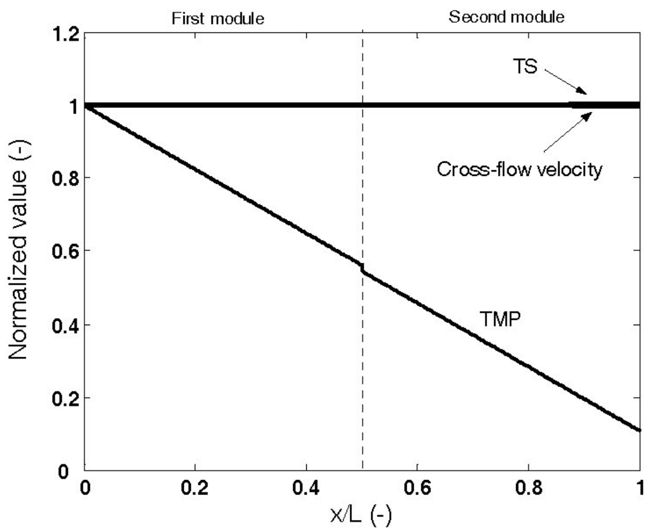

Fig. 4. Predicted TS content, cross-flow velocity and transmembrane pressure, along the flow channel at 90°C. Values are normalized to the values at the inlet of the module: TMP, 200 kPa; cross-flow velocity, 4 m/s; TS, 31%.

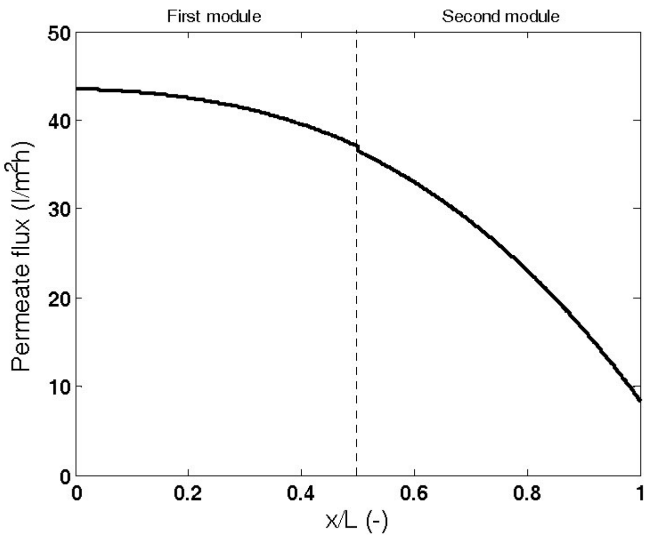

Fig. 5. Predicted permeate flux along the flow channel. Same basic data as in Fig. 4 were used for the simulations.

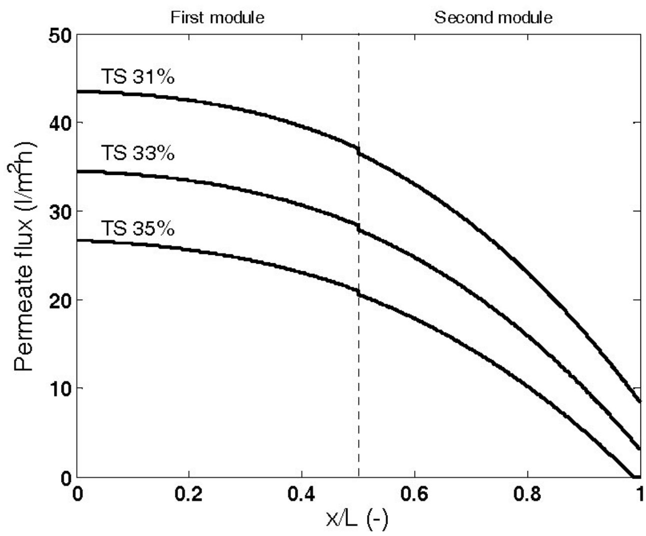

Fig. 6. Predicted permeate flux along the flow channel for three concentrations of black liquor at 90°C. TMP, 200 kPa; cross-flow velocity, 4 m/s.

flux decrease along the flow channel is therefore attributed to the frictional pressure drop reducing the transmembrane pressure. The predicted flux along the membrane is shown in Fig. 5. The average flux in the first module is 41 L/m^{2}h and in the second 25 L/m^{2}h.

The flux may even be zero at the end of the second module, as shown in Fig. 6. This means that there is no net driving force (NDF) towards the end of the second module. The liquor with 44% TS content can not be processed in a unit with two modules in series at the cross-flow velocity illustrated in Fig. 6 because the frictional pressure drop would exceed the inlet TMP. This is also the reason why 6 m/s can be used only in single-module configurations.

3.3. Influence of cross-flow velocity

High cross-flow velocity increases mass transfer [8–19]. However, a high velocity also increases the energy requirement and can also reduce the flux under certain circumstances [5,20].

The predicted flux along the flow channel for different cross-flow velocities is shown in Fig. 7. The curves are

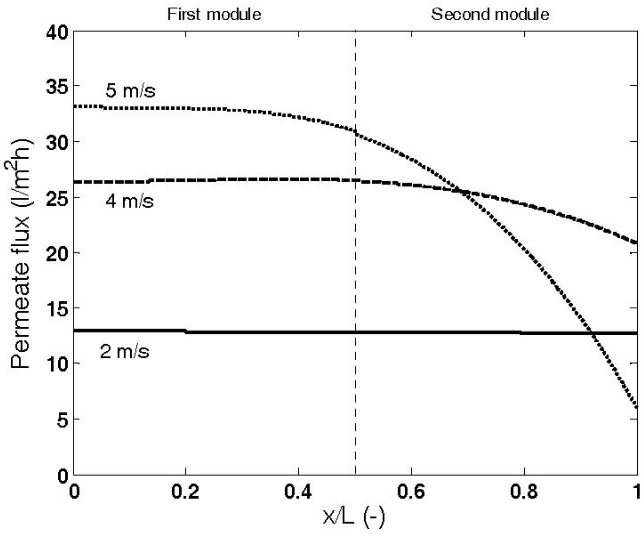

Fig. 7. Flux along the flow channel at various inlet cross-flow velocities. TMP at the inlet, 300 kPa; TS of feed solution, 35%.

steeper for higher cross-flow velocities, i.e. the variation with axial distance is larger. This effect can be observed since the flux is dependent on both TMP and cross-flow velocity. For low cross-flow velocities, the concentration polarization is high but the frictional pressure drop is low. When increasing the cross-flow velocity, the concentration polarization is reduced but the frictional pressure drop increases.

The constant flux along the flow channel at 2 m/s in Fig. 7 is due to the fact that at this cross-flow velocity the flux is independent of pressure in the interval between the TMP at the inlet and the TMP at the outlet.

For longer flow channels and more viscous liquids, the effect of the frictional pressure drop can be even more pronounced [5,20]. The average flux can then decrease with increasing velocity since an increase in velocity has a small positive effect on the flux at the beginning of the module, but this positive effect is completely cancelled out by the negative effect of the higher pressure drop. The same effect was seen by Yeh et al. [21] where UF in hollow-fibre modules was simulated.

Values of flux and TMP at the operating conditions illustrated in Fig. 7 are summarized in Table 3. The average flux is about the same for 4 m/s and 5 m/s, although the average TMP is higher for 4 m/s. The flux per unit pressure is thus higher for 5 m/s. The increase in cross-flow velocity thus has a positive effect on the flux, despite the fact that the NDF is lower.

Another interesting observation is that a higher cross-flow velocity is always beneficial in the first module. In the second module, however, the NDF has been reduced sufficiently to have a substantial negative effect on the performance. For 5 m/s, this effect is so marked that the

Table 3
Transmembrane pressure and flux at the operating conditions illustrated in Fig. 7

Cross-flow velocity at inlet	2 m/s	4 m/s	5 m/s
TMP at the outlet (kPa)	220	100	30
Average TMP (kPa)	260	200	160
Flux at the inlet (L/m^{2}h)	13	26	33
Flux at the outlet (L/m^{2}h)	13	21	6
Average flux (L/m^{2}h)	13	26	27
Average flux in 1st module (L/m^{2}h)	13	27	33
Average flux in 2nd module (L/m^{2}h)	13	25	21

flux is lower than for 4 m/s in the second module. When performing experiments on bench scale, this effect could easily be overlooked. This underlines the importance of taking the variation along the flow channel into account when predicting the performance of full-scale installations, even when the configuration only includes short modules in series.

3.4. Influence of transmembrane pressure

The predicted flux for different TMPs at the inlet of the module is shown in Fig. 8. The flux is the same at the beginning of the first module as the flux is independent of pressure in this TMP interval (see Fig. 2). At the beginning of the module, the flux is thus constant. However, the TMP decreases along the flow channel due to the frictional pressure drop, and the pressure-dependent region is reached. The flux decrease is more pronounced at the lower inlet pressure, since the relative decrease in TMP is larger. The same effect has been noted by Yeh et al. [21,22] when simulating UF in hollow-fibre and tubular modules. The explanation lies in the decrease in the NDF due to frictional pressure drop. For lower TMPs the pressure drop has a larger negative effect on the flux since the NDF towards the end of the flow channel is low.

The decrease in NDF along the membrane is a well-known problem in microfiltration (MF). In MF applications, relatively low TMPs and high cross-flow velocities are used. This means that the frictional pressure drop is often of the same magnitude as the TMP and the NDF , and the flux thus decreases significantly along the flow channel. As a result of this, a filter cake may form at the beginning of the module. This can have negative consequences such as different retention characteristics along the module. One method employed to obtain a uniform NDF throughout the entire membrane module is the Bactocatch process. Here a forced flow on the permeate side of the membrane is adjusted so that a pressure drop corresponding to the pressure drop on the feed side is obtained [23,24]. Commercial ceramic membranes are also available with a longitudinal porosity gradient or with a membrane thickness gradient [25,26]. Another way to deal with this problem is to have separate permeate outlets with valves on each module. This allows separate adjustment of the TMP in each module.

In RO, problems associated with decreasing NDF along the membrane arose when ultra-low-pressure (ULP) membranes were developed. These membranes have a much higher permeability than previous generations of RO membranes, which enables operation at lower TMPs. In RO, the osmotic pressure can be significant and for ULP membranes it can reach the same magnitude as the TMP at the end of the module, resulting in a low NDF . To minimize the negative effects, the design of plants must therefore be approached differently than for RO plants with the old type of membranes [27,28].

3.5. Flux and energy requirement

Optimizing the process design of membrane plants is generally a balance between high flux and low energy requirement. The capital cost and the costs of membrane replacement and electricity required for pumping are, in most cases, the dominating costs for an UF plant. The capital and membrane replacement costs are related to the flux, and the energy requirement in the circulation pump to cross-flow velocity and frictional pressure drop, according to the correlation:

$$W_{recirc} = \left(\frac{\Delta p_f \cdot Q}{\eta \cdot (J \cdot A)}\right) = \frac{\Delta p_f \cdot v \cdot D}{4\eta \cdot J \cdot L} \tag{3}$$

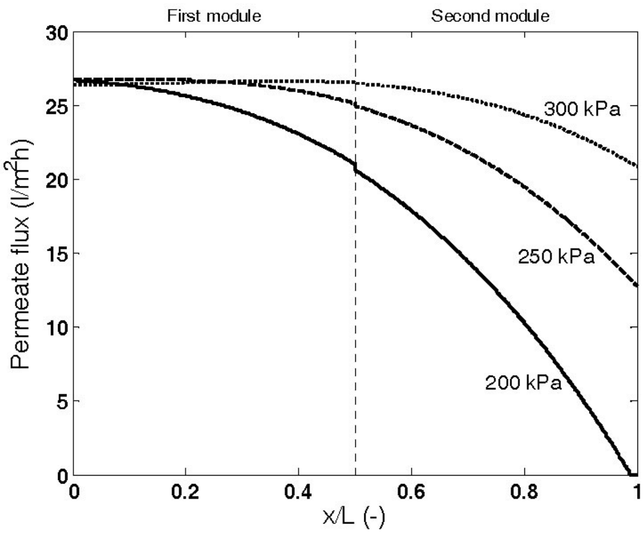

Fig. 8. Flux along the flow channel for evaporated black liquor with a ceramic membrane at different TMPs at the inlet of the first module. Cross-flow velocity at the inlet, 4 m/s; TS of feed solution, 35%.

where W_{recirc} is the energy required per m^3 permeate, Δp_f is the frictional pressure drop, Q is the feed flow, J is the flux and A is the membrane area of a module. η is the pump efficiency, v is the velocity, D is the diameter and L the length of a flow channel in the membrane module. A pump efficiency of 0.8 was assumed in the calculations. The energy requirement in the feed pump is usually of minor importance in UF plants [29].

The average flux and the energy requirement for a membrane plant operating at the three concentration levels studied in the parametric study are shown in Fig. 9. The frictional pressure drop sets the limit for the maximal cross-flow velocity that can be used at each concentration and inlet TMP. The Reynolds number at the lowest velocity (2 m/s) was 6500, 4400 and 1100 at 31%, 35% and 44% TS content, respectively.

As can be seen in Fig. 9, there is a maximum in the flux curve, which is shifted towards higher velocities with increasing inlet TMP. This behaviour is due to the frictional pressure drop. For low inlet TMPs, the TMP remaining at the end of the second module is low, giving a low flux. When the velocity is increased, the frictional pressure drop increases and the effect is more pronounced.

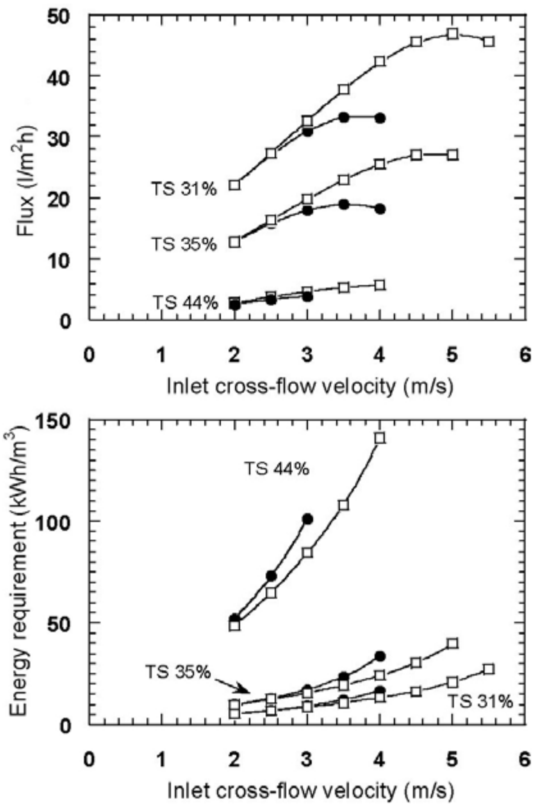

Fig. 9. Calculated average flux (above) and energy requirement (below) at three feed concentrations. Pump efficiency was set to 0.8. □: inlet TMP, 300 kPa; and ●: inlet TMP, 200 kPa.

A drastic increase in energy requirement per m^3 permeate can be seen at a TS content of 44%. This is due to the combined effect of a higher frictional pressure drop and low permeate flux at this concentration.

5. Conclusions

Drastic variations in flux along the flow channel, even in short modules, were shown in this work. The variation in cross-flow velocity and concentration between inlet and outlet was minor because of the relatively low permeate flux and short channel length. The decrease in flux was thus due to the frictional pressure drop, which was significant because of high cross-flow velocities, high bulk concentration (i.e. high viscosity) and small flow channel diameter.

It was shown that there is an optimal cross-flow velocity at which the flux is maximal for a given inlet pressure. The optimal velocity depends on pressure and concentration. The optimal velocity decreases as TMP decreases and bulk concentration increases. A marked increase in the energy requirement was seen as bulk concentration and cross-flow velocity were increased.

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