Indian Journal of Chemical Technology
Vol. 3, May 1996, pp. 136-139

An efficient and novel approach for clarification of sugarcane juice by micro- and ultrafiltration methods†

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The efficacy of sugarcane juice clarification was studied with a polysulphone hollow fibre ultrafiltration membrane module having molecular weight cut-off of 20,000. The fouling index, *p*H optimization and permeate sterility were evaluated at 1.2 kg/cm^2 transmembrane pressure using the same module. The turbidity value of ultrafiltered sugarcane juice was found to be 0.5 NTU as compared to 200-250 NTU in the raw sugarcane juice. The maximum flux with least fouling was observed when the UF was carried out with a juice prelimed to bring it to about *p*H of 7.5 and the permeate was found to be free from microbes.

Raw sugarcane juice contains 12-15% sucrose and various impurities like reducing sugars, organic acids, amino acids, proteins, starches, gums, colouring matters and other suspended matters that impart dark colour and high turbidity to the juice. To get a clarified juice devoid of these impurities, liming, carbonation, sulphitation or phosphatation followed by sludge separation are being done in sugar industries. It is very much energy intensive, cumbersome, time-consuming and labour-intensive. This also introduces, due to the large dosages of chemicals, an increased amount of dissolved inorganic salts in the clarified juice. The salts impede crystallization of sugar due to the detrimental effects like scale formation in evaporator and enhance sucrose content in the molasses.

Ultrafiltration of cane juice results in the rejection of relatively large molecular weight substances while low molecular weight substances can pass through the membrane easily along with water, thus, giving a clear, light coloured and highly purified juice. Ultrafiltration, as it is operated at milder temperatures (30-60°C) and relatively moderate pressures (1-5 kg/cm^2), is not energy intensive. Further, quality of the sugar is expected to be superior and the juice will be free from microbial contamination. This technique has been a subject of investigation to many research workers[1-6]. Lower fluxes and fouling of membrane are major limitations of clarification of sugarcane juice by ultrafiltration. Kishihara *et al.*[4] reported that the problems of the flux reduction and fouling could be minimized by increasing the *p*H of the feed solution by liming it and by the addition of materials like bagasse. Based on their work, it was observed that membrane filtration can become a unit operation in the sugar industry, considering the advantages it offers by way of economy and the sugar quality.

Present investigation dealt with the study of effects of changing the *p*H of the sugarcane juice on the flux during clarification by ultrafiltration using a polysulphone-based hollow fibre membrane module and investigating the microbial contamination in the permeate as well as on fouling behaviour of the membrane attributed to various organic, inorganic and microbial contaminants.

Experimental Procedure

Sugarcane juice, calcium hydroxide (Analytical Grade), CUNO MICROWYND II micron filter (DCCPB & DCCPC), hollow fibre polysulphone ultrafiltration module, refractometer for measuring sucrose concentration and Hach Turbiditimeter (Model 2100 A) for measuring the clarity of various juices.

Liming — Raw sugarcane juice (5 L) was diluted to 12% sucrose and was limed using calcium hydroxide. Liming was done to increase the *p*H of the sugarcane juice from a base level of 5.5 and the studies were done at various *p*H like 7.5, 8.5 and 10 and also with unlimed juice. Liming was done at 70°C under agitation. This juice was stirred for an hour while being heated at 70°C. After that, the juice was cooled to settle down the suspended matters. The supernatant liquid was decanted and the decanted juice (referred to as limed juice) was used for further experiments. Turbidity and refractive index (R.I.),

†This paper was presented in the Indo-French International Meet and XIII National Conference of Indian Membrane Society on "Recent Advances in Membrane-Based Separation Science and Technology", Karnatak University, Dharwad, 27-28 February 1995.

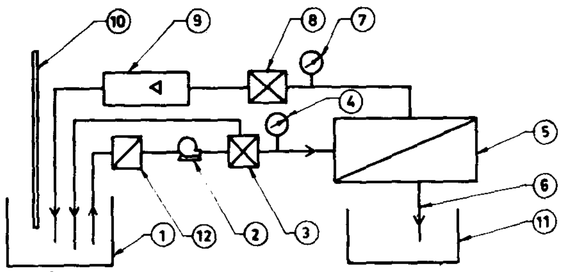

Fig. 1—Flow diagram of the sugarcane juice clarification by HF ultrafiltration system. [1 Feed tank, 2 pump, 3 by-pass valve, 4 pressure gauge (feed), 5 hollow fibre UF membrane module, 6 permeate line, 7 pressure gauge (reject), 8 reject valve, 9 flow-meter, 10 thermometer, 11 permeate tank, and 12 microfilter]

with a precalibrated refractometer, were measured for the juice before and after liming and also for the permeate of microfiltration and ultrafiltration.

Microfiltration—Microfiltration was carried out at 0.1 kg/cm^2 pressure using CUNO micron depth filters – DCCPB (pore size 10 μm), followed by DCCPC (pore size 5 μm); at room temperature. The turbidity and the R.I. of the permeate, which was used as the feed during clarification by ultrafiltration, were measured.

Clarification—The microfiltered juice was further clarified by ultrafiltration using a hollow fibre polysulphone cross flow module, having an effective area of 0.1873 m^2 and a molecular weight cut-off of 20,000, assembled by R&D Centre, IEI, Bombay and the experimental set-up is shown in Fig. 1. The transmembrane pressure (TMP) was maintained at 1.2 kg/cm^2. The feed flow rate was maintained at 550 mL/min at a constant temperature of 32°C with a volume of 4 L during all the trials.

Measurement of flux was done at various time intervals and the turbidity and R.I. of both the permeate and reject were noted at that time. After four hours of recirculation, the permeate was collected separately and was analyzed for sucrose content as well as calcium content (using flame photometry). The efficiency of the clarification was evaluated by comparing the turbidity in the composite permeate with that of the feed solution.

The intrinsic membrane characteristic, the pure water permeability (PWP), before and after the sugarcane juice run, was used to study the amount of fouling. The PWP of the membrane after every sugarcane juice trial was restored to its initial value by washing it with the cleaning solution developed by IEI, Bombay for half an hourandslushing it with demineralized water for two hours.

The raw juice, microfiltered juice and the UF permeate were analyzed for microbial contamination by Total Bacterial Count (TBC) method to find out **whether the permeate is sterile or not.**

Results and Discussion

India is one of the largest sugar producing countries in the world. Sugar is one of the most essential commodities which is supplied by more than 400 sugar industries in our country. More than 25% of the sugar originally present in the mixed juice is lost due to various treatment processes that occur in the multistage conventional process. This is one of the energy intensive industries. Advanced technologies evolving in the recent times will alleviate consumption of energy in these industries. Membrane separation is one such technology that will compete favourably for improving sugar quality and minimizing the cost involved in the consumption of energy, chemicals, etc. In this work, an attempt has been made to clarify sugarcane juice first by microfiltration followed by ultrafiltration.

Liming of raw sugarcane juice—Pre-treatment of raw sugarcane juice before microfiltration is unavoidable but the dosage of lime can be brought down to a large extent for this operation compared to the conventional coagulation procedures. The other processes like suphitation and phosphatation can be eliminated altogether by using this modern technology. It was observed that *p*H is the major factor contributing to fouling of the membrane[6]. The *p*H level to which the juice should be limed was optimized by testing at various *p*H 7.5, 8.5 and 10. It was found that for this membrane, least fouling took place at *p*H 7.5. The calcium level in the decanted juice was 75 ppm, which is well below the level to cause scaling in the evaporator which follows the UF step. The clarity of the juice improved by this process by decrease in turbidity from 200 to 50 Nephelometric Turbidity Unit (NTU).

Microfiltration of sugarcane juice—Microfiltration (MF) is generally done prior to all ultrafiltration (UF) processes. MF is essential, because any suspended materials, if present, will foul the UF membrane rapidly. A rapid flux decline was observed during microfiltration of unlimed juice due to the presence of a large quantity of colloidal and gummy materials. This observation led to the idea of liming of juice prior to MF. In this case, no appreciable flux declination was observed under identical conditions. The clarity of the permeate obtained was also better when compared with the microfiltrate of unlimed juice as can be seen from Table 1. Therefore, liming is important before microfiltration. The clogging of microfilters was removed by backflushing with demineralized water after each experiment.

Clarification of microfiltrate by hollow fibre ultrafiltration membrane—Polysulphone based ultrafiltration membranes in hollow fibre form

Table 1—Effect of pH on turbidity in the clarification of sugarcane juice in each process

Process	Turbidity, NTU			
	Raw juice	Limed juice		
	$pH=5.6$	$pH=7.5$	$pH=8.5$	$pH=10$
Initial or unlimed	125	185	160	120
After liming	NA	65	70	60
After MF	110	40	40	25
After UF	0.5	0.3	0.2	0.1

Table 2—Study of clarification of limed sugarcane juice having initial sucrose concentration of 12% and pH of 7.5, by hollow fibre ultrafiltration membrane module having an area of 0.1873 m^2 at 1.2 kg/cm^2 TMP

Time min	Flux LMH	% Sucrose rejection	Turbidity of permeate (NTU)
10	0.242	8.19	0.35
25	0.223	6.44	0.4
60	0.193	4.68	0.3
90	0.183	4.68	0.3
120	0.173	3.51	0.35
150	0.168	3.51	0.3
180	0.155	3.51	0.3
210	0.153	3.51	0.3
240	0.150	3.51	0.3
270	0.146	3.51	0.3
300	0.145	3.51	0.3
330	0.142	3.51	0.3
360	0.142	3.51	0.3

LMH = Litre/square metre/hour.

Table 3—Study of effect of pH on the efficacy of the filtration system in terms of its fouling characteristics at 1.2 kg/cm^2 TMP

	Raw juice	Limed juice		
	$pH=5.6$	$pH=7.5$	$pH=8.5$	$pH=10$
Flux (LMH)	0.043	0.168	0.151	0.158
% Rejection of sucrose	5.7	3.51	3.8	5.7
PWP before juice run (LMH)	1.118	1.118	1.119	1.101
PWP after juice run before restoration (LMH)	0.152	0.482	0.432	0.457
PWP after juice run after restroation (LMH)	1.037	1.038	1.025	1.024

having molecular weight cut-off of 20,000 developed by us were used for these clarification studies. These membranes were characterized with polyethylene glycol with a polydispersity in molecular weight ranging from 1,000 to 20,000 Daltons. The microfiltrates obtained from unlimed and limed juices (various pH) were used to investigate the fouling characteristics and to optimize the pH for this clarification process. The best clarification along with a maximum and steady flux was obtained from the feed juice limed to pH of 7.5 (Table 2). It can be seen from Fig. 2, that the flux of the limed juice is more than double the flux of the unlimed juice and it can be said for certain that the liming of the juice is unavoidable. It is also observed that beyond the pH of 7.5, no significant improvement occurs in flux and in fact flux decreases to an extent. This behaviour may be attributed to colloidal precipitation at higher pH, which leads to fouling at the membrane surface, which in turn results in a decline in the flux.

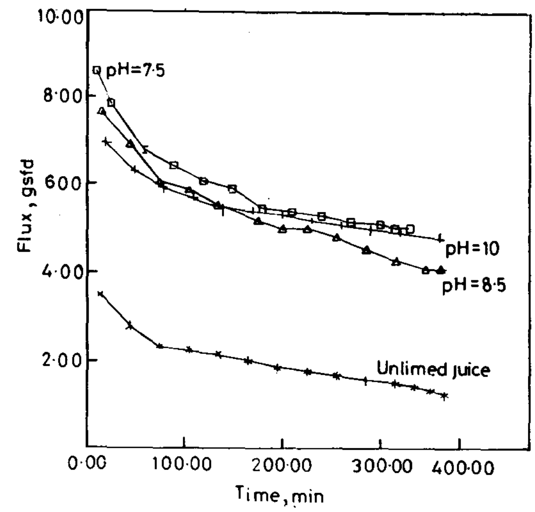

Fig 2—Rate of fouling of HF ultrafiltration membrane at various pH of the feed solution

Fouling is an inherent property of any membrane when it is subjected to macromolecular filtration. Sugarcane juice is one of the extremely complex colloidal solutions among the natural products. As a result, the flux decline during the clarification step is rapid initially and thereafter quickly stabilizes once a dynamic equilibrium of macromolecular diffusion is established between the bulk and the gel membrane while the thickness of the gel layer remains constant. Percentage rejection of sucrose was observed to be a mere 3.51% (Table 3) whereas that for macromolecules, as can be expected, is 100%, which was qualitatively analyzed by viscosity measurements of both feed and permeate samples. A very clear, light coloured juice devoid of macromolecules was obtained from this clarification process. Since most of the macromolecules and gummy materials are removed, it is anticipated that the sugar content in the molasses will also be minimized and an improved quality of the molasses will be obtained for better fermentation. The crystallization of sugar will be faster with a high yield and low coloured raw sugar.

After each ultrafiltration trial, the hollow fibre membrane module was washed with demineralized water for one hour, after which it was observed that the pure water flux is reduced to the extent of 50%. This clearly indicates that a gel layer is strongly adsorbed at the membrane surface. In order to remove this layer, the module was flushed with a cleaning solution developed by IEI, Bombay. The cleaned module gave a water flux which was 95% of its initial value. Since the cleaning solution contains anti-microbial agents, it is expected that any microbial attachment on the membrane surface would be eliminated.

The clarified juice is a rich nutrient medium for microbial growth and leads to a rapid fermentation. To investigate the shelf life of this ultrafiltered juice, the microbial content of the juice was analyzed. It is observed that the permeate is free from any form of microbes, since it is predictable that the 20,000 molecular weight cut-off membrane will not allow them to permeate. The microbial count in the juice was nil even after storing the juice in a sterilized container for 24 h at room temperature. It is evident from this study, that the filtrate can be stored without any deterioration for a minimum of 24 h which is otherwise impossible.

Conclusions

This polysulphone based hollow fibre membrane developed by Ion Exchange (India) Ltd, Bombay, is suitable for the clarification of sugarcane juice with favourable characteristics. The energy utilization by the complex conventional processes can be largely minimized by using this modern, simple and hygienic clarification process using synthetic polymeric membranes having wide *p*H tolerance which is essential for this purpose.

References

1 Sourirajan S, *Ind End Chem Proc Design Dev*, 6 (1967) 154.
2 Madsen R F, *Int Sugar J*, 75 (1973) 163.
3 Kishihara S, Fujji S & Komoto M, *Int Sugar J*, 83 (1981) 35.
4 Kishihara S, Fujji S & Komoto M, *Int Sugar J*, 85 (1983) 99.
5 Kofod Nielsen W, Kristensen S & Madsen R F, *Sugar Technol Rev*, 9 (1982) 59.
6 Tragardh G & Gekas U, *Desalination*, 69 (1988) 9.