Chicory juice clarification by membrane filtration using rotating disk module

Zhu, Zhenzhou; Luo, Jianquan; Ding, Luhui; Bals, Olivier; Jaffrin, Michel Y.; Vorobiev, Eugene

Published in:
Journal of Food Engineering

Link to article, DOI:
10.1016/j.jfoodeng.2012.10.028

Publication date:
2013

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

Link back to DTU Orbit

Citation (APA):
Chicory juice clarification by membrane filtration using rotating disk module

Zhenzhou Zhu a, Jianquan Luo a, Luhui Ding a,*, Olivier Bals a, Michel Y. Jaffrin b, Eugene Vorobiev a

a EA 4297 TIMR, Technological University of Compiegne, 60205 Compiegne Cedex, France
b UMR 7338, Technological University of Compiegne, 60205 Compiegne Cedex, France

A R T I C L E   I N F O

Article history:
Received 9 July 2012
Received in revised form 8 October 2012
Accepted 14 October 2012
Available online 26 October 2012

Keywords:
Chicory juice
Clarification
Microfiltration
Ultrafiltration
High shear

A B S T R A C T

Clarification is the first step of inulin production from chicory juice, and membrane filtration as an alternative can greatly simplify this process, increase juice yield, improve product quality, and reduce the cost and waste volume. In this study, a rotating disk module (RDM) was used to investigate the clarification of chicory juice by four micro- and ultrafiltration membranes. Compared with dead end filtration, the RDM had a much higher permeate flux and product quality. High rotating speeds produced high permeate fluxes and reduced flux decline, because of the strong back transport of foulant from fouling layer to feed solution. At high rotating speeds of 1500–2000 rpm, the permeate flux increased with membrane pore size and transmembrane pressure (TMP), while at low rotating speeds (<1000 rpm), permeate flux was independent of membrane type and TMP due to a thick deposited fouling layer as a dominant filtration resistance, while carbohydrate transmission decreased at higher TMP because of denser cake layer as an additional selective membrane. The highest carbohydrate transmission (~98%) and desirable permeate turbidity (2.4 NTU) was obtained at a TMP of 75 kPa and a rotating speed of 2000 rpm for FSM0.45PP membrane. With the RDM, the Volume Reduction Ratio (VRR) could reach 10 with a high permeate flux (106 Lm⁻²h⁻¹) in the concentration test, and permeate was still rich in carbohydrate and well clarified. Chemical cleaning with 0.5% P3-ultrasil 10 detergent solution was able to recover 90% water flux of fouled membrane.

© 2012 Elsevier Ltd. All rights reserved.

1. Introduction

Inulin, first separated from the extract of Inula helenium in 1804 and named by Thomson in 1818, is an energy reserving material classified as non-digestible carbohydrate (Kim et al., 2001). Chemically, inulin is a kind of fructan, consisting of 2–70 repeating fructose units (De Leenheer and Hoebrugs, 1994). Being classified as non digestible carbohydrate, inulin has been widely used as a fat substitute in food industry (Vendrell-Pascaus et al., 2000). Among those various inulin containing plants (chicory root, dahlia tuber, Jerusalem artichoke, burdock root, onion bulb and leek bulb) (Vanloo et al., 1995), chicory root, dahlia tuber and Jerusalem artichoke, because of their high inulin content (>10%), are considered as good candidates for industrial production of inulin. Especially, chicory root is the favored one for extraction of inulin, due to its easy cultivation, high inulin yield (Franck and De Leenheer, 2005) and stable long-chain production (Toneli et al., 2008).

Conventional production of inulin involves several steps (Franck and De Leenheer, 2005): (1) extraction of inulin from the sliced chicory roots with hot water in a counter-current diffuser, at a temperature of 70–80 °C during 1–2 h; (2) clarification (primary purification) of raw juice by liming and carbonation at high pH; (3) further refining using cationic and anionic ion-exchange resins for demineralization, and active carbon for decolorization; (4) spray drying of refined juice to obtain dry inulin powder.

In contrast to the conventional inulin extraction process, which requires high temperature and long time, alternative technologies have been carried out, including supercritical carbon dioxide (CO2) (Mendes et al., 2005), ultrasound (Wei et al., 2007), simultaneous ultrasonic/microwave (Lou et al., 2009) and pulsed electric field (PEF) assisted extraction (Loginova et al., 2010; Zhu et al., 2012). These technologies pointed out the possibility to extract inulin with a less polluting, less energy consuming and high efficient process. According to Zhu et al. (2012), a PEF pre-treatment of chicory cossettes could significantly decrease the diffusion temperature by 20 °C and promote inulin transport from chicory to extracted juice.

The classical method for inulin clarification requires multiple operation steps (pre-liming, liming, a first carbonation, a first filtration, a second carbonation and second filtration (Franck and De Leenheer, 2005) and also high temperature (80–90 °C), which may lead to the hydrolysis of inulin molecular in the extracted juice (Kim et al., 2001). Although, these purification steps can effectively remove insoluble and certain soluble substances, they may also introduce additional calcium ions into clarified juice that requires further purification treatments. Thus, this widely used classical clarification technology is also regarded as labor intensive and time-consuming. Membrane technologies (especially
microfiltration and ultrafiltration) have been proposed and investigated as an alternative for juice clarification (Cassano et al., 2007; De Bruijn et al., 2003; Mirsaeedghazi et al., 2010; Youn et al., 2004) because of their advantages such as high productivity, low operation cost and high product quality.

However, the use of membrane filtration is limited by flux decline with filtration time due to the concentration polarization, cake formation and membrane pore fouling (De Bruijn et al., 2003; Luo et al., 2012a; Rai et al., 2010). In juice filtration fouling is caused by the accumulation of macromolecular or colloidal species (such as pectin, proteins and colorants) on the membrane surface as well as in the membrane pores. Efforts have been made to eliminate or control membrane fouling, including fabrication of anti-fouling membranes (Liu et al., 2006; Zhao et al., 2008), pretreatment of feed juice (Gokmen and Cetinkaya, 2007), and use of shear-enhanced process (Fillaudeau et al., 2007; Luo et al., 2010, 2012a). Among these available approaches, operating at high shear rate is technically sound and economically attractive. For example, the rotating disk module (RDM) which can generate a high shear rate \((1-3 \times 10^5 \text{ s}^{-1})\) has been successfully applied in various areas such as wastewater treatment (Luo et al., 2012b) and bio-separation (Mellal et al., 2008).

Although clarification of juice by membrane technologies has been widely investigated, this study is the first one using a RDM in micro and ultra filtrations of chicory juice. The goals of present work were to compare the filtration behavior and clarification efficiency of dead-end filtration and RDM, to investigate the effect of process parameters, i.e. membrane pore size, rotating speed and transmembrane pressure (TMP), on flux, permeate turbidity and carbohydrate transmission, and to evaluate the membrane performance for clarification of chicory juice in concentration mode.

2. Materials and methods

2.1. Extraction of chicory juice

Fresh chicory roots provided by COSUCRA, Belgium, were used for juice extraction. After pulsed electric field (PEF) pretreatment of 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. Construction and principle of operation of the diffuser and details of the diffusion experiments were reported in previous work (Zhu et al., 2012). The extracted juices from different diffusion conditions were pre-filtered by mesh of 0.25 mm pore size and mixed, then divided into portions of 1.5 L and stored at the temperature of \(-20 \degree C\) until further use. The main characteristics of the chicory juice are presented in Table 1.

2.2. Clarification of chicory juice by dead end filtration

Dead-end filtration was performed in a stirred cell Amicon 8200 (effective membrane area \(3.17 \times 10^{-3} \text{ m}^2\) and maximal volume 180 mL) (Millipore, Billaica, USA). The temperature of the feed juice was maintained at 50 ± 1 °C during the filtration. Compressed air was used to supply a TMP of 1 bar. Membranes with molecular weight cut-off (MWCO) or pore sizes of 100 kDa, 0.15, 0.2 and 0.45 μm were used to clarify the feed juice, and their main properties are presented in Table 2. A magnetic stirrer fixed over the membrane surface provided a constant stirring speed of 350 rpm. For each experiment, 180 mL of feed juice was concentrated to 90 mL. The mass of permeate was recorded with time by a computer during filtration for further calculation. Filtration permeate (90 mL) and the corresponding retentate (90 mL) were collected for subsequent analysis.

2.3. Clarification of chicory juice by RDM

2.3.1. RDM set-up

The RDM module, shown in Fig. 1, was used for clarifying chicory juice. A flat membrane, with an effective area of 176 cm² (outer radius \(R_1 = 7.72 \text{ cm}\), inner radius \(R_2 = 1.88 \text{ cm}\)), was fixed on the cover of the cylindrical housing in front of the disk. The disk equipped with 6 mm-high vanes, which can generate very high shear rates on the membrane, can rotate from 500 to 2500 rpm. The module was fed from a thermostatic and stirred tank containing 12 L of fluid by a volumetric diaphragm pump (Hydra-cell, Wanner, USA). The peripheral pressure \((P_c)\) was adjusted by a valve on outlet tubing and monitored at the top of the cylindrical housing by a pressure sensor (DP 15–40, Validyne, USA), and the data was collected automatically by a computer. Permeate was collected in a beaker placed on an electronic scale (B3100 P, Sartorius, Germany) connected to a computer in order to measure the permeate flux.

2.3.2. Clarification procedure

For each experiment, a new membrane was used. The membranes (100 kDa, 0.15, 0.2 and 0.45 μm) were soaked in ethanol solution (50%) for 30 min, then washed with deionized water and soaked in deionized water for at least 24 h prior to use. Membranes were pre-pressured with deionized water for 60 min under a pressure of 200 kPa at 25 °C and then the pure water flux of membranes was measured at TMP of 50, 100, 150 and 200 kPa.

Experiments were carried out in both full recycling mode and concentration mode. For full recycling tests, 4 L of feed juice was used. Before the experiments started, feed juice was heated to 50 °C, and the membrane was re-stabilized, at a feed flow rate of 120 L h\(^{-1}\), TMP of 75 kPa, at rotating speed of 2000 rpm during 20 min. After stabilization, the rotating speed was decreased from 2000 to 1500, 1000 and then 500 rpm. Under each rotating speed, TMP was increased gradually from 75 to 150 kPa and then decreased directly back to 75 kPa to study the flux decline. Samples were collected at 75 and 150 kPa for each rotating speed. After filtration, the RDM system was cleaned with water for 30 min and then with a P3-ultrasil 10 (Ecolab, USA) detergent at two concentrations (0.1% and 0.5%, thus, pH = 10 and 11, respectively). After cleaning, pure water flux was measured again at rotating speed of 500 rpm.

In concentration tests, 12 L feed juice were concentrated to a retentate of 1.2 L. Feed juice was heated to 50 °C, before the experiments started. Then the membrane was re-stabilized with full recycle mode, at a rotating speed of 2000 rpm and a TMP of 100 kPa for 20 min. After stabilization, a concentration test was carried out under same operating conditions. Samples were taken from every

<table>
<thead>
<tr>
<th>Component concentration (%)</th>
<th>Value</th>
</tr>
</thead>
<tbody>
<tr>
<td>Dry mass</td>
<td>12.3–12.9</td>
</tr>
<tr>
<td>Soluble matter</td>
<td>8.5–12</td>
</tr>
<tr>
<td>Inulin</td>
<td>9–11</td>
</tr>
<tr>
<td>Carbohydrate</td>
<td>11.5–12</td>
</tr>
<tr>
<td>Pectin</td>
<td>NA</td>
</tr>
<tr>
<td>Protein</td>
<td>0.2–0.4</td>
</tr>
</tbody>
</table>

<table>
<thead>
<tr>
<th>Solute molecular weight range (kDa)</th>
<th>Value</th>
</tr>
</thead>
<tbody>
<tr>
<td>Inulin</td>
<td>0.34–11.2</td>
</tr>
<tr>
<td>Pectin (Robert et al., 2008)</td>
<td>300–800</td>
</tr>
<tr>
<td>Protein</td>
<td>14.2–66 (Cyr and Bewley, 1990)</td>
</tr>
<tr>
<td>pH (at 25 °C)</td>
<td>5.3–5.8</td>
</tr>
<tr>
<td>Viscosity (mPa s)</td>
<td>1.7 ± 0.3 (25 °C), 0.92 ± 0.25 (50 °C)</td>
</tr>
</tbody>
</table>
0.5 L (0.3 L when retentate less than 1.5 L) of collected permeate for analysis.

2.4. Analytical methods

Turbidities of retentate and permeate were measured with a Ratio Turbidimeter (Hach, USA). Soluble matter contents (°Brix) were measured by means of a digital refractometer PR-32a (ATAGO Co., Ltd., Japan). Carbohydrate contents of samples were measured by the phenol sulphuric acid method (Dubois et al., 1956; Wei et al., 2007) using inulin (Arcro Organics) as standard.

2.5. Calculated parameters

The permeate flux \( J \) was calculated by:

\[
J = \frac{1}{A} \frac{dV_p}{dt}
\]

where \( A \) is the effective membrane area (m²), \( V_p \) is the total volume of permeate (m³), and \( t \) is the filtration time (s).

The mean TMP is obtained by integrating the local pressure over the membrane area as follows (Luo et al., 2010):

\[
TMP = p_c - \frac{1}{4} \rho k^2 \omega^2 R^2
\]

where \( p_c \) is the measured peripheral pressure (Pa), \( \rho \) is the density of the fluid (kgm⁻³), \( k \) is the velocity factor (0.89 for this system), \( \omega \) is the disk angular velocity (rad s⁻¹) and \( R \) is the housing inner diameter (m).

The solute transmission is defined as:

Table 2

<table>
<thead>
<tr>
<th>Membrane</th>
<th>Manufacturer</th>
<th>Surface material</th>
<th>Pore size</th>
<th>Max. temperature (°C)</th>
<th>pH range</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>
</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>
</tr>
<tr>
<td>MV020T</td>
<td>Microdyn-Nadir</td>
<td>PVDF</td>
<td>0.2 μm</td>
<td>95</td>
<td>2–11</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>
</tr>
</tbody>
</table>

PVDF: Polyvinylidene fluoride
PSH: Permanently hydrophilic polysulphone.

Fig. 1. Schematic diagram of the RDM (a) and experiment set-up (b).
Transmission(%) = \frac{C_p}{C_{R,av}} \times 100 \tag{3}

where \(C_p\) is the solute concentration in permeate (%), and \(C_{R,av}\) is the average concentration in retentate during the filtration period (%).

The flux decline can be expressed as a percentage of feed permeate flux decrease after TMP stepping operations (75 kPa): 

\[
FD = \frac{J_{ai} - J_{af}}{J_{ai}} \times 100
\tag{4}
\]

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

The Volume Reduction Ratio (VRR) is defined as:

\[
VRR = \frac{V_i}{V_c}
\tag{5}
\]

where \(V_i\) is initial feed volume (m\(^3\)) and \(V_c\) concentrate volume (m\(^3\)).

3. Results and discussion

3.1. Dead end filtration

The variation of filtrate volume with filtration time for various membranes is presented in Fig. 2. Permeate fluxes at the beginning period (45 s) and after 2400 s of filtration are also shown. As expected, the initial permeate flux increased with membrane pore size, but at the end of filtration, permeate fluxes for different membranes were fairly close. This can be explained by the different separation mechanisms and fouling situations at different filtration stages. During the initial filtration stage, since the membrane is relatively clean, the permeate flux is governed by membrane permeability (e.g. pore size and hydrophilicity) under same operating conditions. While, at the end of filtration, the foulants, i.e. pectin, proteins and suspended solids, deposit tightly on the membrane, resulting in a thick and compact cake layer, and the permeate flux is mainly controlled by cake fouling layer. In order to study the fouling behavior during filtration, Eq. (7), which can be deduced from Eq. (6) (Wan et al., 2012), was used to model the filtration behavior.

\[
t = \frac{t}{P_{tm}K} \ln \left( \frac{KP_{tm}r + 1}{\mu R_m} \right)
\tag{6}
\]

where \(P_{tm}\) is the membrane resistance (m\(^{-1}\)), \(K\) is the exponential fouling coefficient (m\(^{-1}\)), \(r\) is an empirical constant defined by De La Garza and Boulton (1984), presenting the rate of filtration resistance increase, \(A\) is the filtration area (m\(^2\)), \(P_{tm}\) is the mean transmembrane pressure (Pa) and \(\mu\) is the viscosity (Pa s) of feed. This model, proposed by De La Garza and Boulton (1984), was successfully used for filtration of soluble rice bran fibers, apple juice and stevia extract (Gokmen and Cetinkaya, 2007; Reis et al., 2009; Wan et al., 2012).

By fitting modeling results (dashed lines in Fig. 2) and experimental data (closed points in Fig. 2), fouling coefficient, \(K\), can be calculated and is presented in Table 3. The values of correlation coefficients (>0.99) show good agreement of the model with experimental results. As seen in Table 3, the fouling coefficients for four different membranes are comparable, indicating that permeate flux was mainly controlled by the compact fouling layer. However, the fouling coefficient slightly decreases with increase of membrane pore size, implying that more foulants are retained by the membrane with smaller pore size and deposit on the membrane surface.

The permeate turbidity, transmissions of carbohydrate and soluble matter are listed in Table 4, in order to evaluate the clarification performance of used membranes and carbohydrate and soluble matter losses during the filtration. Permeate turbidity shows that effective clarification can be achieved by filtration, especially when using the 100 kDa membrane. The value of turbidity can be decreased to 3.2 NTU compared with a feed turbidity superior to 300 NTU after 10 times dilution. However, with increase of pore size, the transmissions of carbohydrate and soluble matter were higher and the inulin loss after clarification by membrane is much less. From a practical point of view, membranes with high permeate flux, low permeate turbidity and high carbohydrate transmission are preferable. Because the membrane performance is mainly governed by fouling layer in dead end filtration with low stirring speed, the membrane selection should be further verified in RDM filtration.

3.2. RDM filtration with full recycling

Fig. 3 shows effects of rotating speed and TMP on permeate flux and turbidity for RDM filtration. Compared with dead end filtration (Fig. 2), permeate fluxes during RDM filtration are obviously higher. It can be noted that a higher rotating speed can result in bigger permeate flux. Higher rotating speed permits to generate higher shearing force on membrane surface, which can help to remove or reduce the membrane surface fouling, and therefore, a higher permeate flux can be obtained. For a membrane of fixed pore size, significant increase of flux with TMP can be observed at high rotating speed (1500 or 2000 rpm). However, this trend disappears when the rotating speed decreases to 500 and 1000 rpm. Higher TMP can offer more driving force and result in higher permeate flux. But a fouling layer of higher thickness or density on membrane surface can be formed at higher TMP, thus there exists a compromise between positive and negative effects of TMP increase.

<table>
<thead>
<tr>
<th>Pore size</th>
<th>Fouling coefficient (m(^{-1}))</th>
<th>Correlation coefficient ((r^2))</th>
</tr>
</thead>
<tbody>
<tr>
<td>100 kDa</td>
<td>62.17</td>
<td>0.992</td>
</tr>
<tr>
<td>0.15 µm</td>
<td>58.45</td>
<td>0.994</td>
</tr>
<tr>
<td>0.2 µm</td>
<td>55.65</td>
<td>0.992</td>
</tr>
<tr>
<td>0.45 µm</td>
<td>53.58</td>
<td>0.995</td>
</tr>
</tbody>
</table>
on permeate flux. At high rotating speeds, the fouling layer can be effectively controlled and the positive effect of TMP increase is dominant. While at low rotating speeds, further increase of TMP will not increase flux, which means that additional fouling cancels the enhanced driving force. This maximum stationary permeate flux is called “limiting flux” (Luo et al., 2012b), which should be avoided in practical operations.

Permeate turbidity is significantly decreased by membrane filtration, as shown in Fig. 3, compared with turbidity of the feed. For US100P, FSM0.15PP and FSM0.45PP membranes, permeate turbidity was near 1.0 NTU and irrespective of TMP (except for FSM0.45PP at 2000 rpm). However, for the MV020T membrane, permeate turbidity was higher than others and increased with TMP, especially at 2000 rpm, which was probably caused by the wide pore size distribution of this membrane. As seen in Fig. 3c, permeate turbidity decreased at lower rotating speeds due to fouling layer formation.

The results of carbohydrate transmission for different membranes are presented in Table 5, showing that carbohydrate transmission goes up with increase of membrane pore size. At a fixed rotating speed, carbohydrate transmission is higher for lower TMP. For example, when TMP increased from 75 to 150 kPa, the carbohydrate transmission of FSM0.45 pp membrane decreased by 10% at 2000 rpm, 11% at 1500 rpm, 12% at 1000 rpm and 16% at 500 rpm. This tendency can be explained by two transport mechanisms—convection and diffusion. The first was controlled by permeate flux due to the “dilution effect”, where more solvent (i.e. water) that passed through the membrane at higher TMP could dilute permeate, resulting in a lower solute concentration; the second was governed by solutes diffusion across fouling layer and membrane, and the denser fouling layer formed on the membrane at higher TMP could serve as an additional selective membrane, causing a higher solute retention. Both factors would increase carbohydrate loss in filtration retentate. At 2000 rpm, the first factor is dominant (as flux increased by 30% from 75 to 150 kPa) and the second is negligible at such high shear rate, while at 500 rpm, the first factor had no effect on carbohydrate transmission because the permeate flux did not increase at higher TMP (see Fig. 3).

According to the permeate flux and turbidity (Fig. 3) and carbohydrate transmission (Table 5), a 0.45 μm membrane and rotating speed of 2000 rpm were selected for concentration experiments with low applied pressure. In order to compare with dead end filtration, the same TMP (100 kPa) was chosen for the concentration test.

Table 4

<table>
<thead>
<tr>
<th>Membrane</th>
<th>100 kDa</th>
<th>0.15 μm</th>
<th>0.2 μm</th>
<th>0.45 μm</th>
</tr>
</thead>
<tbody>
<tr>
<td>Carbohydrate transmission (%)</td>
<td>85.0</td>
<td>88.4</td>
<td>99.9</td>
<td>96.2</td>
</tr>
<tr>
<td>Soluble matter transmission (%)</td>
<td>91.6</td>
<td>92.7</td>
<td>93.7</td>
<td>94.1</td>
</tr>
<tr>
<td>Permeate turbidity (NTU)</td>
<td>3.2</td>
<td>9.6</td>
<td>12.5</td>
<td>12.6</td>
</tr>
</tbody>
</table>

Fig. 3. Flux and permeate turbidity at different TMP (75, 100, 125, and 150 kPa) for membranes (a) US100P, (b) FSM0.15PP, (c) MV020T and (d) FSM0.45PP.
3.3. Comparison of dead end and RDM filtrations

Permeate fluxes for dead end (350 rpm) and RDM filtrations (500–2000 rpm) are shown in Fig. 4, indicating that permeate flux is governed by membrane characteristics (e.g. pore size) at high rotating speeds, but at rotating speeds of 500 and 350 rpm, fluxes seem identical for these four membranes with different pore size. It has been confirmed by previous researchers that not only the membrane but also the deposited layer can affect the flux (Rai et al., 2010). In this case, when at a high rotating speed, due to the strong shear-induced back transport of foulants, the fouling layer is negligible or very thin and loose, and thus the permeate flux is controlled by the membrane, while at a low rotating speed, the foulants deposit easily on the membrane, resulting in a thick and compact cake layer, and thus the permeate flux is mainly controlled by cake fouling layer. Therefore, a high-shear membrane filtration with RDM has obvious advantages in clarification of chicory juice, not only for increasing permeate flux, but also for minimizing membrane fouling and improving inulin transmission.

3.4. Fouling and cleaning of membranes

In order to study the membrane fouling during RDM, flux decline, as shown in Fig. 5, was calculated according to Eq. (4) when TMP increased gradually from 75 to 150 kPa and then returned directly to 75 kPa. The flux declines for different membranes are very similar at same rotating speeds, implying that surface fouling on the membrane (mainly controlled by shear rate) is dominant and pore fouling (mainly governed by pore size) is not very important. For a given membrane, the flux decline decreases with increase of rotating speed. At 2000 rpm, the flux decline was 15%, but reached nearly 40% at 500 rpm. These results confirm that it is necessary to use a high rotating speed of 2000 rpm for concentration experiments.

### Table 5

<table>
<thead>
<tr>
<th>Rotating speed (rpm)</th>
<th>TMP (kPa)</th>
<th>100 kDa</th>
<th>0.15 μm</th>
<th>0.2 μm</th>
<th>0.45 μm</th>
</tr>
</thead>
<tbody>
<tr>
<td>2000</td>
<td>75</td>
<td>78.5</td>
<td>78.5</td>
<td>97.0</td>
<td>97.8</td>
</tr>
<tr>
<td>150</td>
<td>78.7</td>
<td>65.7</td>
<td>90.1</td>
<td>87.9</td>
<td></td>
</tr>
<tr>
<td>1500</td>
<td>75</td>
<td>92.5</td>
<td>74.4</td>
<td>84.0</td>
<td>94.0</td>
</tr>
<tr>
<td>150</td>
<td>85.9</td>
<td>70.3</td>
<td>83.2</td>
<td>83.5</td>
<td></td>
</tr>
<tr>
<td>1000</td>
<td>75</td>
<td>82.1</td>
<td>86.2</td>
<td>90.8</td>
<td>89.6</td>
</tr>
<tr>
<td>150</td>
<td>78.9</td>
<td>83.8</td>
<td>90.1</td>
<td>79.0</td>
<td></td>
</tr>
<tr>
<td>500</td>
<td>75</td>
<td>82.3</td>
<td>83.2</td>
<td>87.6</td>
<td>92.1</td>
</tr>
<tr>
<td>150</td>
<td>76.3</td>
<td>73.2</td>
<td>75.1</td>
<td>77.4</td>
<td></td>
</tr>
</tbody>
</table>

Fig. 4. Flux versus rotating speed for membranes with various pore sizes, TMP was fixed at 100 kPa.

Fig. 5. Flux decline (at TMP = 75 kPa) during filtration with membranes with different pore size at 100 kDa, 0.15, 0.2 and 0.45 μm.

3.3. Comparison of dead end and RDM filtrations

Fig. 6. Flux recovery by water cleaning and chemical cleaning at different cleaning agent concentration.

Fig. 7. Filtration flux and permeate characteristics (turbidity and soluble matter content (CSM)) versus VRR during concentration process.
Cleaning by water and P3-ultrasil 10 (Ecolab, USA) detergent at different concentration was carried out to study the regeneration ability. As presented in Fig. 6, flux recovery, defined as the ratio of water flux before clarification and after cleaning, was used to evaluate the efficiency of these three cleaning methods. After water cleaning, 60% water flux can be regained. But after chemical cleaning of 30 min at a concentration of 0.5%, flux recovery can reach 90%. This high regeneration of membrane permeability shows a potential application in industrial juice clarification.

3.5. Concentration tests

Based on previous experiments in full recycling mode, a high rotational speed (2000 rpm) and membrane with the largest pore size (0.45 µm) were selected for concentration tests. As shown in Fig. 7, the flux remained nearly stable when VRR varied from 1 to 3, and then decreased linearly with VRR in semi-log coordinates when VRR increased from 5 to 10, which corresponded to the mass transfer limited regime (Luo et al., 2010). It should be noted that the flux in concentration mode was lower than that for full recycling experiments in spite of using the same membrane, TMP and rotating speed. One possible explanation might be the different stabilization method at the beginning of these experiments. Compared with a TMP of 75 kPa, which was the stabilization TMP in full recycling mode, a denser fouling layer may be formed when 100 kPa was used for stabilization in concentration mode.

Fig. 7 also shows the variation of soluble matter content and permeate turbidity at various VRR. The increase of soluble matter with VRR reflects the soluble matter augmentation in feed when VRR increased. The turbidity decreased initially and then increases at the end stage, and the same tendency was also found in concentration of dairy effluent by US100P membrane (Luo et al., 2012c). This can be explained by the first stage fouling and the increase of impurity content at the end stage.

Characteristics of feed, permeate and retentate at VRR = 2 and 10, are presented in Table 6. Compared with dead end filtration, at the same VRR (=2), permeate from RDM filtration has both a high carbohydrate content (9.88%) and low turbidity (3.4 vs. 12.6 NTU for dead end filtration). RDM permitted to achieve a high VRR of 10, with high permeate flux. And 10.8 L permeate with turbidity of 3.9 was obtained. Large differences in appearance and color for feed, permeate and retentate were observed in experiment.

<table>
<thead>
<tr>
<th>Sample</th>
<th>Feed</th>
<th>Permeate (VRR = 2)</th>
<th>Retentate (VRR = 2)</th>
<th>Permeate (VRR = 10)</th>
<th>Retentate (VRR = 10)</th>
</tr>
</thead>
<tbody>
<tr>
<td>Carbohydrate (%)</td>
<td>10.85</td>
<td>9.88</td>
<td>13.91</td>
<td>10.79</td>
<td>15.68</td>
</tr>
<tr>
<td>Soluble matter (%)</td>
<td>10.2</td>
<td>9.5</td>
<td>11.3</td>
<td>10</td>
<td>16</td>
</tr>
<tr>
<td>Turbidity (NTU)</td>
<td>&gt;300</td>
<td>3.4</td>
<td>&gt;300</td>
<td>3.9</td>
<td>&gt;300</td>
</tr>
</tbody>
</table>

4. Conclusions

The RDM was applied to clarification of chicory juice obtained from pilot diffusion assisted by PEF pretreatment. Compared with dead end filtration, the RDM showed the advantage of higher permeate flux and lower permeate turbidity, resulting from high membrane shear rate due high rotating speed, which also reduced membrane fouling and led to higher carbohydrate transmission. At rotating speeds of 1500–2000 rpm, the permeate flux increased with pore size, while at low rotating speeds of 350–500 rpm, it was low and independent of membrane type, as it was limited by cake fouling. Higher TMP caused lower carbohydrate transmission due to “dilution effect” by higher flux at high rotating speed or “retarding effect” by a thicker fouling layer at low rotating speed. For F5045PP membrane, the highest carbohydrate transmission (98%) was obtained at 2000 rpm and TMP = 75 kPa while permeate turbidity was 2.4 NTU. Feed juice can be concentrated to VRR = 10, still with a high flux (106 Lm⁻²h⁻¹), and the resulting permeate was both high in carbohydrate (10.8%) and well clarified (3.9 NTU). The pure water flux of used membrane can be effectively recovered to over 90% by cleaning with a 0.5% P3-ultrasil 10 detergent solution. Therefore, its throughput, moderate flux decline and perfect product quality make membrane clarification by RDM suitable to treat raw chicory extract in industry and these results from laboratory-scale tests could serve as valuable guide for extrapolating the process to industrial production.

Acknowledgements

The authors would like to acknowledge COSUCRA, Belgium for the kind supply of chicory roots and also would like to acknowledge the financial support of China Scholarship Council for Jianquan Luo’s and Zhenzhou Zhu’s thesis fellowship. The authors thank Alfa Laval Naksakov A/S Company for supplying the membranes.

References


