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Research Paper

Refinement of Cane Molasses with Membrane Technology for Clarification and Color Removal

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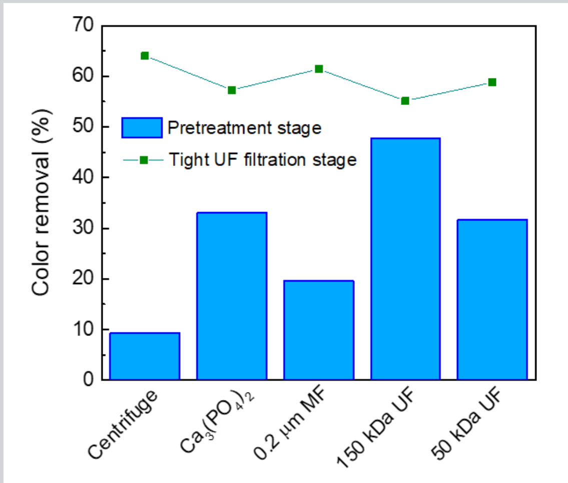
Highlights

- Decoloration of cane molasses with tight 2 kDa UF
- Sugar recovery increased using operation modes with diafiltration
- Improved flux obtained with pretreatment using 50 kDa UF

Graphical abstract



Abstract

Recovery of sugars from cane molasses (i.e. the by-product of sugar industries) is of great interest to industry and academia. The prerequisite for refining cane molasses is removal of pigments and suspended solids present in the molasses. In this work, the utilization of membrane separation for clarification and decoloration of cane molasses has been examined. Three operation modes (dilution-concentration, dilution-concentration-diafiltration, and dilution-diafiltration-concentration) were employed for color removal with a tight 2 kDa ultrafiltration (UF) membrane. Results showed that the operation modes with diafiltration could result in higher sugar recovery than dilution-concentration mode, though the latter had the higher permeate flux. Then, in order to further improve the permeate flux of the 2 kDa UF, five pretreatments were carried out to remove suspended solids and large pigments. It was found that the pretreatment with ceramic membrane filtration was better than centrifuge and precipitation, particularly in terms of permeate flux for the tight UF membrane. Although the permeate flux for the tight UF after a UF pretreatment equipped with a 150 kDa UF membrane was higher than that with a 50 kDa UF membrane, the permeate flux during the pretreatment was the highest for the 50 kDa UF. The obtained decolorized syrup can be further purified by nanofiltration for the separation of sucrose and reducing sugars. Thus, refinement of cane molasses with membrane technology provides an alternative to conventional refinement methods and shows promising prospects of industrial application.

1. Introduction

Cane molasses is the mother liquid from cane sucrose crystallization process where no more sugar can be obtained economically at sugar mills. As the by-product of sugar manufacturing, cane molasses is sold at substantially cheaper price than refined sugar. The compositions of cane molasses vary significantly due to the difference in cane type, growing conditions of cane and production processes. Generally, it is characterized by high sucrose concentration varying between 30-40% (dry weight) and high reducing sugars (mainly glucose and fructose) contents in the range of 15-20% (dry weight). In addition, cane molasses also contains pigments, crude protein, amino acid, minerals and trace amount of vitamins, etc. [1]. Due to the high sugar content

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and a variety of nutritional elements, cane molasses has been widely used for feeding animals [2] and culturing microbial fermentation [3].

The high sucrose concentration in the cane molasses has also promoted sugar manufacturers and researchers to develop high-efficient methods for recovering sucrose from the final molasses to increase the profitability of sugar mills [4, 5]. However, because of the high invert sugar concentrations (glucose and fructose), as well as the presence of large amounts of suspended solids, inorganic ions and high color, separation of sucrose from cane molasses is difficult and only at the margin of economic viability, in sharp contrast to recovery of sucrose from beet molasses [6]. Totally different from cane molasses, beet molasses is generally high in sucrose contents (50-60%, dry weight) and low in reducing sugars concentrations (0.5-1.5%, dry weight) and suspended solids [4], which makes desugarization of beet molasses relatively easier and more economically viable. Separation of sucrose from beet molasses using chromatographic technology has become the standard practice in beet sugar mills [7]. It was reported that ion-exclusion chromatography could allow extracting as high as 90% of sucrose present in the beet molasses with a purity of up to 90% [8].

However, recovery of sucrose from cane molasses faces more challenges when compared to state of the art methods used for desugaring beet molasses. Although chromatographic separation of beet molasses has been well established in beet sugar industries, it is not cost-efficiently used in the cane sugar mills resulting from the high contents of impurities such as suspended matters, divalent ions and pigments present in cane molasses, which can severely foul the resin and shorten its life. Despite that previous references have reported the removal of suspended solids using centrifugation combined with heating, flocculation, and chemical treatment as well as removal of divalent ions using ion-exchange resins [9], so far there are still not cost-effective and efficient pretreatment methods available to make chromatographic treatment of cane molasses for recovering sucrose commercially successful.

Membrane separation technology has been widely used in food industry, but it does not find its large-scale application in cane sugar industry. Treatment of sugarcane juice with membrane filtration has been extensively investigated in the past nearly 50 years [10]. Most recently, an integrated membrane process was proposed for refinement of sugarcane juice compromising loose ultrafiltration (UF) for clarification, tight ultrafiltration for decoloration and nanofiltration (NF) for purification and concentration of sucrose [11]. However, to the best of our knowledge, few investigations dealt with cane molasses by using membrane technology. Kochergin [12] tested the microfiltration (MF) of cane molasses from various sources and found that the filtrated stream was adequately used as the feed for chromatographic separator. Thompson [9] reported the use of 40 kDa ceramic UF for removing suspended solids from cane B-molasses and followed by removal of Ca^{2+} from the UF permeate using a strong acid cation exchange resin. Even though the physicochemical properties of beet molasses are less complex than that of cane molasses, there are also limited references regarding the membrane treatment of beet molasses. Bernal et al. [13] applied an activated charcoal/UF process to remove pigments present in beet molasses. Color removal of 96.5% was achieved by the activated charcoal dynamic membrane formed on the surface of 100 kDa ceramic membrane surface.

Cane molasses is a highly viscous liquid with dark brown color. The pigment mainly results from melanoidins [14], which can take part in Maillard reactions during storage of cane molasses, thus causing changes in the color and compositions of molasses. The main aim of the present study was to evaluate the use of tight 2 kDa UF membrane for color removal from cane molasses, with focus on utilization of diafiltration to increase the permeate flux and sucrose recovery. Meanwhile, in order to further increase the flux and reduce the membrane fouling during tight 2 kDa UF decoloration, five pretreatments including centrifuge, chemical precipitation, ceramic MF and loose ceramic UF, were employed to remove the suspended matters before color removal.

2. Experimental

2.1. Materials

Molasses was provided by a cane sugar mill in Guangdong Province, China. Ceramic MF membrane with a pore size of 0.2 μm and ceramic UF membranes with molecular weight cut-offs (MWCOs) of 50 and 150 kDa were purchased from TAMI industries, France. These ceramic membranes are tubular with an area of 4.7×10^{-3} m^2 for MF membrane and 1.1×10^{-2} m^2 for UF membranes. The tight UF membrane used for color removal was 2 kDa spiral wound polyethersulfone (PES) membrane purchased from Sepro membranes, USA. This polymer membrane is tolerant to temperatures as high as 70 °C and have an area of 0.34 m^2. All the used chemical reagents were of analytic grade and purchased from Beijing Chemical Works, China.

2.2. Experimental Set-up

The home-made apparatus equipped with ceramic and polymeric membranes were used for pretreatment and purification of molasses, respectively. The ceramic membrane was equipped with a feed tank having a working volume of 600 mL, the pressures before and after the membrane module were read from the pressure gauges installed at the inlet and outlet of modules. Transmembrane pressure (TMP) was calculated as the average of inlet and outlet pressure. A digital flow meter installed after the module displayed the cross velocity, while for the polymeric membrane equipment, the feed tank with a capacity of 5 L was used. TMP and cross velocity were read from the operating panel and flow meter, respectively. Feed tanks of both ceramic and polymeric membrane set-ups had jackets through which the temperature of the feed was controlled by circulating bath water.

2.3. Filtration of cane molasses by tight 2 kDa UF

Three operation modes with 2 kDa tight polymeric membrane were carried out to compare the filtration performance in terms of flux, color removal and sucrose recovery. The parameters of those three operation modes are shown in Table 1. A total of 4200 mL deionized water was added to 800 mL molasses at different stages. In experimental run 1, 4200 mL deionized water was directly added to 800 mL molasses for dilution before starting UF operation. Whereas in runs 2 and 3, 4200 mL deionized water was added separately. In run 2, 800 mL molasses was diluted with 2400 mL of deionized water, and after the 2400 mL permeate was collected, another 1800 mL deionized water was added to the feed tank for diafiltration. While in run 3, 800 ml deionized water was first used for dilution before UF and another 3400 mL deionized water was used as diafiltration solvent during the entire UF process. For all the operation modes, 4200 ml permeate was obtained at the end of UF process.

In above experiments, the pH of molasses solution was adjusted to 7.0 after dilution of molasses in order to prevent the inversion or hydrolysis of sucrose to its components glucose and fructose at acid pH, and then centrifuged at 8000 rpm for 20 min. Tight UF was operated at a temperature of 60 °C and a TMP of 10 bar. After each use, UF membrane was first rinsed with deionized water and then thoroughly cleaned with chemical reagent to ensure that the water permeability differences before and after filtration were within 5%. The tight 2 kDa UF membrane was cleaned with 0.5% alkaline cleaning agent for 30 min followed by another 30 min cleaning with 200 ppm NaClO solution.

Table 1
Three operation modes for filtration of molasses solution by tight UF membrane at 60 °C and 10 bar (Run No.1: dilution-concentration; Run No.2: dilution-concentration-diafiltration; Run No.3: dilution-diafiltration-concentration).

Run	Molasses volume （mL）	Dilution water (mL)	Diafiltration water (mL)	Total permeate volume (mL)
1	800	4200	0	4200
2[a]	800	2400	1800	4200
3[b]	800	800	3400	4200

[a] diafiltration operation started after feed solution was concentrated 5.33 times
[b] diafiltration operation started at the beginning of UF.

2.4. Pretreatment of cane molasses

Five pretreatments were conducted in attempts to improve the flux of UF of molasses solution. These pretreatments include centrifuge, chemical precipitation and ceramic membrane filtration. Before each pretreatment, the raw molasses was diluted with three-times volume of deionized water followed by adjusting its pH to 7.0. Centrifuge was operated at 8000 rpm for 20 min. Chemical pretreatments were conducted as follows: after mixed with 1% (w/v) $Ca_3(PO_4)_2$, the diluted molasses solution was boiled for 8 min and then cooled to room temperature, then followed by centrifugation at 8000 rpm for 10 min. Three ceramic membranes (0.2 μm, 50 and 150 kDa) were used to pretreat the diluted molasses solution under concentration mode. The experiments were carried out at a TMP of 2 bar, a temperature of 70 °C and a cross velocity of 11.7 L/min. After each pretreatment, the clarified molasses solution was filtrated using tight 2 kDa polymeric UF membrane under continuous concentration mode.

To evaluate the cleaning effect, water permeability was measured and compared before each filtration test of molasses solution and after chemical cleaning, respectively. All the ceramic membranes were cleaned with 2%

(w/v) NaOH solution at 80 °C for 30 min. The filtration and cleaning conditions of tight 2 kDa UF membrane was the same as that described previously (section 2.3).

2.5. Analytical methods

Brix is typically used to measure the refractometric dry substance in solutions. It is defined as the amounts of dissolved solids per weight of total solution. In sugar industry, Brix is widely used to roughly estimate the sugar contents of an aqueous solution. Brix of feed, retentate and permeate were determined by a digital refractometer (WZ-108, Beijing Wancheng Beizeng Precision Instrument Co., Ltd., China).

The color of molasses solution was determined spectrophotometrically at 560 nm using a UV-9000S spectrophotometer (Shanghai Yuanxi Instruments Co., Ltd., China). Color removal is defined as the difference in the absorbance units of feed and permeate divided by the absorbance unit of the feed. The turbidity was measured using a Hach 2100Q portable Turbidimeter. The content of reducing sugars was quantified by the method of Miller [15] with dinitrosalicylic acid reagent. The concentration of total carbohydrates was measured by phenol-sulfuric acid method as described by Nielsen [16]. Sucrose content was estimated from the difference in concentrations of total carbohydrates and reducing sugars. The pH of molasses solution was measured by Seven Compact S210 pH meter (Mettler Toledo, Switzerland) fitted with a temperature compensator.

3. Results and discussion

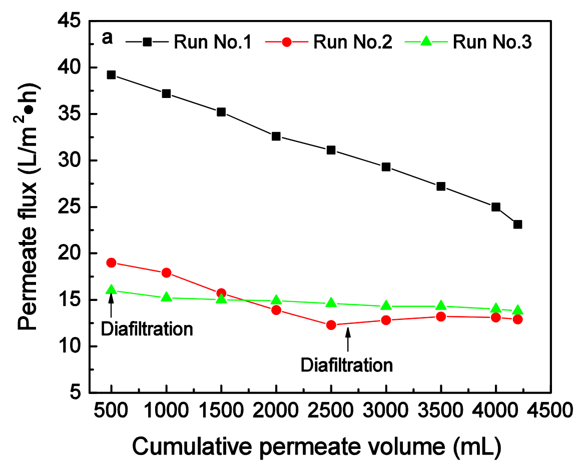
3.1. Filtration of molasses solution by tight 2 kDa UF membrane for color removal

Color value is widely recognized as the sole determinant of grade and quality of sugar. Lower color value means that cane sugar is subject to more processing, thus the sugar product with less pigments has more superior quality. In light of this, removal of colorant in the molasses solution was investigated by tight UF membrane with a MWCO of 2 kDa, which exhibited excellent ability of decoloring sugarcane juice in our recent study [11]. However, our study also found that this 2 kDa UF membrane showed partial rejection of sucrose. This can result in low sugar recovery in the UF permeate. In the present work, the 2 kDa UF membrane was used for color removal of molasses solution as well. Three different operation modes, dilution-concentration, dilution-concentration-diafiltration and dilution-diafiltration-concentration, were employed to improve the sugar recovery. Dilution strategy aimed to enhance permeate flux, and diafiltration strategy was used to recover sugars from the concentrate. While concentration operation before diafiltration was conducted to reduce the volume of UF concentrate, which, in return, decreased the volume of deionized water used for subsequent diafiltration.

The results of molasses solution filtration with above-mentioned three operation modes are shown in Figure 1-a. As expected, under dilution-concentration mode, the filtration of diluted molasses demonstrated the highest permeate flux, although the permeate flux declined by 41.07%, from 39.20 to 23.10 L/m^2·h, with increasing cumulative permeate volume. As for the second operation mode, the permeate flux during concentration of molasses solution diluted with 3 times volume of deionized water decreased from 19.60 to 12.30 L/m^2·h. The following diafiltration, however, progressively increased the permeate flux to 13.00 L/m^2·h. The permeate flux decline during concentration process in the first and second operation modes was likely due to the membrane fouling as well as increased osmosis pressure resulting from accumulated sugars and pigments in the concentrate. When diafiltration was performed, the rejected sugars gradually washed out through the membrane, leading to reduced osmosis pressure, which might be the main cause of gradual increase of permeate flux during diafiltration stage. As far as the third operation mode was concerned, the permeate flux changed a little, slightly decreased from 16.00 to 13.80 L/m^2·h.

Although the diafiltration operation can remove the sugars in the concentrate, resulting in a higher permeate flux, this positive effect on flux was likely offset by the flux decline due to membrane fouling formation, which was caused by pigments, suspended solids, etc. As could be observed in Figure 1-b, a significant difference was observed for sucrose recovery. Run no. 2 had the highest sucrose recovery of 85.21%, followed by Run no. 3 which showed a sucrose recovery of 82.05%. However, Run no. 1 without diafiltration operation had the lowest sucrose recovery of 73.58%. When it came to reducing sugars recovery, it was similar to sucrose recovery, i.e. the reducing sugars recoveries of Run no. 2 and 3 were 10.25% and 6.85% higher that of Run no.1, respectively.

As pointed out by Luo and co-workers, the high permeate flux in Run no.1 had a contradict effect on solute recovery [17]. On the one hand, high permeate flux promotes the formation of concentration polarization layer, which increases the transport of solute across the membrane into the permeate. This can increase the sugar recovery (referred to "concentration polarization effect"); on the other hand, high permeate flux can induce "dilution effect". As it implies, high permeate flux can dilute the solute across the membrane, which in turn increase the solute rejection [18], i.e. decreases the sugar recovery. In addition, membrane fouling could also contribute to decreasing sugar recovery to some extent due to the fact that foulants can block and/or narrow the membrane pores, increasing the solute rejection (referred to "membrane fouling effect"). The low sugar recovery in Run no.1 indicated that "dilution effect" and "membrane fouling effect" greatly outperformed "concentration polarization effect". While in concentration-diafiltration operation mode (Run no. 2), concentration operation was able to decrease the volume of sugar solution. This could lead to higher volume reduction ratio (defined as the volume of feed before diafiltration divided by the volume of diafiltration water) during following diafiltration operation compared to diafiltration-concentration operation mode in Run no.3. Therefore, higher sugar removal efficiency (i.e. higher sugar recovery) was observed in Run no. 2 compared to Run no.3.



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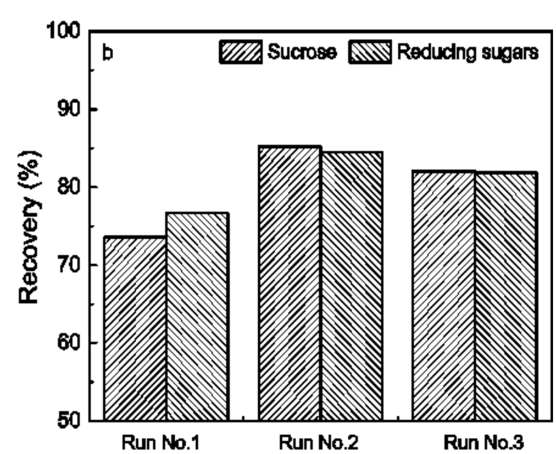


(b)

Fig. 1. Filtration of molasses solution by tight 2 kDa UF membrane at 60 °C and 10 bar: (a) permeate flux, (b) recoveries of sucrose and reducing sugar. Runs No.1-3 are detailed in Table 1.

Table 2 shows the characteristics of tight UF permeates in the three experimental runs. As could be observed Table 2, the color values of the permeates from those three runs did not show significant differences. Brix is a measurement of mass ratio of dissolved solutes in the permeate. It is approximately equal to the mass sum of reducing sugars and sucrose in the tight UF permeate. Higher Brix value means that more sugars are recovered in the UF permeate. The Brix values for the three operation modes were in the following order: Run no.2 > Run no. 3 > Run no. 1. It was in good agreement with recoveries of reducing sugars and sucrose shown in Figure 1-b.

In spite of the relatively high sucrose recovery, Run no. 2 and 3 demonstrated low average permeate fluxes (<15 L/m^2·h), which were too low to meet practical application. In the following studies, different pretreatments were performed to remove impurities present in the molasses in order to improve the permeate flux of the tight 2 kDa UF membrane.

Table 2
Characteristics of tight UF permeates. UF was operated at 60 °C and 10 bar.

Items	Tight UF permeate		
	Run No.1[a]	Run No.2[a]	Run No.3[a]
Brix (%)	14.50	17.50	16.00
Color (absorbance units)	0.21	0.23	0.22
Turbidity (NTU)	0	0	0

[a] Run Nos.1-3 are detailed in Table 1.

Table 3
Characteristics of clarified molasses solutions obtained after five pretreatments. Pretreatment conditions were detailed in Section 2.4.

Items	Feed	Centrifuge	$Ca_3(PO_4)_2$	0.2 µm MF	150 kDa UF	50 kDa UF
Brix (%)	26.00	26.00	26.00	24.50	25.00	25.20
Turbidity (NTU)	38.52	19.26	11.42	0	0	0
Reducing sugar (%)	5.63	5.59	5.61	5.36	5.57	5.46
Sucrose (%)	21.08	21.06	21.11	19.63	20.28	20.06

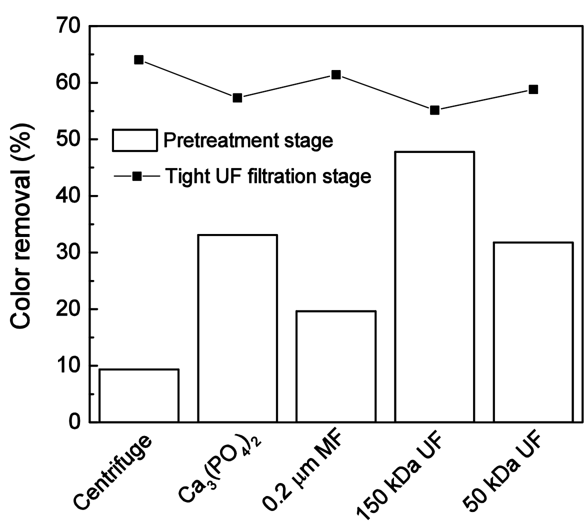


Fig. 2. Color removals of pretreatment and tight UF filtration stages for five pretreatments.

3.2. Pretreatment of molasses solution

Five pretreatments including centrifuge, chemical precipitation by $Ca_3(PO_4)_2$, ceramic membrane filtrations (0.2 µm MF, 150 kDa UF and 50 kDa UF) were compared when treating the molasses. The characteristics of molasses solution after pretreatments are shown in Table 3. As can be seen from Table 3, molasses solution pretreated by centrifuge and chemical reagent had the same Brix value as the feed solution, indicating that centrifuge and chemical precipitation did not lead to loss of sugars, while ceramic membrane pretreatments caused slight loss of sugars due to the membrane retention effect. A turbidity reduction of 100% was obtained for ceramic membrane pretreatments, while centrifuge and chemical precipitation merely achieved turbidity reductions of 50.00% and 70.35%, respectively. Five pretreatments did not cause significant difference in the reducing sugars contents, but led to slight difference in sucrose concentrations, which was in accordance with Brix results.

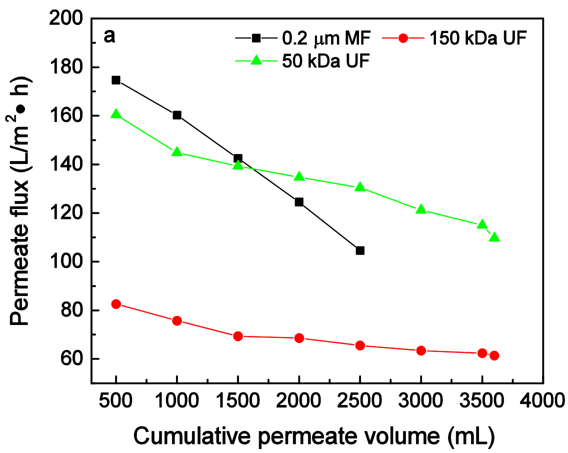
As for color removal in pretreatment stage, it can be seen from Figure 2 that centrifuge pretreatment had the poorest decoloration and it only removed 9.36% of raw color followed by 0.2 µm MF pretreatment, whose color removal was 19.64%. It was interesting to find that color removal of 150 kDa UF reached up to 47.80%, the highest decoloration among the five pretreatments, although the pore size of 150 kDa is larger than that of 50 kDa. This phenomenon was likely caused by the more serious fouling formation for 150 kDa UF membrane, which was confirmed by the lowest permeate flux in the pretreatment stage as shown in Figure 3-a, and the resulting pore narrowing effect and/or cake layer as the additional selective layer could increase the retentions of sugars and pigments (see Figure 2 and Table 3). It was worth mentioning that 0.2 µm MF membrane was seriously polluted after filtrating the molasses solution. The pure water permeability could not be recovered even after thorough alkali cleaning. In sharp contrast to MF, fouling for 150 and 50 kDa UF membranes was less and their pure water permeability could be completed recovered, which suggested their promising potential for molasses pretreatment.

Pretreated molasses solutions were further processed by tight 2 kDa UF membrane under continuous concentration mode to investigate the effects of different pretreatments on tight UF process. It can be seen from Figure 3-b that both centrifuge and chemical pretreatment had the lower permeate flux during tight UF process than ceramic membrane pretreatments, indicating that these two pretreatments were not efficient enough. Among the three ceramic membrane pretreatments, the highest permeate flux of 54.26 L/m^2·h was observed for tight UF of molasses solution pretreated by 150 kDa UF membrane, where permeate flux was 23.09% higher than that pretreated by 50 kDa UF. This observation could be explained by the pore narrowing effect and/or cake layer formation for 150 kDa membrane resulting from severe fouling during pretreatment, which led to better clarification results, in turn, it facilitated subsequent tight 2 kDa UF filtration. The properties of tight UF permeates with five pretreatments are shown in Table 4. Compared to the feed solutions (shown in Table 3), the Brix values of tight UF permeates were 10.00-15.38% lower, showing the partial retention of sugars by tight 2 kDa UF membrane. The contents of reducing sugars and sucrose in the tight UF permeates did not show significant differences when using different pretreatments. As could be observed in Figure 2, color removals after tight UF stage were in the range of 55.16% and 64.05%, with the highest removal rate achieved by centrifuge and the lowest by 150 kDa UF pretreatment. This can be explained by the effect of pretreatments on the fouling situation during tight UF process. In detail, due to the inferior pretreatment result by centrifuge, the obtained molasses solution would cause severe fouling to the tight 2 kDa UF membrane as can be seen from the lowest permeate flux shown in Figure 3-b and this fouling layer could increase the pigment retention and lead to a higher color removal. While for 150 kDa UF pretreatment, since it had the best pretreatment results because of its fouling layer as additional selective "membrane", the permeate flux during tight UF with 150 kDa UF pretreatment was the highest (see Figure 3-b), implying that the fouling evolution for tight UF was the lowest in this case and such a thin fouling layer could not improve the color removal.

Based on the above discussion, it could be clearly seen that molasses solution pretreated by the 150 kDa UF membrane showed the best performance during the following tight UF treatment, especially in terms of permeate flux. However, in filtrating molasses solution, the lowest permeate flux was observed for the 150 kDa UF membrane among the three ceramic membranes. Taking into account of the permeate flux results in the pretreatment stage as well as the decoloration stage, 50 kDa UF membrane might be the best choice for molasses clarification.

4. Conclusions

Refinement of cane molasses with membrane separation technology for clarification and color removal was investigated in the present study. Tight UF with MWCO of 2 kDa membrane could remove the majority of pigments while it also partially rejected sugars. In order to improve the recoveries of reducing sugars and sucrose, three operation modes, that is, dilution-concentration, dilution-concentration-diafiltration and dilution-diafiltration-concentration, were performed with a tight 2 kDa UF membrane. It was found that the dilution-concentration-diafiltration mode led to an average permeate flux of 13.00 L/m^2·h. A slightly higher permeate flux of 13.80 L/m^2·h was obtained for dilution-diafiltration-concentration mode. The permeate fluxes for these two operation modes were 43.72% and 40.26% lower than that for dilution-concentration mode, respectively. The recoveries of sucrose for these two operations with diafiltration, however, were 15.81% and 11.51% higher than the operation without diafiltration. Furthermore, five pretreatments were carried out to compare their effect on the permeate flux in the tight UF decoloration stage. The results showed that pretreatments with three ceramic membranes having difference pore sizes could remove more suspended solids and produced a more clarified molasses solution, which in turn resulted in higher permeate flux than centrifuge and chemical precipitation during tight UF treatment. Pretreatments of molasses with three different ceramic membranes showed the comparable clarification results, while 50 kDa UF membrane demonstrated the best performance compared to 0.2 µm MF membrane and 150 kDa UF membrane, in terms of the highest permeate flux during clarification stage and the acceptable permeate flux during decoloration stage. This study provides some fundamental data for the application of membrane technology in cane molasses refinement. Our further study will be focused on separating sucrose from reducing sugars as well as desalinating reducing sugars solution, with the aim to obtain the sucrose and reducing sugars present in molasses at a high purity.



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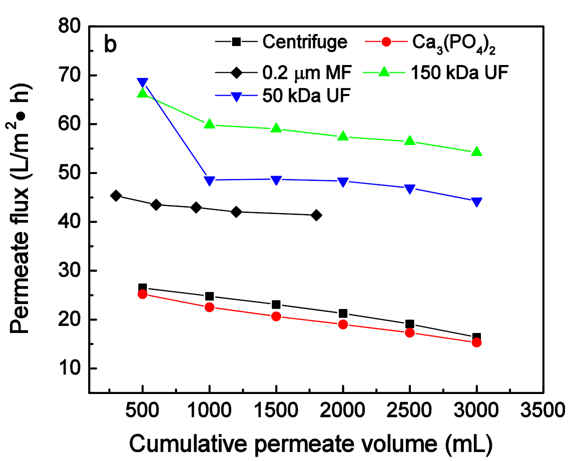

(b)

Fig. 3. (a) Pretreatment of molasses solution by ceramic membranes, and (b) tight UF of molasses pretreated by five pretreatments. Ceramic membrane filtration was operated at 70 °C and 2 bar. Tight UF was operated at 60 °C and 10 bar.

Table 4
Characteristics of permeates obtained from tight UF with five pretreatments. UF was operated at 60 °C and 10 bar. Feed was concentrated 6 times for all pretreatments except 0.2 µm MF pretreatment, where feed was concentrated 4 times.

Pretreatment methods	Centrifuge	$Ca_3(PO_4)_2$	0.2 µm MF	150 kDa UF	50 kDa UF	150 kDa UF	50 kDa UF
Brix (%)	22.00	22.20	22.50	21.80	22.50	25.00	25.20
Turbidity (NTU)	0	0	0	0	0	0	0
Reducing sugar (%)	5.25	5.16	4.79	5.19	4.73	5.57	5.46
Sucrose (%)	16.96	16.85	17.18	17.61	17.68	20.28	20.06

5. Acknowledgement

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