

This article was downloaded by:

On: 25 January 2011

Access details: *Access Details: Free Access*

Publisher *Taylor & Francis*

Informa Ltd Registered in England and Wales Registered Number: 1072954 Registered office: Mortimer House, 37-41 Mortimer Street, London W1T 3JH, UK



## Separation Science and Technology

Publication details, including instructions for authors and subscription information:

<http://www.informaworld.com/smpp/title~content=t713708471>

### SIGNIFICANCE OF MEMBRANE TYPE AND FEED STREAM IN THE ULTRAFILTRATION OF SUGARCANE JUICE

M. Balakrishnan<sup>a</sup>; M. Dua<sup>a</sup>; P. N. Khairnar<sup>b</sup>

<sup>a</sup> Tata Energy Research Institute, New Delhi, India <sup>b</sup> Sugarcane Technology Mission, New Delhi, India

Online publication date: 31 March 2001

**To cite this Article** Balakrishnan, M. , Dua, M. and Khairnar, P. N.(2001) 'SIGNIFICANCE OF MEMBRANE TYPE AND FEED STREAM IN THE ULTRAFILTRATION OF SUGARCANE JUICE', *Separation Science and Technology*, 36: 4, 619 — 637

**To link to this Article:** DOI: 10.1081/SS-100102950

URL: <http://dx.doi.org/10.1081/SS-100102950>

### PLEASE SCROLL DOWN FOR ARTICLE

Full terms and conditions of use: <http://www.informaworld.com/terms-and-conditions-of-access.pdf>

This article may be used for research, teaching and private study purposes. Any substantial or systematic reproduction, re-distribution, re-selling, loan or sub-licensing, systematic supply or distribution in any form to anyone is expressly forbidden.

The publisher does not give any warranty express or implied or make any representation that the contents will be complete or accurate or up to date. The accuracy of any instructions, formulae and drug doses should be independently verified with primary sources. The publisher shall not be liable for any loss, actions, claims, proceedings, demand or costs or damages whatsoever or howsoever caused arising directly or indirectly in connection with or arising out of the use of this material.

## SIGNIFICANCE OF MEMBRANE TYPE AND FEED STREAM IN THE ULTRAFILTRATION OF SUGARCANE JUICE

**M. Balakrishnan,<sup>1,\*</sup> M. Dua,<sup>1</sup> and P. N. Khairnar<sup>2</sup>**

<sup>1</sup>Tata Energy Research Institute, New Delhi, India

<sup>2</sup>Sugar Technology Mission, New Delhi, India

### ABSTRACT

This work examines the purification of sugarcane juice in a cross-flow ultrafiltration (UF) system. Experiments were conducted on-site at a sugar mill using fresh feed drawn from the appropriate stage in the sugar manufacturing process. Different polymeric membranes with a nominal molecular weight cutoff rating in the 10–50 kD range were evaluated. The 20 kD polyethersulphone membrane was identified to be the most effective in terms of acceptable flux coupled with significant removal of nonsugar impurities. Subsequent trials were performed on four feed streams viz. mixed juice, raw juice, rotary vacuum filtrate, and clarified juice. It was observed that with the exception of the clarified juice, the fine suspended particles (bagacillo) in all the other streams formed a secondary filtration layer on the membrane surface during the course of filtration. The permeate displayed a 1.5–3 unit rise in juice purity, which is a remarkable improvement over the 0.5–1 unit rise obtained in the liming-sulphitation process. In ad-

---

\*Corresponding author. E-mail: malinib@teri.res.in

dition, the UF permeate was typically over three times as clear with a fivefold color reduction when compared to the clarified juice produced by the conventional process.

*Key Words:* Membrane; Sugarcane juice; Ultrafiltration; Flux; Purity rise.

## INTRODUCTION

Ultrafiltration (UF) is a promising alternative to the liming-sulphitation process for the purification of sugarcane juice in the manufacture of plantation white sugar (1). Sugarcane juice is a multicomponent feed, which apart from 10–21% sucrose, contains up to 2.5% of nonsugar impurities such as dextrans, proteins, fats, gums, and waxes (2). The conventional clarification scheme is incapable of completely eliminating these macromolecular impurities. This imparts cloudiness to the dark yellowish brown clarified juice that results in sugar crystals with undesirably high color. Treatment by UF, on the contrary, produces a superior juice with a better clarity, much lower viscosity, and noticeable color removal (3,4). Table 1 summarizes the different types of membranes and modules that have been investigated for this application.

In an earlier paper (1), we presented a broad overview of the application of UF for juice quality improvement in the cane sugar manufacturing process. The field tests were further continued to investigate the effect of operating parameters on the UF of fresh mixed juice obtained from the milling station (13). The on-site trials were motivated by the need to use a feed sample that is representative of the actual processing conditions in a sugar mill. Further, this would also reflect the variations in juice characteristics owing to differences in cane variety, soil and growing conditions, weather patterns, and season as well as any fluctuations in the manufacturing process itself.

This work presents a systematic study of the UF characteristics of sugarcane juice streams encountered in the production of plantation white (mill white) sugar. The manufacturing process generates four different juice streams (Fig. 1) viz. mixed juice, raw juice, rotary vacuum filtrate (RVF), and clarified juice. The UF of each of these streams is investigated. Further, the suitability of different polymeric membrane materials for this application is also examined. This study is expected to aid in identifying the optimum combination of membrane(s) and process stream(s) that would be appropriate for the next stage of pilot trials (14).

## MATERIALS AND METHOD

### Ultrafiltration Runs

The experiments were performed on-site at the Simbhaoli Sugar Mills Limited, which is located about 100 km from Delhi at Simbhaoli village, Ghazi-



**Table 1.** Performance of Various Membrane Types for Sugarcane Juice UF

Membrane Details	Module	Flux (LMH)	Remarks	Reference
50 kD, polyvinylidene flouride (Koch, USA)	Spiral wound	40–60	Field trials with clarified juice at 95°C	(5)
0.02 $\mu$ m, $\text{ZrO}_2$ coated ceramic "Kerasep" (Tech Sep, France)	Tubular	210–330	Field trials with clarified juice at 98°C	(6,7)
0.1–0.2 $\mu$ m SELECTFLO™ (Dow, USA)	Hollow fiber	NA	Field trials	(8)
6–25 kD GR60P, GR61P, and GR81P (DDS, Denmark)	Plate and frame	50–130	Field trials with clarified juice at 80°C	(9)
20 kD polysulfone (Ion Exchange, India)	Hollow fiber	0.043–0.168	Laboratory studies on limed juice	(3)
50kD-0.45 $\mu$ m $\text{TiO}_2/\alpha\text{-Al}_2\text{O}_3$ or $\text{ZrO}_2$ coated ceramic Carbosep™ 40 (TechSep, France)	Tubular	40–450	Laboratory trials at 90°C with limed mixed juice	(10)
Ceramic (TDK Corporation, Japan)	Tubular	24–105	Trials at 60°C with raw, limed, and vacuum-filtered juice with both hand-milled and factory samples	(4)
10 kD PM series (Amicon, USA)	Stirred	18–60	Laboratory trials at 60°C with raw and limed juice	(11)
5kD G-05T (Bio-Engineering)	Stirred	24	Laboratory trials at 60°C with raw and limed juice	(11)
200 kD, UK 200 (Toyo Roshi, Japan)	Stirred	39.6	Laboratory trials at 60°C with raw and limed juice	(11)
300 kD XM series (Amicon, USA)	Stirred	97.2	Laboratory trials at 60°C with raw and limed juice	(11)
5kD & 30kD YM series (Amicon, USA)	Stirred	>60 LMH	Laboratory trials at 85°C with raw and limed juice	(12)



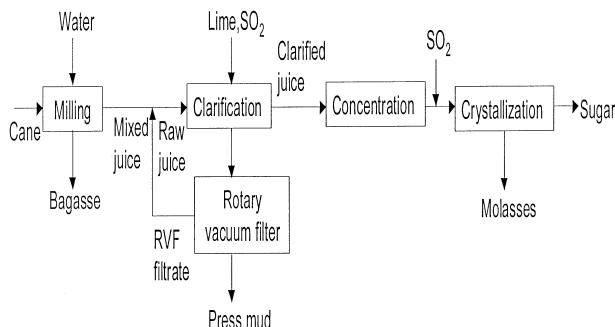


Figure 1. Schematic of plantation white sugar manufacturing process.

abad district (Uttar Pradesh). The UF was conducted in a cross-flow module (Rayflow, TechSep, Miribel, France) with an effective membrane area of  $400 \text{ cm}^2$  employing commercially available flat sheet membranes (Table 2). The membranes were used without further modification, unless specifically stated otherwise. Select runs on PPE0106 membranes were conducted after hydrophilizing the membrane surface by the adsorption of polyvinyl alcohol (PVA). The procedure involved circulating a 0.1% PVA solution over the membrane surface for approximately 5 min. The system was then flushed with water to remove the excess, unadsorbed PVA before conducting the UF.

Fresh juice was collected from the appropriate stage in the manufacturing process and was pretreated, as required, before UF. The mixed juice and the RVF filtrate were hot limed at 60–70°C to neutral pH. The raw juice pretreatment procedure involved liming, flocculation and settling (15). The clarified juice was tested without any further treatment. All the feed streams were prefiltered through a series of stainless steel (SS) sieves (60 mesh followed by 120 mesh) screens to remove suspended particles prior to UF.

Table 2. Membrane Characteristics

Membrane	Manufacturer	NMWCO (kD)	Membrane Material	PWP <sup>a</sup> (LMH per kg/cm <sup>2</sup> )
IRIS UF3028	TechSep, France	10	Polyethersulphone	72.1
PPE0106	Permeionics, India	20	Polyethersulphone	119.5
IRIS UF3028	TechSep, France	30	Polyethersulphone	281.9
PPU0105	Permeionics, India	50	Polysulphone	326.9
IRIS UF3042	TechSep, France	50	Acrylic	395.5
IRIS UF3050	TechSep, France	50	Acrylic	572.1

<sup>a</sup> Experimental values, evaluated at 40°C with demineralized water.



A known volume, i.e., 5–7 L, of the treated feed was then filled in a jacketed SS vessel connected to a constant temperature circulator (Julabo Labortechnik, Seelbach, Germany). The feed temperature was maintained at 45°C. The feed was pumped to the UF unit through a variable speed peristaltic pump (Cole Parmer, Vernon Hills, IL, U.S.A.) with a maximum flow rate of about 7.5 L/min. As the filtration module was constructed of transparent acrylic, it enabled the visualization of the membrane surface during the course of the experiment. The UF was performed in a batch mode with complete retentate recycle. Once 600–1000 mL permeate was withdrawn, the old feed was discarded and the module thoroughly flushed with water. The UF was resumed thereafter with fresh feed, obtained after the necessary pretreatment steps.

At the end of each experiment, the membranes were thoroughly flushed with demineralized water that was prefiltered through 120-mesh SS filters. The membrane fouling, expressed as a percentage drop in the water permeability, was estimated as follows:

$$\text{Fouling (\%)} = \{(PWP_{clean} - PWP_{fouled})/PWP_{clean}\} \times 100$$

where  $PWP_{clean}$  and  $PWP_{fouled}$  represent the pure water permeability before and after the cane juice UF respectively. The membranes were then cleaned in place using an appropriate cleaning solution before storing in 1% formalin till the subsequent run.

### Juice Analysis

The juice analysis was performed as per the mill's standard practice following the norms prescribed by the Sugar Technologists Association of India (16). All the chemicals used in these studies were analytical grade.

#### Brix

The brix is a measure of the total dissolved solids in the juice. The juice sample was filled to overflow in a cylinder and was allowed to stand for about 20 min to allow all air to escape. The standardized brix spindle (0–10 or 10–20 brix range, Reige, Germany) was then gradually lowered into the cylinder. The brix value was read out once the spindle became steady. The sample temperature was also noted and the corrected brix value obtained from standard tables.

#### Pol

The pol, which is a measure of the total polarizable substances in the juice, is taken to represent the juice sucrose content. 200–250 mL of the juice sample



was treated with 2–3 g dry subacetate of lead before filtering through dry filter paper. The clarified liquid was filled in a 200-mm pol tube, taking care to eliminate all air bubbles. The pol reading was taken in a polarimeter (Schmitz and Heinsch, Germany). From the observed pol and the uncorrected brix, the corresponding pol percent juice was read directly from Schmitz's table.

#### Purity

The juice purity is defined as follows:

$$\text{Purity (\%)} = (\text{Pol percent juice}/\text{Corrected brix}) \times 100$$

The purity rise across UF was calculated as:

$$\text{Purity rise (−)} = (\text{Purity})_{\text{permeate}} - (\text{Purity})_{\text{feed}}$$

#### Color

The juice color was estimated by measuring the absorbance at 580 nm using a spectrophotometer (Systronics, India).

#### Calcium Oxide (CaO) Content

150 mL of the juice sample was clarified by adding lead subacetate and some active carbon, if required. About 60 mL of this clear solution was treated successively with small quantities of powdered potassium ferrocyanide till no further precipitate formation was observed. The sample was thoroughly mixed and filtered and the filtrate checked for the absence of lead with potassium iodide. A few drops each of liquid ammonia and the indicator (Eriochrome Black T) solution were added to 10 mL of the lead free filtrate thus obtained. The sample was then titrated against M/56 ethylenediaminetetraacetic acid (EDTA) solution till the endpoint, characterized by a sharp change of color from red to blue, was observed. The CaO content of the sample was calculated as follows:

$$\text{CaO (mg per L)} = (V \times 100 \times 100)/B$$

where  $V$  = titer value of EDTA and

$B$  = brix of the juice sample.



### Calculations

The retention of sugar and nonsugar components was estimated as follows:

$$\begin{aligned}
 \text{Sugar rejection (\%)} &= \{1 - (Pol)_{\text{permeate}}/(Pol)_{\text{feed}}\} \times 100 \\
 \text{Nonsugars rejection (\%)} &= \{1 - \{(Brix - Pol)_{\text{permeate}}/(Brix - Pol)_{\text{feed}}\} \times 100 \\
 \text{Brix rejection (\%)} &= \{1 - (Brix)_{\text{permeate}}/(Brix)_{\text{feed}}\} \times 100
 \end{aligned}$$

## RESULTS AND DISCUSSION

The reported results are based on trials conducted over one complete cane crushing season spanning nearly 150 days between November 1997 and May 1998. The data was obtained over 40 sets of experiments, each of 10–12 hours duration. One complete set of experiments involved the following steps:

1. Water UF for estimating the PWP of the clean membrane
2. Juice UF
3. Water UF for estimating the PWP of the fouled membrane

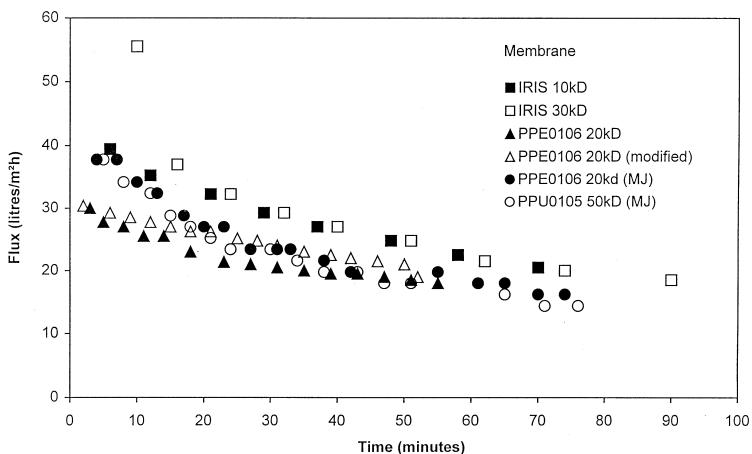
The juice flux variation was within  $\pm 10\%$  of the mean value. In contrast, the juice purity data displayed noticeable scatter, and the values were reproducible within  $\pm 25\%$ . The brix values were reproducible within  $\pm 20\%$ . This larger deviation could be attributed to the significant variation in the juice feed depending on such parameters as the cane variety, growing conditions, and weather conditions. Further, the juice samples are prone to microbial degradation with time, and thus any delay in sample analysis adversely affected the purity values.

### Membrane Type

Different polysulfone (PS) and polyethersulfone (PES) membranes in the 10–50 kD range were examined with the aim of improving the sugarcane juice clarity while reducing the color. The choice of the membrane material was based on their high temperature compatibility (typically up to 70°C) and wide pH range (1–13). Figure 2 displays the flux profiles for different membranes. The focus was on the performance of the indigenous PPU0105 and PPE0106 membranes, though other select membranes were also tested for comparison.

It was observed that the average permeate flux over 1 h of operation was in the 22–30 LMH range. The fluxes were marginally higher with both the 10 kD and the 30 kD TechSep membranes (28.9 LMH and 29.5 LMH, respectively) in com-





**Figure 2.** Comparison of different membranes for raw juice UF (MJ: mixed juice) (feed flow rate 6.8 liters/minute; pH 7.06–7.2, TMP 1.55–1.65 kg/cm<sup>2</sup>, temperature 45°C).

parison to the 20 kD and 50 kD Permionics membranes. The latter exhibited fluxes in the 22–25 LMH range. This could be possibly because the IRIS membranes were new whereas the PPU0105 and PPE0106 membranes had been used in over five earlier experiments. The juice purity rise with the TechSep membranes was 0.60 units with the IRIS 10 kD and 0.82 units with the IRIS 30 kD membranes. These values are comparable to the 0.5–1 unit rise that is obtained in the conventional liming-sulphitation process. The Permionics membranes, on the contrary, consistently displayed a significantly higher purity rise with typical values of 2.5 units and 1.2 units with the PPE0106 (modified) and PPE0106 (unmodified) membranes respectively. The UF permeate in all the experiments was clear and transparent. However, an examination of the permeate color indicated that the Permionics membranes produced a perceptibly lighter filtrate with absorbance values in the 0.08–0.15 range. In contrast, the permeate from the TechSep membranes was darker and comparable to the conventional clear juice (absorbance 0.5–0.75 units). Similar results were obtained with the IRIS 50 kD acrylic membranes that produced a clear but dark-colored permeate.

From these experimental results, it is observed that both the PPE0106 membranes exhibit a noticeably higher purity rise. It would be expected that a larger NMWCO rating of the membrane would result in lower rejection of the nonsugar impurities. This, in turn, would translate to a lower purity rise. Our experimental observations, however, do not follow this trend. Although the reason for this anomaly is not clear, it is possible that the fluctuations in the feed juice properties are partly responsible.



The final choice of the membrane would be determined by both the juice flux as well as clarity and color of the filtrate. Literature reports indicate that hydrophilic membranes perform perceptibly better than hydrophobic membranes with highly fouling streams (17). For instance, with raw cane juice, the tight regenerated cellulose based YM5 membrane gives better flux with little decline as compared to the polysulphone based PM10 and PM30 membranes even though the water flux with the PM series is higher (12). In the present investigations, both the PPU0105 and PPE0106 membranes displayed average fluxes in the 22–25 LMH range. These flux values at 45°C are a significant improvement over the extremely low values (below 0.2 LMH) reported with other trials on Indian juice employing polymeric membranes (3). More recently, Nene et al. (10) reported fluxes of 40–60 LMH in the UF of limed mixed juice through 50 kD ceramic Carboset™ membranes. However, these are initial fluxes observed in the first 15–20 minutes of UF. Further, the high operating temperature of 90°C would also be partly responsible for flux enhancement.

In the present studies, the permeate purity rise with the PPU0105 and PPE0106 membranes was in the 1.2–2.5 unit range. This compares favorably with the 1.5–3 unit purity rise reported by Kishihara et al. (4,11) in laboratory trials on both hand-milled juice as well as factory samples, under controlled conditions. With 5–30 kD membranes, they observed a 6- to 8-fold lower permeate color when compared to limed, clarified juice. Further, it is also reported that permeate color is only marginally reduced with higher molecular weight cutoff UF membranes (6,7,10,11).

In our investigation, both the PPU0105 and PPE0106 Permionics membranes demonstrated satisfactory flux in combination with significant purity rise and color removal. Thus, these membranes were employed exclusively for all further testing.

### Feed Stream

The conventional clarification process in plantation white sugar manufacture involves liming and sulphitation of the raw juice, which is thereafter boiled and allowed to settle in a clarifier. Four major juice streams can be identified in this scheme.

- a) *Mixed juice:* This stream was obtained from the milling station that was a tandem of four mills. The juice is usually a 70:30 mixture of the concentrated primary juice from the first mill and the thin secondary juice from the remaining mills. The mixed juice was a grayish green opaque solution with an average sucrose content of 10–13%. It was usually at 28–32°C and had a pH of 5.4–5.7. In addition to dissolved nonsugar impurities, the juice also contained 7–15 g/L suspended solids.



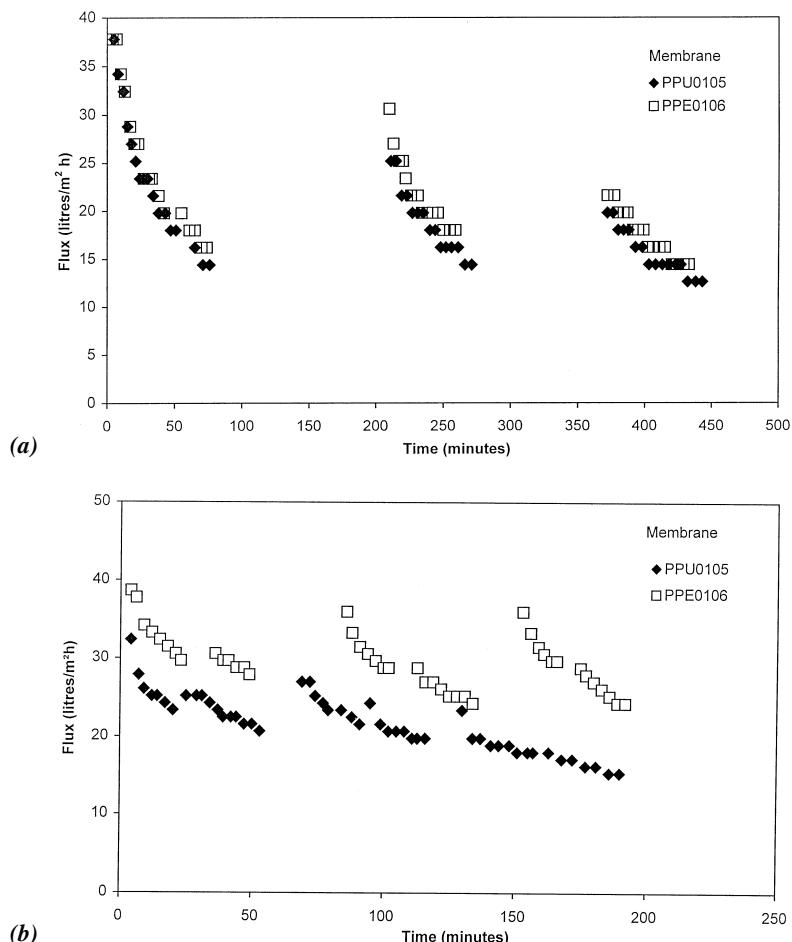
- b) *Raw juice*: The mixed juice from the milling station combines with the RVF (rotary vacuum filter) filtrate to form the raw juice. This stream is heated to 70°C in the raw juice heaters prior to the clarification step. The raw juice was turbid and dark grayish green with a pH between 5.7 and 5.9. It also had a high suspended solids content of 15–25 g/L.
- c) *RVF filtrate*: The muddy juice from the clarifier is filtered through rotary vacuum filters equipped with 0.5 mm screens to produce the RVF filtrate. This stream, which constitutes 12–15% of the total juice volume, was turbid and greenish brown in color and was normally at about 60°C. The sucrose concentration was 8–10% and the pH was between 6.3–6.8. This stream was also characterized by a significant suspended solids content due to the addition of bagacillo (approximately 30 g/L on a dry basis) in the clarification step.
- d) *Clarified juice*: The overflow from the clarifier constitutes the clarified juice stream, which is taken to the evaporators for concentration. The conventional clarified juice approximately at 98–102°C and an average pH of 6.95–7.05, was yellowish brown. Though the juice was normally free from visible suspended particles, it was usually slightly hazy due to the presence of colloidal matter.

Figure 3 displays the flux characteristics of the various juice streams. Figs. 3a, b, and c exhibit the profiles for mixed juice, RVF filtrate, and clarified juice with PPU0105 and PPE0106 membranes. Figure 3d describes the trend for raw juice with the unmodified and surface modified PPE0106 membrane.

It was observed that the prefiltration of the various juice streams was not effective in eliminating the fine bagacillo particles. Though the addition of a cationic flocculant such as Magnafloc LT2 (Allied Colloids) aided considerably in the formation of large flocs that could be removed during prefiltration, complete removal of the fine bagacillo particles could not be effected, even by passing the juice twice through 150-mesh sieves. Consequently, during the course of UF, a particulate layer emerged on the membrane surface. This layer formation was pronounced with all the juice streams except with the clarified juice. The deposit, which became progressively denser with increasing feed concentration and with increasing operation time, was typically slimy to touch. It could be easily wiped off with a wet filter paper but could not be dislodged by flushing with water. Thus, during UF, this accumulated particulate matter behaved as a secondary membrane layer and controlled the juice filtration characteristics.

Figures 3a, b, and c clearly indicate that the performance of the tighter PPE0106 membrane is superior to that of the PPU0105 membrane. The average flux was higher with the PPE0106 membrane in all the three cases even though the clean membrane permeability of the larger cutoff PPU0105 membrane was over two-fold higher at the beginning of the trials. This is in agreement with the general observation that a membrane with a lower molecular weight cutoff is less





**Figure 3.** Effect of feed stream on sugarcane juice flux. **a)** Mixed juice (feed flow rate 6.8 liters/min; pH 7.0, TMP 1.55–1.60 kg/cm<sup>2</sup>, temperature 45°C). **b)** RVF filtrate (feed flow rate 5.2 liters/min; pH 6.3–6.8, TMP 0.63–1.71 kg/cm<sup>2</sup>, temperature 45°C). **c)** Clarified juice (feed flow rate 6.8 liters/min; pH 7.02, TMP 1.55 kg/cm<sup>2</sup>, temperature 45°C). **d)** Raw juice (feed flow rate 6.8 liters/min; pH 7.06, TMP 1.58–1.60 kg/cm<sup>2</sup>, temperature 45°C).

(continued)



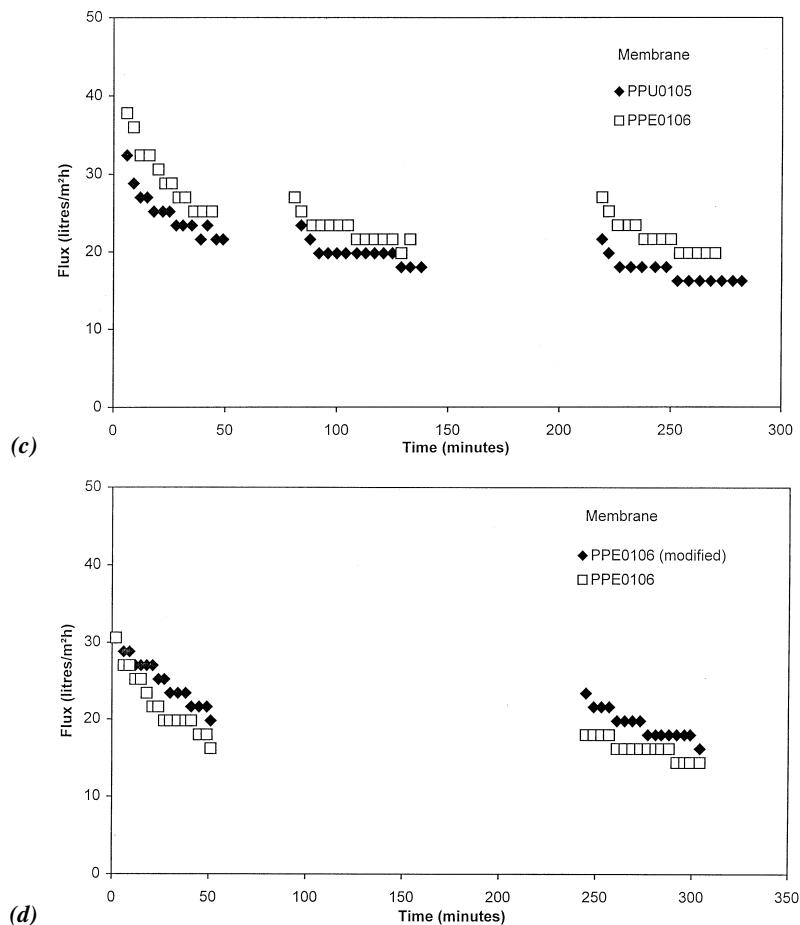


Figure 3. Continued.

prone to membrane fouling and thus exhibits higher average flow rates over long periods of operation (17).

The UF characteristics of cane juice for various process streams are summarized in Table 3. In all the experiments, the purity rise across UF was consistently better than the 0.5–1 unit improvement obtained with the conventional liming/sulphitation process. For the raw and mixed juice streams, an average purity rise of 1.5 units was observed whereas the average rise was over three units with the RVF filtrate. This can be correlated to the higher nonsugars rejection with the RVF stream (up to 30%) in comparison to that for mixed/raw juice (below 20%). The sugar rejection was generally well below 5% with all the feeds.



As there is an observable sugar rejection during the juice UF, it is pertinent to examine the effect of osmotic pressure during this process. Table 4 presents an estimate of osmotic pressure contribution due to the rejected sugar. The values obtained are significant considering the operating pressure was up to 1.7 kg/cm<sup>2</sup>. However, this result has to be interpreted with caution in view of the assumptions made in arriving at these figures. It is assumed that the rejected macromolecular impurities like proteins and polysaccharides contribute little to osmotic pressure (17). In the absence of a detailed analysis of the rejected components, it is assumed that the rejected sugar is the main contributing component. The sugar rejection, in turn, is estimated from the pol values. This again is an approximation because the pol is a measure of the total polarizing substances, assumed primarily to be sugar (sucrose).

The quality of the UF permeate was consistently superior when compared to that of the conventional clear juice (Table 5). The UF filtrate was sparkling clear in all the experiments and was lighter in color. The clarity was typically over threefold higher and the color was over five times lower than that of the conventional clear juice. This was in spite of the fact that sulphitation was avoided with the raw and mixed juice feed prior to UF. Thus, it should be possible to produce low color sugar crystals while eliminating juice sulphitation altogether. An additional benefit is the lower CaO content of the ultrafiltered juice. On an average, the UF permeate had a CaO content in the 950–1250 ppm range in contrast to 1300–1400 ppm with the clarified juice from the conventional process. This would lessen the evaporator fouling that, in turn, would imply reduced downtime for cleaning, in addition to savings on the cleaning chemicals.

As the permeate from the UF process would be directly taken to the evaporators for concentration, it is essential to maintain the permeate pH near neutral (6.95–7.05) as required in the manufacturing scheme. Because all the feed streams tested (except for the conventional clarified juice) were originally at acidic pH, the juices were appropriately limed prior to UF as described in the experimental method. Liming the permeate is not a preferred option, as it may adversely affect the clarity of the juice. However, a pH drop of up to 1.1 units was observed across the membrane during UF. There could be several reasons for this phenomenon. The experiments were performed in a batch mode and the feed was recirculated for 1–2 h during the course of the run. Because cane juice is prone to degradation with time, it is possible that the decrease in pH is partly due to microbial action. Also, mercuric chloride at 0.5–1 g/liter was added to the permeate samples to prevent inversion. This was as per the recommended practice in the mill for the preservation of juice samples for analysis. However, it was noticed that mercuric chloride itself reduces the juice pH at the rate of 0.45 units for every 0.5 g HgCl<sub>2</sub> added per liter of juice. Thus in some of the later experiments, an attempt was made to reduce the filtration time and fresh feed was employed for every 300 mL of permeate sample. Though it is probable that the pH drop was an artifact of the



Table 3. UF Characteristics of Various Cane Juice Streams

## I. Mixed Juice/Raw Juice

Membrane	Flux (LMH)		Feed				Permeate				Purity Rise (-)	Rejection (%)			
	0h	2h	Fouling (%)	B <sub>x</sub> (%)	Pol (%)	pH (-)	B <sub>x</sub> (%)	Pol (%)	pH (-)	B <sub>x</sub>	Nonsugars	Sugar			
PPU0105	29.1	8.8	93.7	13.24	10.26	77.49	7.28	12.62	9.94	78.76	7.40	1.27	4.68	10.07	3.12
PPU0105	28.2	16.2	89.7	14.56	11.62	79.81	7.30	13.90	11.34	81.58	6.34	1.77	4.53	12.93	2.41
PPU0105	33.3	9.0	90.4	14.21	11.32	79.66	7.23	13.60	10.95	80.51	6.69	0.85	4.29	8.30	3.27
PPU0105	34.2	14.4	80.9	13.13	10.51	80.05	7.38	12.52	10.19	81.39	6.29	1.34	4.65	11.07	3.04
PPU0105	38.7	12.6	84.9	13.71	10.88	79.36	8.51	13.23	10.71	80.95	7.90	1.59	3.50	10.95	1.56
PPU0105	38.1	18.5	79.2	14.12	11.54	81.73	7.33	13.49	11.09	82.21	7.44	0.48	4.46	6.98	3.90
PPU0105	43.5	23.5	37.3	14.56	11.65	80.01	7.08	13.84	11.23	81.14	7.08	1.13	4.95	10.31	3.61
PPU0105	40.5	17.0	74.2	14.44	11.93	82.62	7.05	13.95	11.77	84.37	7.01	1.75	3.39	13.15	1.34
PPU0105	23.4	9.0	80.3	14.11	11.55	81.86	—	13.41	11.16	83.22	—	1.36	4.96	12.11	3.38
PPE0106	37.5	19.0	69.4	14.12	11.54	81.73	7.33	13.41	11.17	83.30	7.49	1.57	5.03	13.18	3.21
PPE0106	36.8	23.5	46.7	14.56	11.65	80.01	7.08	14.06	11.42	81.22	7.03	1.21	3.43	9.28	1.97
PPE0106	42.0	17.6	64.9	14.44	11.93	82.62	7.05	13.89	11.78	84.81	6.99	2.19	3.81	15.94	1.26
PPE0106	18.0	7.2	77.2	14.11	11.55	81.86	—	13.17	11.01	83.60	—	1.74	6.66	15.63	4.68
PPE0106	30.6	16.2	60.4	13.61	11.15	81.93	7.25	12.73	10.75	84.45	7.09	2.52	6.47	19.51	3.59

(M)<sup>a</sup>  
Modified membrane, with PVA adsorption.

## ULTRAFILTRATION OF SUGARCANE JUICE

633

## II. RVF Filtrate

Membrane	Flux (LMH)		Feed				Permeate				Purity Rise	Rejection (%)			
	0h	2h	Fouling (%)	B <sub>x</sub> (%)	Pol (%)	Parity (%)	pH (-)	B <sub>x</sub> (%)	Pol (%)	Parity (%)	pH (-)				
PPU0105	33.3	17.1	67.4	10.67	8.12	76.10	6.44	10.19	7.95	78.02	5.83	1.92	4.50	12.16	2.09
PPU0105	32.4	18.9	69.4	10.34	7.81	75.53	6.54	9.90	7.59	76.67	5.88	1.14	4.26	8.70	2.82
PPU0105	51.3	24.3	69.0	8.60	6.24	72.56	7.00	7.71	6.09	78.99	6.96	6.43	10.35	31.36	2.40
PPU0105	46.8	23.4	69.0	9.51	6.97	73.29	7.08	8.39	6.62	78.90	6.99	5.61	11.78	30.31	5.02
PPE0106	38.7	24.3	68.2	9.30	6.77	72.80	6.33	8.82	6.56	74.38	6.31	1.58	5.16	10.67	3.10
PPE0106	38.7	14.4	53.2	8.02	5.87	73.19	6.50	7.25	5.44	75.03	6.40	1.84	9.60	15.81	7.33
PPE0106	28.8	18.0	69.3	9.90	7.33	74.04	7.08	9.26	7.05	76.13	7.06	2.09	6.46	14.01	3.82
PPE0106	35.1	15.3	72.9	10.03	7.32	72.98	6.76	9.03	6.97	77.19	6.63	4.21	9.97	23.99	4.78
PPE0106	43.2	18.9	69.1	8.91	6.59	73.96	7.00	8.24	6.47	78.52	6.91	4.56	7.52	23.71	1.82



**Table 4.** Osmotic Pressure Contribution During Juice UF

Parameter	Mixed Juice		RVF Filtrate	
	PPU0105	PPE0106	PPU0105	PPE0106
Average brix (%)	14.01	14.31	9.78	9.23
Average sugar rejection (%)	2.85	2.78	3.08	4.17
Osmotic pressure (kg/cm <sup>2</sup> )*	0.64	0.64	0.49	0.62

\* II (kPa) = 163.47C-5.882C<sup>2</sup> + 0.1324C<sup>3</sup> where C is the sucrose concentration in w/w% (18).

batch operation and would not be a concern in continuous processing, it is possible that this phenomenon occurs because of the preferential migration of the organic acids across the membrane. This would have to be confirmed by further experimentation.

It was observed that all the juice streams caused heavy membrane fouling. On an average, around 70% drop was observed in the PWP (pure water permeability) after juice UF. Various cleaning regimens were explored to restore the original membrane flux. Because there was a pronounced particulate deposition on the membrane surface with all the feed streams except the clarified juice, the cleaning process is expected to be more effective if the visible layer is wiped off first with a wet filter paper. However, in all the experiments, CIP (cleaning-in-place) was carried out initially without removing the particulate deposit.

As the protein impurity in the juice is known to cause heavy fouling, the membrane cleaning was first done with 0.1 N NaOH (60 min wash at ambient temperature). Further, in order to reduce the cleaning cycle time, different commercially available enzymatic detergents were also tested on the membrane manufacturer's recommendation. Depending on the feed stream and the operating conditions, the CIP involved circulating 0.1–0.3% detergent solution at 40°C for 30–90 min. The PWP recovery was generally in the 75–90% range. The same membrane sheets were used repeatedly, and it was observed that the fouling decreased progressively with each experiment. Figure 4 summarizes the extent of

**Table 5.** Comparison of Clear Juice Properties: UF versus Conventional Clarification

Parameter	Conventional Clear Juice	Ultrafiltration			
		Mixed Juice	Raw Juice	RVF Filtrate	Clarified Juice
Color (–)	0.602	0.107	0.122	0.050	NA
Clarity (%)	25.7	78.3	75.7	89.0	NA
CaO (ppm)	1350	970	995	1026	1220



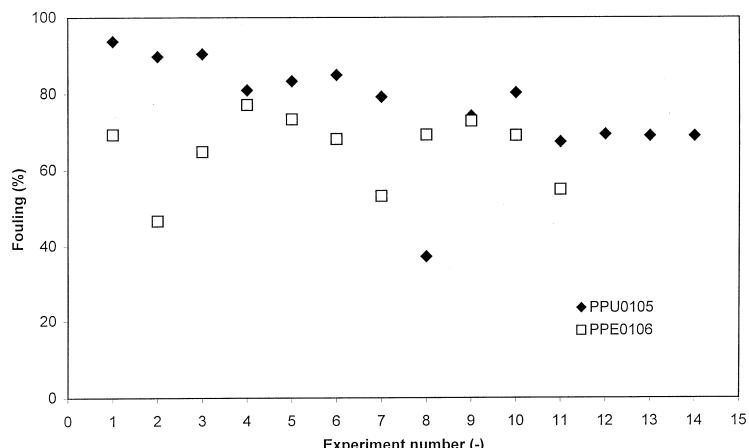


Figure 4. Comparison of membrane fouling.

membrane fouling over a series of experiments. It was observed that the PPU0105 membranes displayed a 76% loss in the PWP of the clean membrane after 14 runs. The loss was marginally less with the tighter PPE0106 sheets, which showed a 65% drop (in comparison to the original value for the new membrane) after 11 runs. This observation further confirms the long-term advantage of using tighter membranes.

From the low PWP recovery after juice UF, it is apparent that the membrane cleaning is not completely satisfactory. This could be due to either inappropriate choice of the cleaning chemicals, unsatisfactory water quality or a combination of both these factors. We suspect that the variation in the properties of the water available on-site is partly responsible for ineffective membrane cleaning. A systematic monitoring of water quality is in progress to validate this hypothesis.

## CONCLUSIONS

This work examines the effect of different membranes and feed streams on UF of sugarcane juice in the plantation white sugar manufacturing process. Of the different polymeric membranes evaluated, the 20 kD polyethersulphone membrane appeared to be best suited for this application in terms of acceptable flux combined with effective color removal. The UF process was very promising as it was accompanied by a 1.5–3 unit permeate purity rise that is a significant improvement over the 0.5–1 unit rise that is obtained in the traditional liming-sulphitation process. However, further investigations on membrane fouling



and cleaning are essential prior to recommending the process for large-scale application.

#### ACKNOWLEDGMENTS

This work was supported by a grant from the Sugar Technology Mission, Technology Information, Forecasting and Assessment Council, Department of Science and Technology, Government of India. We appreciate the support and co-operation provided by the Simbhaoli Sugar Mills Ltd., Simbhaoli, while performing the trials at their factory. We particularly thank Mr. S. N. Misra, Mr. N. C. Sharma, and Mr. Ranga Rao for useful discussions and suggestions. The membrane sheets provided by Mr. S. J. Mayor, Permionics are also gratefully acknowledged.

#### REFERENCES

1. Balakrishnan, M.; Dua, M.; Bhagat, J.J. *Int. Sugar J.* **2000**, *1213*, 21.
2. Mathur, R.B. *Handbook of Cane Sugar Technology*; Oxford and IBH Publishing Co.: New Delhi, 1986; 680.
3. Verma, S.K.; Srikanth, R.; Das, S.K.; Venkidachalam, G. *Indian J. Chemical Technol.* **1996**, *3*, 136.
4. Kishihara, S.; Tamaki, H.; Fujii, S.; Komoto, M. *J. Membr. Sci.* **1989**, *41*, 103.
5. Saska, M.; McArdle, J.; Eringis, A. *Int. Soc. Sugarcane Technologists, XXIII Congress*, New Delhi, India, February 1999.
6. Kwok, R.J. *55th Annual Sugar Industry Technologists Meeting*, Durban, SA, May 1996.
7. Cartier, S.; Theoleyre, M.; Lancrenon, X.; Decloux, M. *Proceedings of the Sugar Processing Research Institute Workshop on Separation Processes in the Sugar Industry*, New Orleans, Louisiana, October 1996.
8. Willet, C.C. *Int. Sugar J.* **1997**, *99* (1177E) 7.
9. Nielsen, W.F.; Kristensen, S. *Madsen, R.F. Sugar Technol. Rev.* **1982**, *9*, 59.
10. Nene, S.N.; Karode, S.K.; Courtois, T.; Mietton-Peuchot, M.; Gupta, B.B.; Ben-Aim, R.; *The 8<sup>th</sup> World Filtration Conference*, Brighton, U.K. May 2000.
11. Kishihara, S.; Fujii, S.; Komoto, M. *Int. Sugar J.* **1981**, *83*, 5.
12. Tako, M.; Sanehisa, N. *Agric. Biol. Chem.* **1986**, *50*, (4), 833.
13. Balakrishnan, M.; Dua, M.; Bhagat, J.J. *Sep. Pur. Technol.* **2000**, *19* (3), 209.
14. Ghosh, A.M.; Balakrishnan, M.; Dua, M.; Bhagat, J.J. *J. Membr. Sci.* in press.



**ULTRAFILTRATION OF SUGARCANE JUICE**

**637**

15. Dua, M.; Balakrishnan, M. Development of a Membrane Process for Clarifying Sugarcane Juice; Tata Energy Research Institute: New Delhi, India, TERI project report no. 96CE11, October 1998.
16. Varma, N.C. System of Technical Control for Cane Sugar Factories in India; The Sugar Technologists Association of India: New Delhi, India, 1988.
17. Cheryan, M. UF and MF Handbook; Technomic Publishing Co.: Lancaster PA, 1998.
18. Sourirajan, S. Reverse Osmosis; Academic Press: New York, 1970.

Received January 7, 2000

Revised June 2000



## **Request Permission or Order Reprints Instantly!**

Interested in copying and sharing this article? In most cases, U.S. Copyright Law requires that you get permission from the article's rightsholder before using copyrighted content.

All information and materials found in this article, including but not limited to text, trademarks, patents, logos, graphics and images (the "Materials"), are the copyrighted works and other forms of intellectual property of Marcel Dekker, Inc., or its licensors. All rights not expressly granted are reserved.

Get permission to lawfully reproduce and distribute the Materials or order reprints quickly and painlessly. Simply click on the "Request Permission/Reprints Here" link below and follow the instructions. Visit the [U.S. Copyright Office](#) for information on Fair Use limitations of U.S. copyright law. Please refer to The Association of American Publishers' (AAP) website for guidelines on [Fair Use in the Classroom](#).

The Materials are for your personal use only and cannot be reformatted, reposted, resold or distributed by electronic means or otherwise without permission from Marcel Dekker, Inc. Marcel Dekker, Inc. grants you the limited right to display the Materials only on your personal computer or personal wireless device, and to copy and download single copies of such Materials provided that any copyright, trademark or other notice appearing on such Materials is also retained by, displayed, copied or downloaded as part of the Materials and is not removed or obscured, and provided you do not edit, modify, alter or enhance the Materials. Please refer to our [Website User Agreement](#) for more details.

**Order now!**

Reprints of this article can also be ordered at  
<http://www.dekker.com/servlet/product/DOI/101081SS100102950>