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Published in:
Journal of Membrane Science

Link to article, DOI:
10.1016/j.memsci.2013.01.057

Publication date:
2013

Document Version
Publisher's PDF, also known as Version of record

Link back to DTU Orbit

Citation (APA):
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Jianquan Luo, Zhenzhou Zhu, Luhui Ding, Olivier Bals, Yinhua Wan, Michel Y. Jaffrin, Eugene Vorobiev

1. Introduction

Chicory, a widely cultivated agro-product, is a rich inulin source and major crop used for the industrial production of inulin [1,2]. Inulin, consisting of a chain of fructose molecules with a terminal glucose molecule, can be classified as a fructooligosaccharide (FOS), which is used either as a fat substitute or as a prebiotic. Moreover, inulin is an important source and major crop used for the industrial production of inulin [1,2]. The raw chicory extract, containing inulin (10–12%) [4], sucrose, pectin, proteins and suspended solids, needs to be clarified before further concentration and precipitation [2]. In the inulin industry, the first clarification step is the clarification of raw juice by limiting and carbonation at high pH [2]. However, this method requires a high processing temperature (>80 °C), which may cause hydrolysis of inulin molecular and deteriorate product quality [2,5].

Membrane filtration as an alternative can greatly simplify the clarification process and decrease operating temperature, which was widely applied to food processing industry [6–9]. Currently, the module configuration most often used for juice clarification in industry is the tubular membrane with crossflow [10,11]. However, due to the limited shear rate, a rapid reduction of permeate flux still occurs, thus limiting the commercial utilization of microfiltration (MF) and ultrafiltration (UF) in juice clarification [12–14]. Hence, a large number of hydrodynamic techniques, based on fluid instabilities, such as pulsating flow, pressure pulsing, periodic backwashing or a “water hammer” operation [15], generation of Dean or Taylor vortices, installation of turbulence promoters or the use of gas–liquid two-phase flow, have been investigated in order to control membrane fouling and flux decline. Dynamic shear-enhanced filtration modules, which create a high shear rate on the membrane by using a rotating disk, or by rotating or vibrating the membranes, have outperformed other available technologies, because of the decoupling of the membrane shear rate from the inlet flow rate into the module, and the consequential minimizing of the pressure drop along the membrane [16]. Several dynamic shear-enhanced membrane
modules have been commercialized, such as DYNO filter (BOKELA), OptiFilter CR (Metso paper), Rotary Membrane System (Spin TeK), single shaft disk filter (SSDF, Novoflow), vibratory shear-enhanced process (VSEP, New logic Research, Inc.), FMX vortex generating module (BKT Co. Ltd.).

On the one hand, with increase of shear rate, the permeate flux will be greatly enhanced by reducing particle deposition on the membrane [12]. On the other hand, a high permeate flux resulting from high shear can build up a cake layer because of additional convective transport of particles towards the membrane. Even when using a rotating disk module, the permeate flux increased only from 60 to 80 L m\(^{-2}\) h\(^{-1}\) when transmembrane pressure (TMP) increased from 1.2 to 2.9 bar in clarification of rough beer by MF [9]. Therefore, choosing an optimum permeate flux (or TMP) is very important to high-shear membrane filtration. Several flux concepts have been proposed to guide permeate flux selection, such as limiting, critical, sustainable and threshold fluxes [17,18]. Except sustainable flux with economic consideration, other flux terms only concern membrane fouling, and they are “critical points” in flux profile with increase of TMP, respectively, labeled according to the fouling evolutions on the membrane. Based on many previous studies [17–21], we propose a collective name “critical flux family” that included critical flux (strong form and weak form), threshold flux, critical flux for irreversibility and limiting flux, as shown in Fig. 1. These “critical points” can be measured by flux stepping and flux cycling procedures, but time scale is a crucial problem [17,22]. Below critical flux, no flux decline with time occurs, so strict steady flux should be ensured and it takes a long time. At and below threshold flux, reversible fouling rate is independent of permeate flux, and above it, fouling rate increases with permeate flux, so only reversible fouling needs to be determined and less time is required [23]. Critical flux for irreversibility is a criterion for the transition between reversible and irreversible fouling; while limiting flux is the plateau of permeate flux with increase of TMP. Actually, threshold flux and critical flux for irreversibility are more applicable for industrial applications, because both extremely low flux with no fouling and high flux with bad fouling are not recommended [17].

Luo et al. [23] reported that for shear-enhanced nanofiltration (NF), threshold flux could be determined by pressure stepping experiments as the point above which flux ceases to increase linearly with TMP, and each flux step only needs to last a few minutes to ensure reversible fouling stabilization at high shear rate. The present work aims at investigating threshold flux in high-shear MF and UF processes. To the best of our knowledge, this is the first time to study permeate flux behavior in clarification of chicory juice by high-shear membrane filtration. In this study, a rotating disk module (RDM) equipped with four MF and UF membranes was used to carry out TMP stepping tests when treating raw chicory root extract. The effects of rotating speed on permeate flux and fouling behaviors, together with flux-TMP profiles were recorded, and long-term operations below, at or above threshold flux, and at limiting flux were compared. Based on our previous study in high-shear NF [23], “threshold flux” concept was further investigated and explained according to present experimental results and a conceptual model. The focus of this work is to understand the flux characteristics in high-shear MF and UF processes and apply “threshold flux” concept to juice clarification for flux optimization and minimization of the decline in flux.

2. Materials and methods

2.1. Experimental set-up and membranes

The rotating disk module (RDM), mainly consisting of one disk mounted on a single shaft and rotating near a fixed circular membranes, was described in detail elsewhere [24]. A flat membrane, with an effective area of 176 cm\(^2\) (outer radius \(R_1\) = 7.72 cm, inner radius \(R_2\) = 1.88 cm), was fixed on the cover of the cylindrical housing in front of the disk. The disk equipped with 6 mm-high vanes can rotate at up to 2500 rpm, inducing high shear rates on the membrane. Four commercial UF and MF membranes fabricated by MICRODYN-NADIR GmbH and Alfa Laval Nakskov A/S were tested in the present study and their properties are summarized in Table 1, according to the manufacturer’s information.

2.2. Test fluid

Fresh chicory roots provided by COSUCRA, Belgium, were used for juice extraction. After pulsed electric field (PEF) pretreatment

![Critical flux family](image)

**Fig. 1.** A general description of “critical flux family”.

<table>
<thead>
<tr>
<th>Membrane</th>
<th>Manufacturer</th>
<th>Surface material</th>
<th>Pore size</th>
<th>(T_{\text{max}}) (°C)</th>
<th>pH range</th>
<th>Water permeability (L m(^{-2}) h(^{-1}) kPa(^{-1}))</th>
</tr>
</thead>
<tbody>
<tr>
<td>US100P</td>
<td>Microdyn–Nadir</td>
<td>PSH</td>
<td>100 kDa</td>
<td>95</td>
<td>1–14</td>
<td>1.1–1.6</td>
</tr>
<tr>
<td>FSM0.15PP</td>
<td>Alfa Laval Nakskov</td>
<td>PVDF</td>
<td>0.15 μm</td>
<td>60</td>
<td>1–11</td>
<td>1.4–1.7</td>
</tr>
<tr>
<td>MV020T</td>
<td>Microdyn–Nadir</td>
<td>PVDF</td>
<td>0.20 μm</td>
<td>95</td>
<td>2–11</td>
<td>1.3–2.0</td>
</tr>
<tr>
<td>FSM0.45PP</td>
<td>Alfa Laval Nakskov</td>
<td>PVDF</td>
<td>0.45 μm</td>
<td>60</td>
<td>1–11</td>
<td>2.0–2.3</td>
</tr>
</tbody>
</table>

PVDF: Polyvinylidenefluoride, PSH: Hydrophilic polysulphone.

* Own measurement at 25°C.
for 50 ms by a pilot PEF generator (Hazemeyer, France), sliced chicory juice was put into a temperature controlled counter-current pilot-scale extractor. The temperature during diffusion varied between 30 and 80 °C, and the diffusion duration was fixed at 90 min. Configuration and principle of operation of the diffuser and details of the diffusion experiments were reported in previous work [4]. The chicory extract from different diffusion conditions were pre-filtered through a mesh of 0.25 mm pore size, mixed and then divided into portions of 1.5 L and stored as a solid at a temperature of –20 °C. The main characteristics of this raw chicory extract are presented in Table 2 [25–27].

2.3. Experimental procedure

A new membrane was used for each series of experiments. New membranes were dipped in 50% ethanol solution for 30 min to remove manufacturing residues from the membrane surface, then soaked in deionized water for at least 24 h prior to use. Once installed, each membrane was compacted for 60 min under a pressure of 200 kPa, and then pure water permeability ($L_p$) of membranes was measured as a function of pressure (200, 150, 100, 50 kPa) at 25 °C. Before the experiments started, 4 L feed was heated to 50 °C, and fully recycled in the system at a feed flow rate of 120 L h⁻¹, TMP of 26 kPa, and rotating speed of 2000 rpm.

### Table 2

Main characteristics of chicory root extract.

<table>
<thead>
<tr>
<th>Characteristics</th>
<th>Value</th>
</tr>
</thead>
<tbody>
<tr>
<td>Component concentration (g L⁻¹)</td>
<td></td>
</tr>
<tr>
<td>Dry mass</td>
<td>123–129</td>
</tr>
<tr>
<td>Inulin [25]</td>
<td>90–110</td>
</tr>
<tr>
<td>Saccharide [25]</td>
<td>115–120</td>
</tr>
<tr>
<td>Pectin [26]</td>
<td>NA</td>
</tr>
<tr>
<td>Protein [27]</td>
<td>2–4</td>
</tr>
<tr>
<td>Solute molecular weight range (kDa)</td>
<td></td>
</tr>
<tr>
<td>Inulin [25]</td>
<td>0.34–11.3</td>
</tr>
<tr>
<td>Pectin [26]</td>
<td>300–890</td>
</tr>
<tr>
<td>Protein [27]</td>
<td>14.2–66</td>
</tr>
<tr>
<td>pH (at 25 °C)</td>
<td>5.6 ± 0.2</td>
</tr>
<tr>
<td>Viscosity (mPa s)</td>
<td>1.7 ± 0.3 (25 °C); 0.92 ± 0.25 (50 °C)</td>
</tr>
<tr>
<td>Density (g L⁻¹)</td>
<td>1182</td>
</tr>
</tbody>
</table>

NA: not available.

Fig. 2. Schematic diagrams of experimental strategies. (a) TMP stepping experiments at different rotating speeds (CP = concentration polarization); (b) TMP stepping experiments followed by long-term constant TMP tests at 1000 rpm (strategy (1) below threshold flux; strategy (2) above threshold flux; strategy (3) at limiting flux).
for 60 min. Then experiments were carried out in total recycling mode (i.e. recirculating both retentate and permeate back to the feed tank), and were divided into three series of tests according to operating procedure.

2.3.1. TMP stepping at different rotating speeds

As illustrated in Fig. 2a, at a rotating speed of 2000 rpm, fluxes at four TMP of 75, 100, 125 and 150 kPa were measured in turn and then TMP was decreased to 75 kPa. Each TMP step lasted 5–10 min and the corresponding flux was measured during the last 2 min. Rotating speed was then decreased to 1500, 1000 and 500 rpm, respectively, with the same procedure of TMP variation. At the end of the test, rotating speed was set back to 2000 rpm at 75 kPa and the flux was recorded with time (during 30 min for MF and 11 min for UF).

2.3.2. TMP stepping followed by constant TMP operations

This series of experiment was carried out at a constant rotating speed of 1000 rpm for membranes with pore size of 100 kDa and 0.20 μm. As shown in Fig. 2b, TMP was increased in steps from 26 kPa to a certain value, and each TMP step lasted 10 min and the corresponding flux was measured every 2 min. TMP intervals were 5–20 kPa as required. Then, three different operations at constant TMP were conducted: (1) when reaching the limiting flux point (flux plateau with increase of TMP), TMP was directly decreased to 1500, 1000 and 500 rpm, respectively, with the same procedure of TMP variation. At the end of the test, rotating speed was set back to 2000 rpm at 75 kPa and the flux was recorded with time (during 30 min for MF and 11 min for UF).

2.3.3. Periodic TMP stepping at a constant rotating speed

This series of experiment was carried out at a constant rotating speed of 1000 rpm only for the 0.20 μm membrane. TMP was increased in steps from 26 to 100 kPa and then directly decreased to 26 kPa. After flux stabilization for 40–60 min, TMP stepping was carried out again. Three cycles were conducted in this series.

2.4. Analytical methods

Turbidities of permeate were measured with a Ratio Turbidimeter (Hach, USA). Soluble matter contents were measured by means of a digital refractometer PR-32a (ATAGO Co., Ltd., Japan). Saccharide contents of samples were measured by the phenol sulphuric acid method [28] using BSA (bovine serum albumin) column (78 mm, 85 °C). The protein content was measured by the Bradford protein assay using BSA (bovine serum albumin) as standard. pH was measured with pH Meter (MP 125, Mettler Toledo, Switzerland). The dynamic viscosity of raw juice was determined by a rheometer (Physica MCR301, Anto-Paar, Austria) at shear rates from 1000 to 1500 s⁻¹. Dry mass was determined by measuring the weight loss after drying samples at 105 °C for 5 h in an oven.

2.5. Calculated parameters

The mean membrane shear rate (γₘ s⁻¹) for our RDM system can be calculated by the following equation [16]:

\[
\gamma_m = \frac{0.0164 R^{0.5} (ko) \omega}{\sqrt{\nu}}
\]  

(1)

where \( R \) is the outer membrane radius (m) and \( k \) is the velocity factor (0.89 for this RDM system), \( \omega \) the disk angular velocity (rad s⁻¹) and \( \nu \) is the fluid kinematic viscosity (m² s⁻¹).

The mean TMP is obtained by integrating the local pressure over the membrane area as follows [16]:

\[
\text{TMP} = \frac{p_e - 1}{\pi k^2 \omega R^2} 
\]

(2)

where \( p_e \) is the measured peripheral pressure (Pa) and \( \rho \) is the density of the fluid (g L⁻¹).

Energy consumption per m³ of permeate (specific energy, \( E_c \)) for the rotating disk is represented as:

\[
E_c = \frac{P_d}{Q_l}
\]

(3)

where \( P_d \) is the power of rotating disk motor (kW), and \( Q_l \) is the permeate flow rate (m³ h⁻¹).
The flux decline can be expressed as a percentage of feed permeate flux reduction after rotating speed decrease or TMP stepping cycle (75 → 100 → 125 → 150 → 75 kPa):

\[
\text{Flux decline (\%) = } \left( \frac{J_{sf} - J_{f}}{J_{sf}} \right) \times 100
\]

(4)

where \( J_{sf} \) and \( J_f \) are stabilized feed permeate fluxes (L m\(^{-2}\) h\(^{-1}\)) at the beginning and end of operations (75 kPa), respectively.

The solute transmission is defined as:

\[
\text{Transmission (\%) = } \left( \frac{C_p}{C_f} \right) \times 100
\]

(5)

where \( C_p \) and \( C_f \) are the solute concentration in permeate and feed (g L\(^{-1}\)), respectively.

Based on Darcy's law, total filtration resistance \( (R_t, \text{m}^{-1}) \) is given by:

\[
R_t = R_m + R_{ads} + R_f = \frac{\text{TMP}}{\eta J}
\]

(6)

where \( \eta \) is the solvent viscosity (Pa s) and \( J \) is the volumetric flux per unit area of membrane (m s\(^{-1}\)). \( R_m \) is the intrinsic membrane resistance, \( R_{ads} \) is the resistance due to adsorption fouling on membrane surface and pore wall, \( R_f \) is the reversible fouling resistance resulting from concentration polarization layer or particle deposit, \( R_p \) is the irreversible fouling resistance due to pore plugging or cake deposit.

3. Results and discussion

3.1. Flux behaviors at different shear rates and filtration stages

3.1.1. Effect of rotating speed on permeate flux

Permeate fluxes at a TMP of 75 kPa under different rotating speeds for four membranes are shown in Fig. 3a. This permeate flux increased with rotating speed due to lower concentration polarization (CP) and/or fouling at high shear rate. Second, at rotating speeds of 2000 and 1500 rpm, the permeate flux increased with pore size, while at 500 rpm, it was almost independent of membrane. This can be explained by the different fouling mechanisms at different shear rates. At a high rotating speed, since cake fouling is negligible, the permeate flux is governed by membrane permeability (e.g., pore size and hydrophilicity). While at a low rotating speed, the foulants, i.e., pectin, proteins, and suspended solids, deposit easily on the membrane, resulting in a thick and compact cake layer, and the permeate flux is mainly controlled by cake fouling layer and is independent of membrane permeability.

Therefore, higher shear rates can reduce solutes accumulation at membrane surface and thus decrease total filtration resistance, leading to a permeate flux at constant TMP proportional to rotation speed. However, an excessive shear rate will waste much energy and increase abrasion of equipments. The specific energy of rotating disk motor consumed per m\(^3\) of permeate for these four membranes was calculated according to the method of [24]. As shown in Fig. 3b, for the 100 kDa membrane with lowest flux, specific energy decreased from 500 to 1000 rpm, then rapidly increased from 1000 to 2000 rpm and the rotation speed of 1000 rpm was energy-optimal; while for other membranes, specific energy decayed at 1000 rpm, and then gradually increased with rotating speed. However, for MF membranes, the specific energy was not much higher at 1500 and 2000 rpm while the membrane area for a given permeate flow rate would decrease. Thus the most efficient process may be to use a MF membrane at a speed of 2000 rpm.

3.1.2. Flux decline and fouling

As seen in Fig. 2a, at 75 kPa, a reduction of rotating speed will cause a flux decline in a short time, resulted from an incremental CP layer at lower shear rate. These experimental results are summarized in Fig. 4a, showing that CP-induced flux decline is highest when rotating speed decreases from 1000 to 500 rpm, and such decline is higher for membrane with larger pore size, due to higher membrane permeability. Thus, a rotating speed of 1000 rpm is not only energy efficient, but also necessary for controlling the CP layer.

After TMP stepping cycle, permeate flux decreases at the same TMP and rotating speed (75 kPa, 2000 rpm), as illustrated in Fig. 2a, which is considered to be induced by fouling occurred during TMP stepping. The flux declines after one TMP cycle (lasting 20~30 min) and four cycles (lasting 130 min) are shown in Fig. 4b. These declines are minimal for the 100 kDa and 0.45 μm membranes. The flux decline after one cycle for the 100 kDa is lowest compared with others, and the 0.45 μm membrane has the smallest increase in decline after four cycles. This can be explained as follows: the first TMP stepping cycle was carried out at a high shear rate (2000 rpm) for a short period (~20 min), and the flux decay was mainly caused by pore adsorption and plugging, so it was more difficult for foulants to enter smaller pores, resulting in lowest fouling for 100 kDa membrane; while after four cycles at different rotating speeds, foulants deposit and cake fouling were the main mechanisms of

![Fig. 4. Flux decline rates due to (a) concentration polarization (CP) and (b) fouling, respectively, for different membranes in the first series of experiments (Fig. 2a). TMP=75 kPa.](image-url)
flux decay, and because of less foulants rejected by the 0.45 μm membrane, its flux decline became much lower than 0.15 and 0.20 μm membranes.

The effect of membrane fouling on saccharide transmission and permeate turbidity is shown in Fig. 5 for the four membranes at a speed of 2000 rpm. Though membrane fouling caused significant flux declines, unexpectedly, it had a negligible effect on solutes transmission, which increased with pore size and may be attributed to the high shear rate. Permeate turbidity was quite low for all membranes tested, less than 2.5 NTU and similar for new and fouled membranes, except for the 0.20 μm one which had a high initial permeate turbidity of 4.8 NTU that decreased to 1.7 NTU after 130 min filtration due to pore fouling (see Fig. 5b).

3.1.3. Flux recovery by increasing disk rotation

Fig. 6 shows on left side the flux decay when the rotation speed was reduced in steps from 2000 to 500 rpm after 100 min of filtration, reaching the same flux of about 35 L m⁻² h⁻¹ for the four membranes. The flux recovery when rotating speed is directly raised to 2000 rpm, is shown on the right side. The relative recovery compared with the initial flux at 2000 rpm, reached 71% after 11 min of filtration at 2000 rpm for 100 kDa membrane, while it was, after 30 min, 55, 56 and 71%, respectively, for the 0.15, 0.20 and 0.45 μm membranes. Such flux recovery was caused by a high shear rate (2.5 x 10⁵ s⁻¹) that accelerated the dispersion of foulants accumulated on the membrane and partially broke the cake layer. It is worth mentioning that shear-induced hydrodynamic diffusivity is proportional to the square of the particle size multiplied by the shear rate [12]. Presumably, the larger flux recovery of 0.45 μm membrane is due to the larger shear-induced hydrodynamic diffusivity of its rejected particles with bigger size.

As described above, a high shear rate produced by a rotating disk near a membrane can reduce membrane fouling, and if
operation has occurred at a lower speed, also provide self-cleaning ability. However, for these advantages, energy consumption is higher. Taking into account the cost, a highest rotating speed (e.g. 2000 rpm) is not suitable for a long-term operation, and according to the results in Fig. 3b, a moderate rotating speed of 1000 rpm was selected to further study, while a higher disk rotation can be employed periodically for fouling removal.

3.2. Threshold and limiting fluxes in high-shear membrane filtration

3.2.1. Determination of threshold and limiting fluxes

The 100 kDa and 0.20 μm membranes were selected to study threshold and limiting fluxes at 1000 rpm, and some representative data of TMP stepping are shown in Fig. 7, indicating that both fluxes reach a plateau at a TMP of about 75 kPa for the 100 kDa membranes and 60 kPa for the 0.20 μm membranes. Fluxes become almost stable within 10 min, indicating that the reversible fouling layer (i.e. concentration polarization and particle deposit) forms very quickly. Permeate fluxes, averaged during the last two minutes of each TMP step of five experiments were plotted as a function of TMP in Figs. 8 and 9a. It can be seen in Fig. 8a that the permeate flux first increases with TMP linearly for the 100 kDa membrane and then the flux levels off when TMP exceeds certain values. The threshold flux was determined by linear regression as previous study [23], being about 130–140 L m\(^{-2}\) h\(^{-1}\) for three tests, while the limiting flux was about 155 L m\(^{-2}\) h\(^{-1}\) for two tests. However, for 0.20 μm membrane in Fig. 9a, no “break point” can be found, and the lowest fluxes for two tests are 140 and 151 L m\(^{-2}\) h\(^{-1}\), and the limiting flux is about 160–168 L m\(^{-2}\) h\(^{-1}\).

According to the results in Figs. 8 and 9a, it was found that, although the flux at 26 kPa for 0.20 μm membrane was three-times that for 100 kDa, their limiting fluxes were very close. Furthermore, if threshold and limiting fluxes were independent of membrane pore size and permeability, the reason for the absence of threshold flux in Fig. 9a might be that, the first measured flux exceeded the value of threshold flux. This hypothesis will be further discussed in Section 3.2.3.

Fig. 10 shows the effect of TMP on permeate turbidity during TMP stepping experiments at 1000 rpm. For 100 kDa membrane, due to its small pore size, permeate turbidity remained low at 1 NTU at all TMP, while for 0.20 μm membrane, it increased exponentially with TMP due to a larger average pore size and wider size distribution. At the same time, more suspended solid
accumulates on the membrane and passes through membrane at higher TMP. Therefore, an inappropriate TMP (or flux) not only aggravates membrane fouling, but also deteriorates permeate quality.

3.2.2. Comparison of long-term flux behaviors

Long-term operations with three different strategies were carried out based on the results from TMP stepping tests (Fig. 8b). The first strategy called “operation below threshold flux”, was to decrease TMP to the “threshold point” when reaching the flux plateau; the second was to increase the flux above

threshold flux after flux stepping operation; the third was conducted at limiting flux after flux stepping. As seen in Fig. 8b, permeate flux increases by 27.8%, to be 124 L m\(^{-2}\) h\(^{-1}\) in the first strategy, but this is still lower than measured threshold flux (135 L m\(^{-2}\) h\(^{-1}\)). When operating above threshold flux, the permeate flux only decreased by 3.2%; for the limiting flux operation, it decayed by 7.4%. As shown in Fig. 9b, the permeate flux increases by 82.1%, to be 118 L m\(^{-2}\) h\(^{-1}\) below threshold flux; while it drops by 15.5% when operating at limiting flux. Actually, at this high-shear rate, flux declines after full-recycling operations for 360 min were not very significant, but it is obvious that threshold flux operation is more suitable for long-term running, and limiting flux operation should be avoided.

3.2.3. Effect of fouling on flux behavior

The effect of membrane fouling on threshold and limiting fluxes for 0.20 μm membrane is shown in Fig. 11. Continuous TMP stepping experiments were carried out to ensure that membrane was fouled gradually, and this could be confirmed by the decreasing fluxes at 26 kPa, 142 L m\(^{-2}\) h\(^{-1}\) in first cycle, 104 and 77 L m\(^{-2}\) h\(^{-1}\) in 2nd and 3rd. As seen in Fig. 11, with increasing fouling, threshold fluxes can be found in the flux-TMP profiles (Fig. 11b2 and b3), and its values are irrespective of the fouling extent, about 135–140 L m\(^{-2}\) h\(^{-1}\), while limiting fluxes are always about 165 L m\(^{-2}\) h\(^{-1}\). Therefore, threshold and limiting fluxes are probably independent of membrane properties and fouling condition in high-shear UF and MF processes, and they are mainly governed by shear rate and temperature [23]. Permeate turbidity increased with TMP in 1st cycle (b1) while it remained lower and nearly constant at higher fouling in b2 and b3 graphs, confirming that there was a lower cake porosity and

Fig. 11. Effect of membrane fouling on flux behaviors for 0.20 μm membrane during periodic TMP stepping experiments (three cycles). Rotating speed = 1000 rpm.
more compact fouling layer after TMP stepping increase and direct release.

Comparing the results in Figs. 9 and 11, it is found that when operating at constant TMP, even at limiting flux, the flux decline is much lower than in periodic TMP stepping operation, and during the same period (360 min), flux decline was 15.5% at limiting flux operation (from 161 to 136 L m⁻² h⁻¹, see Fig. 9b), while permeate flux at 26 kPa decays by 65.1% after three cycles of TMP stepping (from 142 to 49.5 L m⁻² h⁻¹, see Fig. 11a). First, when operating at limiting flux, the irreversible fouling actually was growing and total resistance increased a lot, but the TMP was so high (80 kPa) to offset this incremental resistance, and at the same time, the reduction of driving force would partly decrease the reversible fouling and prevent the occurrence of rapid flux decline. Second, because the shear-induced back diffusion was higher for larger particles [12], when TMP decreased to 26 kPa, the larger foulants deposited on the membrane had a greater tendency to diffuse back into bulk solution, thus reducing the average size of deposit particles. During the cycling of TMP and flux, the deposit layer was rearranged and the cake morphology was reconstructed in a manner that enhanced the specific resistance of cake, thus resulting in higher flux decline during periodic TMP stepping tests. Third, the incremental filtration resistance would produce a greater effect on the variation of permeate flux at lower TMP. That was why the flux decline in limiting flux operation (80 kPa in Fig. 9) was much lower than that during periodic TMP stepping operation (26 kPa in Fig. 11b). Therefore, in practical applications, significant TMP or flux fluctuations and limiting flux operation should be avoided in high-shear membrane filtration.

### 3.2.4. Mechanism discussion

Field and Pearce [18] have first defined threshold flux as the transition between low and high fouling rates, and according to permeability loss data from pilot studies, they considered that fouling rate is almost constant at and below threshold flux. In our previous study, we defined threshold flux in high-shear membrane filtration as “at and below threshold flux, adsorption and reversible fouling are almost stable, irrespective of permeate flux; above threshold flux, reversible fouling increases with flux” [23], as illustrated in Fig. 12a. Above limiting flux, a further increase in flux at any point on the membrane surface leads to another deposited layer, fully compensating the increased driving force, and thus a flux plateau and an increasing filtration resistance occur with increase of TMP (see Fig. 12a). The filtration resistances in present study calculated by Eq. (6) are shown in Fig. 12b, verifying our definition of threshold and limiting fluxes. However, due to the limited accuracy and reliability of our equipments, the threshold and limiting fluxes only can be determined in a flux range (130–140 L m⁻² h⁻¹ for threshold flux, 155–168 L m⁻² h⁻¹ for limiting flux at 1000 rpm and 50 °C). Obviously, threshold flux represents a highest throughput with lowest fouling, which is recommended for practical applications.

From a general point of view, a solute mass balance in the boundary layer under steady state is a combination of a convective flux and the fluxes derived from “dispersive” effect [17]:

\[ J_C = D \frac{dC}{dx} + p(\xi) + q(\tau) \]  
(7)

where \( C \) is solute concentration, \( D \) is the Brownian diffusion coefficient, \( p(\xi) \) represents the term for migration of the solutes/particles due to surface interactions between the membrane and solutes/particles surfaces, and \( q(\tau) \) stands for the effect of the local hydrodynamics on the mass flux (mainly induced by shear force in present study). The possible settlement of non-buoyant particles onto the membrane surface will not be considered. For high-shear membrane filtration, \( q(\tau) \) pays an important role in “dispersive” transport of particles out from polarized layer. The shear-induced hydrodynamic diffusivity is proportional to the square of the particle size multiplied by the shear rate, while the Brownian diffusion coefficient \( D \) is irrespective of shear rate and inversely proportional to particle size [12]. Based on such offset between Brownian and shear-enhanced diffusions, a hypothesis is proposed to explain the presence of threshold flux.

Theoretically, solutes are carried to the membrane surface by convective transport, where the small solutes and solvent easily passes through the membrane and the retained solutes induce an increase in the local concentration that comes to balance the back diffusion of rejected particles into the bulk, which is mainly due to the resulting concentration gradient (Brownian diffusion) and shear-enhanced back transport. With increase of permeate flux, more particles are transported and rejected on the membrane, and some retained particles may assemble together by collisions or adsorption, resulting in a reduction of Brownian diffusion due to larger size of their aggregations. However, the shear-enhanced

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**Fig. 12.** (a) Schematic presentation of threshold flux, critical flux for irreversibility and limiting flux; (b) Experimental results about total filtration resistance variations with increase of permeate flux.
diffusion of these aggregations increases, bringing them back to the bulk solution. That is, at or below threshold flux, the polarized layer on the membrane is dynamic and the rejected particles are floating, inducing an almost constant filtration resistance within threshold flux. When above threshold flux, the shear-enhanced back transport cannot afford the decrease of Brownian diffusion of particle aggregations, and more and more particles accumulate and deposit on the membrane, causing an augment of filtration resistance.

4. Conclusion

These results confirm the high performance of high-shear dynamic filtration in chicory juice clarification by UF and MF both in terms of permeate flux, sugar transmission and permeate clarity. Increasing disk rotation increased permeate flux and controlled polarization and fouling, but consumed more specific energy. A moderate rotating speed of 1000 rpm was not only energy efficient, but also necessary for deposition control, while a high rotating speed of 2000 rpm could be used to remove the fouling layer. Moreover, membrane fouling had negligible effect on both saccharide transmission and permeate quality due to the high shear rate.

A threshold flux was proposed to distinguish between low and high fouling rates, below it, filtration resistance is independent of flux, while above it, filtration resistance is flux-dependent because membrane fouling increases with permeate flux. When reaching limiting flux, the resistance increases rapidly with TMP. This work confirmed that this new threshold flux criterion was applicable to high-shear MF and UF, and threshold and limiting fluxes might be independent of membrane pore size, permeability and fouling condition. The shift in size-dependent shear-induced and Brownian back diffusions may be the essential reason of threshold flux phenomenon. Although operating above the threshold flux would not cause significant flux decline at a constant TMP, it produced a thicker deposit layer and wasted energy, and even resulted in a higher turbidity in permeate. Distinct TMP or flux fluctuation can induce a much higher flux decline because the deposit particles can rearrange into a more compact cake layer during the cycling of TMP and flux. Therefore, in order to minimize fouling and flux decline, operating at threshold flux in constant flux mode is recommended for chicory juice clarification by high-shear membrane filtration.

Acknowledgments

The financial support of China Scholarship Council (CSC) for Jianquan Luo and Zhenzhou Zhu is acknowledged. The authors thank COSUCRA, Belgium for supplying chicory roots and Alfa Laval Nakslov A/S Company for supplying FSM0.15PP and FSM0.45PP membranes.

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