

REMOVAL OF COLOR FROM THE RAW SUGAR MANUFACTURING PROCESS BY MEMBRANE TREATMENT

Chalongsri Flood* and Adrian E. Flood

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ABSTRACT

Color removal from raw sugar was achieved through ultrafiltration (UF) membrane separation of color compounds from the juice exiting the mills, followed by batch crystallization of the purified juice to obtain raw sugar crystals. The process is a potential replacement for the lime/phosphoric acid/SO₂ coagulation and clarification section of the raw sugar process, which produces significant waste material (50 kg mud per ton of raw sugar), and also reduces sugar yield through sugar lost to the settled clarifier mud. It was seen that the process is very good at removing color compounds, with crystals produced from this technique having lower ICUMSA color readings than commercial raw sugar crystals, or raw sugar crystals produced from batch crystallizations of juice clarified by the traditional technique. The most suitable membranes were regenerated cellulose UF membranes with a molecular weight cut-off (MWCO) of 10,000 Daltons (Da), which had suitable permeate flux values and very good removal of color compounds. In the UF separations there was some change in flux due to fouling of the pores of the membrane over a period of about 8 h. Fouling could be reduced in an industrial process by increasing liquid flow velocities over the surface of the membrane using a cross-flow UF membrane.

Keywords: Ultrafiltration, raw sugar, color removal

Introduction

Sugar (sucrose) is one of the highest production high purity chemical products in the world market. The total world production is currently in excess of 140 million tons per year, and the product is typically at 99% purity. Raw sugar is typically produced in regions that have significant areas of sugar cane (Brazil, Australia, South Africa, and Thailand, among others) or sugar beet (North America and Europe) production, and then exported to areas which have insufficient sugar supply in order to be processed into refined sugar, which is used as a domestic

sweetener. Raw sugar may also be used in a number of industrial applications.

Over the previous 5 years, Thailand has had an average yearly production of 6.17 million tons per year, consumption of 1.81 million tons per year, and has exported 4.38 million tons per year, making Thailand the second largest sugar exporting country in the world (behind Brazil) (Koo and Taylor, 2004). Unfortunately the price of bulk sugar on the world market has dropped significantly over the previous decade (from US\$0.17/lb in 1995 to

School of Chemical Engineering, Institute of Engineering, Suranaree University of Technology, 111 University Avenue, Nakhon Ratchasima 30000, Thailand. E-mail: chalongsri@sut.ac.th

** Corresponding author*

US\$0.082/lb in 2005) because of an increase in production by Brazil, and sugar export subsidies and agricultural support programs in the developed world (Kinsella *et al.*, 2005).

A second problem for the Thai sugar industry is that environmental concerns about sugar processing are becoming more strongly emphasized. The net result is that processors will need to produce sugar using more environmentally friendly processes but still produce at very low prices: thus innovation in processing is urgently required.

The major manufacturing processes associated with refining cane sugar have been fully developed for a significant period of time, and it is not expected that the overall process flowchart will undergo very major changes in the foreseeable future (Austin, 1984), however individual units or elements may need to change to some extent as economic variables (such as energy costs) or environmental regulations change. A typical flowchart for the raw sugar manufacturing process is given in Figure 1 (Austin, 1984; Chen and Chou, 1993; The Energy Conservation Center, 2005).

There are several types of materials which are necessary to be removed from the raw sugar manufacturing process: these compounds include salts, invert sugar (fructose and glucose), oligosaccharide sugars (such as raffinose), polysaccharide sugars (particularly dextran and starch), and color compounds. Color compounds are particularly significant because the color of raw sugar is the most significant quality determinant. The color compounds in cane sugar processing have molecular weights in the range of 30 kDa to 1,000 kDa and are larger than those found in beet sugar refining, which are in the size range of 5 kDa to 40 kDa (Godshall *et al.*, 2002; Coca *et al.*, 2004) and in the cane sugar process consist mainly of amino acids, organic acids, caramels, and oligo- and polysaccharides. Several research groups have been actively involved in the study of whether membrane filtration can be used to remove impurities or color from either the beet or the cane raw sugar industries (Kishihara *et al.*, 1981; Decloux and Tatoud, 2000; Karode *et al.*, 2000; Bhattacharya *et al.*, 2001; Ghosh and Balakrishnan, 2003; Hamchi *et al.*, 2003).

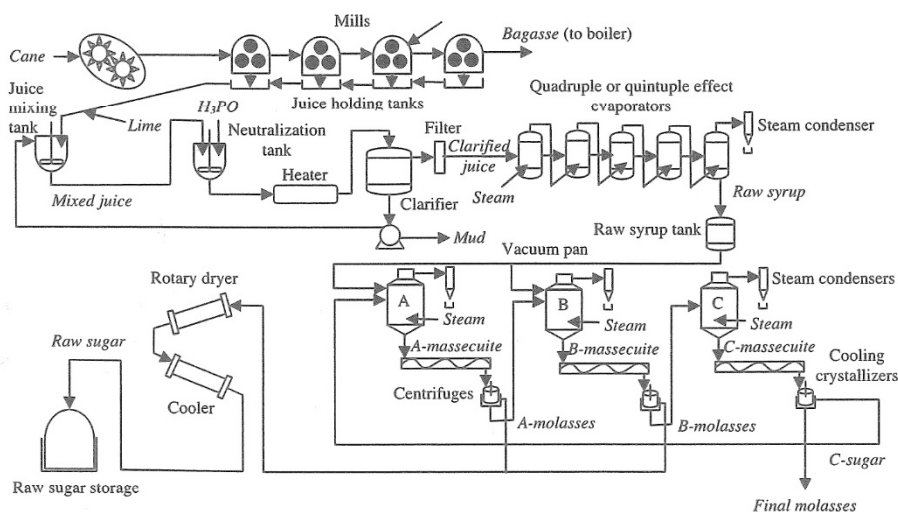


Figure 1. Flow chart for the raw sugar manufacturing process (Austin, 1984; Chen and Chou, 1993; The Energy Conservation Center, 2005). Process streams are named using italic font, and equipment with non italic font. Some auxiliary equipment has been omitted for clarity

The objective of the current research is to determine whether ultrafiltration or microfiltration using a variety of membrane materials, and covering a variety of pore sizes (1,000 to 300,000 Da MWCO for UF membranes and 0.2 and 0.8 μm for MF membranes) is suitable for color removal from the mixed juice in the raw sugar manufacturing process. The main determinate for the quality of the color separation is a comparison of the color of the crystals produced from the permeate product of the membrane process with the color of the crystals prepared from the raw syrup from the factory, which has been purified using the traditional lime treatment process used in the industry. Measurements of the permeate flux and other key parameters of the separation were also performed to determine the viability of the membrane separation process.

Materials and Methods

Materials

The samples used in the research were taken from the process of the Ratchasima Sugar Company. In particular, samples of mixed juice, clarified juice, and raw syrup (Figure 1) were taken from the process, and raw sugar crystals were taken from the raw sugar storage in the factory. Liquid samples were quickly frozen and held at -4°C until required for the experiments, to prevent degradation. Process conditions, such as the Brix, polarization (pol), temperature, purity and reducing sugar contents for various streams, and the color of the product raw sugar crystals, were also monitored during this period. Chemicals required for the process analysis, using methods from the ICUMSA standards (Chen and Chou, 1993), were of ACS grade, as specified in the standard methods.

Commercial membranes of various materials (regenerated cellulose, cellulose triacetate, cellulose acetate, and polysulfone) with pore sizes between 0.2 and 0.8 μm (for microfiltration) and MWCO between 5,000 and 300,000 Da (for ultrafiltration) were purchased from Sartorius AG (Goettingen, Germany) and Millipore Corporation (Billerica, MA, USA).

The membranes used were circular with diameters of 76 mm. Chemical structures of the various membrane materials are shown in Figure 2.

Methods

Membrane separations were performed on a 50 mL stirred cell membrane unit Sartorius AG and a 300 mL stirred cell membrane unit from Millipore Corporation using 76 mm diameter circular membranes. Stirred cells were used so that the mixture on the permeate side of the membrane was well mixed during the separation. The operating pressure for the membrane units was 2.5 bar. Membrane separations were performed after an initial screening using a cloth filter to remove any particulate matter in the process stream. Determination of the permeate flux was carried out at 5 min intervals until the flux reached a constant value. Properties of the permeate stream from the membrane were analyzed for the same properties as the factory samples (pH, Brix, pol, purity, reducing sugars (RS), and reducing sugar ratio (RSR)).

Crystallizations of the membrane permeate streams were performed so that the color of the final product could be determined and compared with the raw sugar crystal product from the current process in the factory, and in-house crystallizations of the raw syrup (the feed to the vacuum pan crystallizers). Evaporation of 800 mL of the juice to the concentrations necessary for crystallization (70% Brix) was performed using a Rotovap evaporator (Buchi Labortechnik AG, Flawil, Switzerland) operated at 0.2 bar absolute, resulting in approximately 50 mL of syrup suitable for crystallizations. The Brix of the solution was measured periodically with an Abbe refractometer (Model 2t, Atago Co., Tokyo, Japan). Crystallizations were performed in a small cell crystallizer, using approximately 0.05 g of seed crystal. The solutions containing the seed crystals were mixed using a shaker bath for 4 h, and then left to equilibrate at room temperature for a further 20 h. The product crystals were washed with isopropanol and analyzed for color via the ICUMSA color method. The same techniques were used on the raw syrup to compare the efficiency of the

membrane separation technique with the current method in the processing plant.

Results and Discussion

Process Conditions in Raw Sugar Manufacturing Process.

Preliminary experiments analyzed the conditions of the process streams in the factory for concentrations and other conditions: these results are necessary to determine suitability for membrane treatment, and also to have a

comparison for streams produced as a result of membrane treatment. Results are shown in Table 1. Note that the composition and purity values are those calculated from standard industry tests (using Brix, pol, density, absorption, and other measurements), and are therefore not necessarily exact. There is considerable change in the pH of the streams as they pass through the process. The mixed juice is acidic, presumably due to the presence of organic acids such as lactic acid, acetic acid, and formic acid. The clarified juice is significantly more neutral than the mixed juice, presumably due to pH adjustment by lime (and,

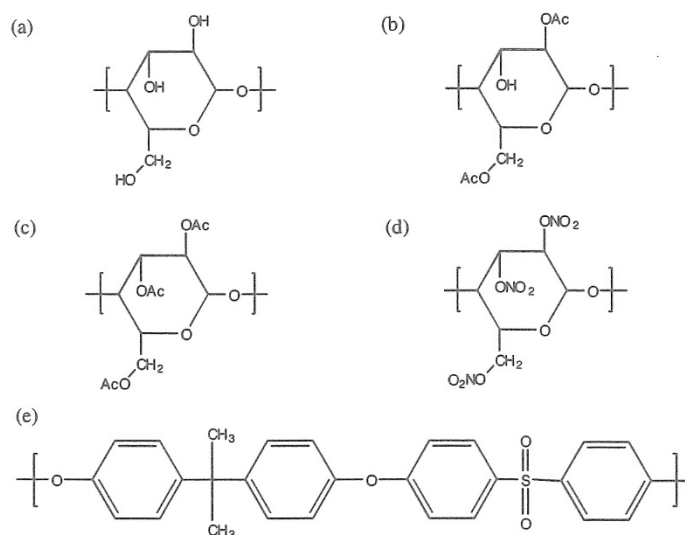


Figure 2. Chemical structures of the membrane materials. (a) Regenerated cellulose, (b) Cellulose acetate (about 70% of OH groups are acetylated), (c) Cellulose triacetate (more than 92% of OH groups are acetylated), (d) Cellulose nitrate, and (e) Polysulfone. (Some hydrogen atoms have been omitted for clarity)

Table 1. Selected processing conditions in the sugar process during the study. Color is by the ICUMSA color measurement technique

	Mixed juice	Clarified juice	Raw syrup
pH	5.26	7.22	6.44
Brix (%)	21.00	17.00	61.00
Pol (%)	16.82	16.82	59.80
Purity (%)	80.10	98.94	98.03
RS (%)	0.50	0.50	2.85
RSR	2.97	2.97	4.77
Raw Crystal	Pol = 98.5 %	Moisture = 0.2 %	Color = 2,434

if necessary, also acid to return from a basic solution to a neutral one). The raw juice is again slightly acidic, possibly due to further decomposition of carbohydrates at the elevated temperatures occurring in the evaporators. The clarification operation increases the purity to more than 98%, and this remains almost constant throughout the evaporation operation, with a small loss possibly due to decomposition of sucrose to the reducing sugars fructose and glucose. The main change during the evaporation process is to increase the Brix to greater than 60%. The raw sugar product crystals had a pol of 98.5% (an approximate purity), moisture of 0.2%, and an ICUMSA color of 2,434 color units, which are all typical values for raw sugars.

The aim of the study is to determine the feasibility of using membrane separation in place of the traditional clarification process, and to achieve this, a series of experiments were performed on the mixed juice, which is the stream which is sent to the clarifying process, in order to determine whether an equivalent or improved separation is achievable. Membrane separation experiments were performed on samples of the mixed juice using a range of different membranes. The results are divided into two sections: the first section describes results of experiments investigating the permeate flux and separation achievable by the membrane process at the start-up of the membrane process. The second section investigates the reduction in permeate flux due to membrane fouling, which

could be used to determine suitable cross-membrane flows in a cross-flow unit, or operating periods possible between membrane washing cycles.

Membrane Processing Conditions at Start-Up

These experiments determined separation performance and permeate flux for a clean membrane (prior to fouling) by measuring initial performance upon start-up of the membrane process, which should result in the maximum flux. The membranes chosen for the study were regenerated cellulose membranes with 1,000, 10,000, and 30,000 MWCO (Table 2), cellulose triacetate membranes with 5,000, 10,000, and 20,000 MWCO (Table 3), polysulfone membranes with 10,000, 30,000, 100,000, and 300,000 MWCO (Table 4), and 0.2 and 0.8 μm pore size cellulose acetate and 0.2 μm pore size cellulose nitrate microfiltration membranes (Table 5). Experiments were performed at 30°C.

Performance Characteristics of Regenerated Cellulose Membranes (Table 2)

It can be noted that the regenerated cellulose membranes have a permeate Brix value very similar to the feed solution, except for the membrane with a 1,000 MWCO, where the Brix value is only about half of that of the feed. The Brix is strongly related to the concentration of sucrose in the solution, and this result suggests that the sucrose molecule easily passes through

Table 2. Performance of the 300 mL membrane separation unit for ultrafiltration, using regenerated cellulose membranes. The feed to the membrane unit was mixed juice with the properties given in the second column of the table

	Mixed juice	Molecular weight Cut-Off		
		30,000	10,000	1,000
pH	5.26	5.31	5.24	5.35
Brix (%)	21.00	19.00	21.00	11.00
Pol (%)	16.82	19.68	19.30	9.00
Purity (%)	80.10	50.95	91.90	81.82
RS (%)	0.50	0.67	1.08	5.60
RSR	2.97	6.92	5.60	5.89
Flux ($\text{mL}/\text{m}^2\text{s}$)	-	4.52	4.30	0.43

the regenerated cellulose membrane if the pore size is large enough. The partial retention of sucrose at 1,000 MWCO is not surprising: sucrose is a molecule with a molecular weight of 342, but the shape of the molecule (which contains two monosaccharide sugars joined by a glycosidic bond) is unusual: the shape of the sucrose molecule significantly deviates from a sphere, and the molecule is also very flexible (due to the glycosidic bond), and this causes the molecule to be inhibited in its diffusion even in pores that might allow higher molecular weight molecules to pass more easily. The flux for the regenerated cellulose membranes are good for the higher MWCO (4.5 and 4.3 mL/m²s) but poor for the 1,000 MWCO membrane, suggesting pore blockage by sucrose molecules.

In the regenerated cellulose membrane the purity of the 1,000 MWCO is poor (only slightly larger than that of the feed), largely due to an increase in the fraction of reducing sugars. This is almost certainly because the reducing sugars (mainly glucose and fructose) are significantly smaller than sucrose, having molecular weights of only 180: thus, these sugars may pass through the membrane while sucrose is partially retained, increasing the fraction of reducing sugars on the permeate side of the membrane. The 10,000 MWCO regenerated cellulose membrane is able to purify the mixture, increasing the feed purity of 80.1% up to 91.9% in the permeate.

Performance Characteristics of Cellulose Triacetate Membranes (Table 3)

Cellulose triacetate ultrafiltration membranes were less successful in treating the mixed juice than the regenerated cellulose membranes. The higher molecular weight membranes (10,000 and 20,000 MWCO) were able to purify the juice, increasing the purity to 85.0% and 89.8% respectively, but only at the cost of a reduced Brix in the permeate, indicating that significant amounts of sucrose were retained in the retentate. In addition, the fraction of reducing sugars in the permeate increases as the MWCO decreases, indicating that these sugars can pass through the low MWCO membranes more easily than sucrose, and this has a degrading effect on the permeate purity. The 20,000

MWCO cellulose triacetate membrane has a good flux (4.0 mL/m²s) but the flux from the lower MWCO membranes is not sufficient for effective operation. It is likely either that sucrose is strongly adsorbed to the surface of the pores in the cellulose triacetate membrane thus causing a reduction in flux as pore blockage takes place in low MWCO membranes, or that a component of the mixed juice causes the cellulose triacetate to swell, also leading to pore blockage.

Performance Characteristics of Polysulfone Membranes (Table 4)

Polysulfone membranes did not give large increases in purity for any MWCO, and this may be due to allowing color impurities to pass (for large MWCO membranes) or due to increases in the reducing sugar fraction (relative to the amount of sucrose) in the 10,000 MWCO process. The fluxes achieved in the larger (30,000 - 300,000) MWCO membranes were good (3.9 - 4.6 mL/m²s) but the flux through the 10,000 MWCO membrane was very low at 0.55 mL/m²s, which again suggests pore blocking by sucrose or larger molecular weight carbohydrates present in the mixture.

Performance Characteristics of Microfiltration Membranes (Table 5)

Although all three microfiltration membranes had good permeate fluxes, these membranes did not purify the mixed juice at all, and are therefore unsuitable for the process. Microfiltration is typically suited to molecules in excess of 500,000 Da (and also micro-particles), and it is unlikely that the color compounds formed in the sugar processing are of such large molecular weights.

Discussion of Permeate Flux vs. Molecular Weight Cut-Off for all Membranes

The data representing the permeate flux of particular membrane types as a function of the MWCO is shown in Figure 3. It is clear that the plots for the different membrane types follow a similar trend, with the flux initially increasing very rapidly with increasing MWCO, and then the increase in flux becoming much less significant after a 'critical MWCO' value. If

Table 3. Performance of the 50 mL membrane separation unit for ultrafiltration, using cellulose triacetate membranes. The feed is mixed juice of the same properties as in Table 2

	Molecular weight Cut-Off		
	20,000	10,000	5,000
pH	5.32	5.31	4.95
Brix (%)	11.00	12.00	11.50
Pol (%)	9.88	10.20	7.56
Purity (%)	89.82	85.00	65.74
RS (%)	0.58	0.93	1.31
RSR	5.87	9.12	17.33
Flux (mL/m ² s)	4.00	1.30	0.30

Table 4. Performance of the 50 mL membrane separation unit for ultrafiltration using polysulfone membranes. The feed is mixed juice of the same properties as in Table 2

	Molecular weight Cut-Off			
	300,000	100,000	30,000	10,000
pH	5.35	5.40	5.31	4.76
Brix (%)	17.00	10.50	10.00	15.50
Pol (%)	11.65	8.77	8.03	12.26
Pur. (%)	68.53	83.52	80.30	79.10
RS (%)	0.75	0.49	0.44	1.24
RSR	6.44	5.59	5.48	10.11
Flux (mL/m ² s)	4.58	4.41	3.90	0.55

Table 5. Performance of the 50 mL membrane separation unit for microfiltration using cellulose nitrate (CN) and cellulose acetate (CA) membranes

	Mixed juice	Pore size (μm) and material		
		0.2 CN	0.2 CA	0.8 CA
pH	5.26	5.37	5.28	5.30
Brix (%)	21.00	11.00	16.00	15.50
Pol (%)	16.82	8.50	9.76	8.37
Purity (%)	80.10	77.27	61.00	54.00
RS (%)	0.50	0.47	0.71	0.88
RSR	2.97	5.53	7.27	10.51
Flux (mL/m ² s)	-	4.41	3.80	4.25

the graph is normalized based on this critical MWCO value then the curves essentially superimpose, having consistent flux values. The flux at the corner MWCO are approximately 4.0 mL/m²s. The critical MWCO value is a strong function of the membrane material, and it is likely that this is due to the degree of chemical interaction between sucrose and the membrane material.

The optimum MWCO for ultrafiltration operation is almost certainly at the critical MWCO. This will result in better purification than if a larger MWCO membrane was used with little loss of potential permeate flux. If a membrane with a MWCO lower than the critical MWCO cut-off is used then this results in a large reduction in the flux of the operation, which is likely to make it less cost effective due to the larger membrane areas that would be required for a particular treatment rate.

The optimum membrane process was determined to be a regenerated cellulose membrane with a 10,000 MWCO due to this membrane having high values of permeate flux and purity, and no significant reduction in dry solids (total solute) content due to the separation.

Results of Studies on Crystallization of Process Streams and Permeate

Crystallizations were performed on the permeate from this membrane (concentrated by vacuum evaporation up to 70% Brix), and also the raw syrup from the process factory at 70% Brix (which is the stream that is being crystallized in the factory), and the clarified juice from the factory process evaporated up to 70% Brix. The yield of the crystallization was approximately 30% in each case (higher yields could be achieved by evaporative crystallization). After the crystallization, the crystals were dissolved to produce an approximately 8% Brix solution (of density close to 1 g/mL) that is suitable for color determination by the ICUMSA technique. The results are presented in Table 6. The results show that the membrane purified juice produced a crystal that was significantly cleaner than that produced from either of the process streams. Even the crystals prepared from the process stream had a relatively good color value (color for raw sugars is typically in the range of 5,000 units), and this may be due to our crystallizations being performed for lower yields than achieved in the industrial process, leading

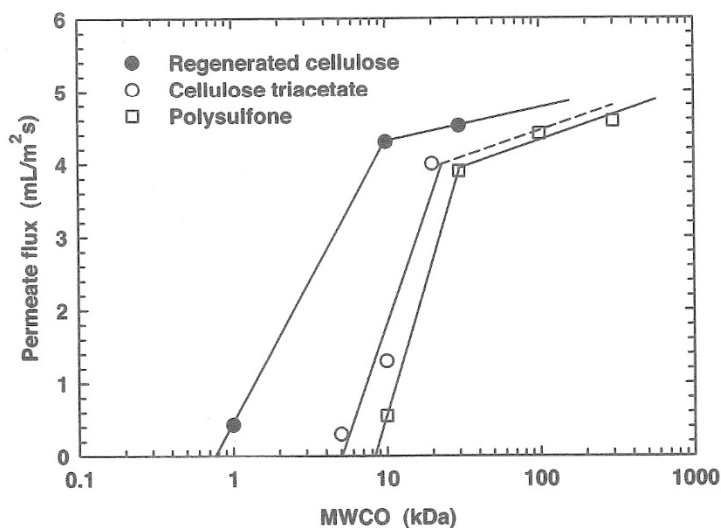


Figure 3. Initial permeate flux of mixed juice (prior to lime and heat treatment) through ultrafiltration membranes of various types

Table 6. Color of the raw sugar crystal product from the process raw syrup, evaporated clarified juice, and evaporated UF treated juice. ICUMSA is measured in solutions of dissolved crystals of low Brix. The apparent specific density is that of the solution with which the color is measured

	Brix (%)	Apparent specific density	Color (ICUMSA)
Raw syrup	8.0	1.0289	2,434
Clarif. juice	8.0	1.0289	2,531
UF Permeate	8.5	1.0309	1,244

Table 7. Properties of the feed (mixed juice) and the permeate of the regenerated cellulose UF membranes as a function of the molecular weight cut-off

Property	Mixed juice	Molecular Weight Cut-Off (MWCO)			
		5,000	10,000	30,000	100,000
pH	5.22	5.21	4.92	4.65	4.67
Brix (%)	13.80	13.00	12.00	13.50	13.40
Pol. (%)	7.53	11.34	10.25	9.86	10.22
Apparent Purity (%)	54.57	87.23	85.42	73.04	76.27
Max. Flux (mL/m ² s)	-	2.13	2.20	2.46	3.30
Steady-state Flux (mL/m ² s)	-	1.21	1.36	1.40	1.58

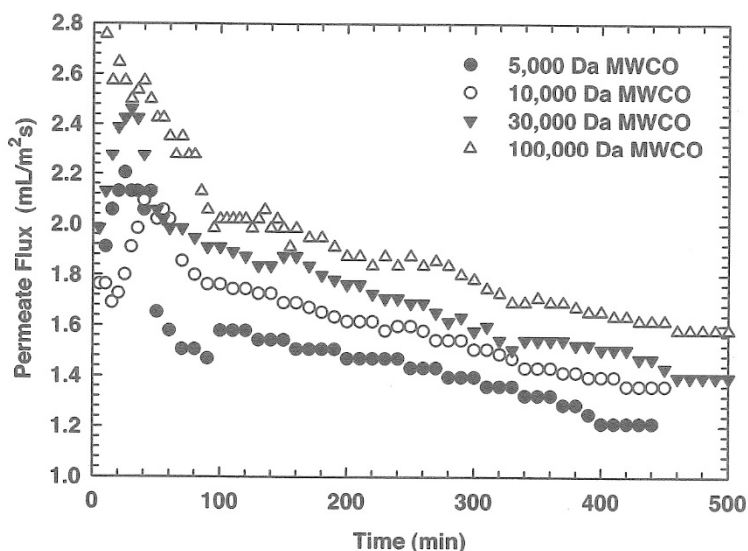


Figure 4. Changes in the permeate flux of mixed juice (prior to lime and heat treatment) through regenerated cellulose membranes as a result of membrane fouling

to reduced viscosities in the crystallizer and therefore less adsorption of mother liquor on the surface of the crystal.

Non-Steady-State Flux Conditions

To study the unsteady-state permeate flux of the regenerated cellulose membranes (that were determined to be optimal in the study above) the permeate flux was measured over a period of more than 8 h using these membranes in the stirred cell ultrafiltration unit with an operating pressure of 2.5 bar. The unsteady-state permeate flux values are shown in Figure 4. The conditions of the mixed juice feed and the permeate are shown in Table 7.

There are several mechanisms which are potentially responsible for unsteady-state permeate flux values in dead-end membrane separations, including membrane fouling, concentration polarization, or increasing (time dependent) solute concentrations on the retentate side of the membrane causing higher solution viscosity, and therefore lower membrane fluxes. Significant flux reduction due to concentration polarization is unlikely in the current experiments due to the strong mixing effect of the

stirrer near the surface of the membrane. The results of the study above show that the regenerated cellulose membranes do not retain sucrose to any appreciable extent (the Brix value of the permeate is essentially identical to that of the retentate), and this means that the membrane only retains the large molecular weight color compounds, while the sucrose content of the retentate remains essentially constant. Since the high molecular weight compounds appear in the syrup at very low concentrations there is no significant increase in total solute concentration in the retentate, and therefore also no permeate flux deterioration due to increased solute concentration. Therefore the decrease in the permeate flux with increasing time is due to fouling of the membrane caused by solute (color compound) adsorption onto the membrane surface and membrane pore plugging.

The values of the steady-state permeate flux and the apparent purity of the permeate are plotted together in Figure 5. This plot is useful for determination of the optimum MWCO for the membrane separation, because the operation requires a reasonably high value of both the steady-state permeate flux and the apparent

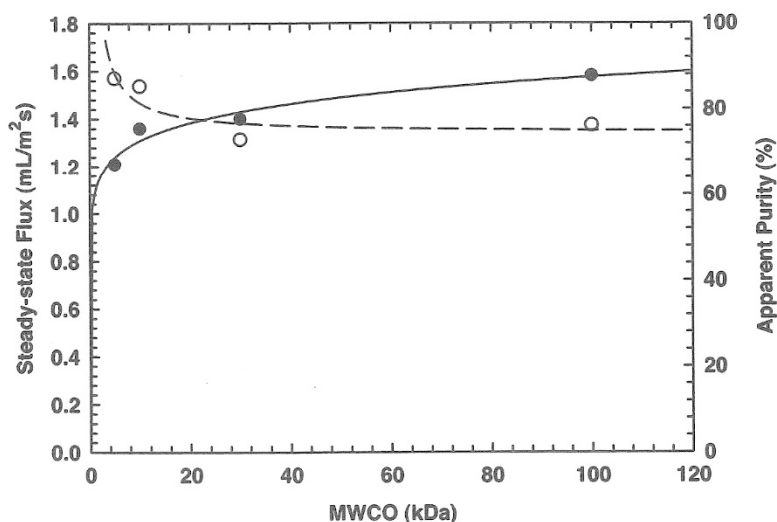


Figure 5. Steady-state membrane flux (●) and apparent purity of the permeate (○) for ultrafiltration of mixed juice (prior to lime and heat treatment) using regenerated cellulose membranes. Lines represent fits of the data taking into account boundary conditions at low MWCO

purity; however the flux curve increases with increasing MWCO while the purity curve decreases with increasing MWCO, resulting in a trade-off between permeate flux and purity. The optimum MWCO appears to be at 10,000 Da, which results in a steady-state permeate flux of 2.20 mL/m²s and an apparent permeate purity of 85.4%.

Conclusions

The findings of this study indicate that regenerated cellulose membranes with a MWCO of 10,000 Da show good performance in the separation of color compounds from the mixed juice from the raw sugar manufacturing process. This membrane has a high permeate flux and permeate purity. Crystals produced from the permeate of the UF membrane separation have significantly lower color values than crystals produced from the raw syrup from the factory, which has been purified from the mixed juice by the traditional lime treatment process. This indicates a higher purity and a high value product, because the selling price of raw sugar is dependent on the color value of the crystals.

The permeate flux of the UF membranes decreases over time due to surface fouling and pore blocking, which has been observed to take place over a time period of greater than 8 h in a classical dead-end unit. In an industrial process the fouling could be minimized by operating at higher temperatures, and/or by increasing liquid flow velocities over the surface of the membrane in cross flow operation.

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