



# Ceramic membrane filtration of factory sugarcane juice: Effect of pretreatment on permeate flux, juice quality and fouling

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## ABSTRACT

The effect of pretreatment of sugarcane juice and juice composition on the performance of a ceramic membrane with pore size of 20 nm has been studied. Pretreatment options assessed included heating juice (60, 75 and 90 °C) with and without sedimentation at specified pHs (7.2, 7.5 and 7.8) and using evaporator supply juice (ESJ). The average permeate flux at 75 °C and pH 7.8 for limed juice, and partially clarified juice, and ESJ were 278 L/m<sup>2</sup>h, 248 L/m<sup>2</sup>h and 160 L/m<sup>2</sup>h in that order. It was shown that with ESJ, where the juice is boiled, there are higher proportions of crystalline and microcrystalline phases, and other impurities which reduces the efficiency of the membrane filtration process as the cake resistance ( $1.17 \times 10^{12}$ /m) is 1.7 times higher than of the partially clarified juice. With respect to the juice quality, the results of the permeate derived from the partially clarified juice at 75 °C, show that the lowest pH value of 7.2 gave the largest impurity reduction, though liming at pH 7.5 would be the preferred treatment for industrial application because of pH drop during processing. The fouling mechanism for the partially clarified juice was shown to be the combined cake filtration-complete blocking model.

## 1. Introduction

The solid/liquid separation process is a necessary step in the production of good quality sugar in high yields as it affects the juice quality and properties, as well as the rate of sugar crystallization. In the sugar manufacturing process, the juice is clarified to remove suspended and colloidal particles and non-sugar impurities (polysaccharides, waxes, proteins, salts etc) so as to improve juice quality, evaporator efficiency, sucrose crystallization and the quality and quantity of the sugar produced (Doherty et al., 2010). The traditional sugar manufacturing process normally adopts the defecation method for raw sugar production, and the sulfitation and carbonation methods for plantation white sugar.

Membrane technology has been recognized as a standard tool in the food and beverage industry and is being employed in the processing of fruit and vegetable juices (Saha et al., 2007). The benefits of membrane filtration for sugarcane juice streams have been proven as it significantly reduces juice colour and colour precursors, and reduces low

and high molecular weight proteins and polysaccharides. The reduction of these macromolecules lowers syrup viscosity, which allows higher quality and lower colour sugars to be produced and improves pan productivity and fugging (which is a process to remove the syrup from the sugar crystals by centrifugation) efficiency (Steindl, 2001). As a consequence, over the last two decades, clarification of sugarcane juice by microfiltration and ultrafiltration has been extensively explored using both polymeric and inorganic membranes (Steindl and Doyle, 1999). Due to the lower cost of polymeric membranes, exhaustive investigations were conducted which focused on operating parameters and juice pretreatment procedures (Balakrishnan et al., 2000; Sim et al., 2009), sugar retention and the fouling mechanisms (Saha et al., 2006, 2007, 2009; Bhattacharya et al., 2001). A pilot demonstration carried out in an Indian sugar factory with polymeric membranes exhibited low permeate flux and revealed that the juice quality was a critical parameter that influenced the membrane performance (Ghosh and Balakrishnan, 2003).

In recent times, ceramic and stainless steel membranes have been

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evaluated for sugar manufacturing because they can be operated at high temperatures thereby reducing the impact of the juice viscosity on the permeate flux. Their use have resulted in the production of high quality sugar (Fechter et al., 2001; Jegatheesan et al., 2012). However, commercial factories using these types of membranes have ceased operation mainly due to high maintenance and operational costs, low process productivity, and value adding to the retentate which contains reasonable amounts of sucrose.

Jegatheesan et al. (2009) considered a pretreatment option to improve permeate flux. In the study, pretreated filtered raw juice was passed through a 250  $\mu\text{m}$  sieve with lime to raise the pH to 7.5 and the floc particles formed allowed to settle prior to decantation and filtering the clear juice with a 125  $\mu\text{m}$  sieve. The permeate flux dropped from  $\sim 94 \text{ L/m}^2\text{h}$  in 1 h– $45 \text{ L/m}^2\text{h}$  in 4 h with a ceramic membrane having a pore size of 50 nm, levels, partly due to the low temperature, of  $60^\circ\text{C}$  and transmembrane pressure (TMP) value of 0.1 MPa. Besides, the study was conducted with no heating of the limed juice. Li et al. (2017) complemented this study by working with mixed juice collected from a Chinese sugar factory and the limed juice heated at  $85\text{--}95^\circ\text{C}$  under similar conditions used by sugar factories. The study showed a ceramic membrane with 50 nm pore size achieved a permeate flux of between 119 and  $142 \text{ L/m}^2\text{h}$  over 10–25 h and purity increase over filtration of  $> 1.2$  units, which is slightly higher than those obtained by juice clarification methods. Recent work by Li et al. (2018) obtained similar average permeate fluxes with limed heated juice. The improvement in performance may be due, in part, to the juice pretreatment process used in these studies. However, none of these studies compared the different pretreatment/clarification options on the performance of the ceramic membranes in order to identify preferred options for industrial application of membrane technology for sugarcane juice processing.

Membrane fouling during the filtration process is a critical issue that must be addressed for the successful application of membrane technology for the sugarcane industry (Luo et al., 2016; Urošević et al., 2017). Various mathematical expressions exist to describe the fouling mechanism. Four mechanistic models typically used include standard blocking, complete blocking, intermediate blocking, and cake filtration (Giglia and Straeffler, 2012; Hou et al., 2017). Jegatheesan et al. (2009) reported that the cake filtration model was the best model to describe the fouling in ceramic membranes with pore sizes of 20, 50, or 100 nm with partially lime juice. Other studies suggest that while cake filtration is predominant, multiple fouling mechanisms occur and no single model can be used to fully explain the fouling behaviour in processing sugarcane juice (Li et al., 2018). So, when more than one fouling mechanisms are simultaneously occurring, the theoretical tools to interpret fouling behaviour are limited (Katsoufidou et al., 2005). In this regard, researchers have combined two or more models (Yuan et al., 2002; Bolton et al., 2006a, 2006b). A combined cake filtration-complete blocking model was used provide the best fit for large macromolecules/high molecular weight compounds in biological fluids (Bolton et al., 2006b), it was envisaged that it would do the same for the foulants in sugarcane juice as they also contain macromolecules – proteins and polysaccharides.

The present study investigated, for the first time, the effect of pre-treating factory sugarcane juice at various temperatures and pHs on permeate flux, juice quality and membrane fouling. The physico-chemical attributes of the treated juice properties that accounted for differences in the permeate flux among the treated juices are discussed. The composition and morphological features of the surface of the fouled membranes were examined and different fouling mechanism models during the filtration of sugarcane juice using a ceramic membrane with a pore size of 20 nm. The foulants were characterized by scanning electron microscopy (SEM) equipped with an energy dispersive analysis of x-rays (EDAX), Fourier transform infrared resonance (FTIR) spectroscopy, two-dimensional nuclear magnetic resonance (2D-NMR) spectroscopy heteronuclear single quantum coherence (HSQC), and X-ray powder diffraction (XRD). The overall outcome of the study

provides further insights on the use of membranes towards developing more efficient technology for processing sugarcane juice. Further research would also be required to address the other major impediment to membrane technology application in sugar production which is associated with adding value to the retentate stream.

## 2. Materials and methods

### 2.1. Sugarcane juice

The membrane filtration experimental program was conducted at Rocky Point Sugar Mill, Queensland, Australia during the 2017–2018 crushing season. Raw mixed juice was collected from the outlet of the factory mixed juice tank. The mixed juice comprises juice obtained from the Nos. 1 and 2 mills. Raw juice was also collected from the boot of the No. 2 mill. The No. 2 mill juice is of lower purity than No. 1 mill juice and hence contains less sucrose. The collected mixed juices were filtered with 150  $\mu\text{m}$  sieve to remove bagacillo and solids prior to pretreatment. Factory clarified juice was collected from the outlet of the evaporator supply juice (ESJ) tank.

### 2.2. Membranes

Two types of membranes, designated A and B, having the same pore size of 20 nm but obtained from different manufacturing processes were investigated. The specifications of the membranes are given in Table 1.

Before selecting the membrane pore size of 20 nm, the particle size distribution of partially clarified juice and permeate obtained with membrane A were analyzed using a Zetasizer. Three main size ranges 1–5 nm, 100–500 nm and 3000–5000 nm were obtained for the juice. After the juice was filtered through the membrane the particle size in the range from 500 to 5000 nm was completely removed. The particle size in the range 1–5 nm did not change indicating that working with a membrane size of 20 nm would allow sucrose molecules to pass through.

### 2.3. Pretreatment strategy

Fig. 1 shows the various pretreatment strategies studied: Strategy 1: Filtered and heated ( $60, 75$  and  $90^\circ\text{C}$ ) mixed juice was limed with lime saccharate to a specific pH (7.2, 7.5 and 7.8). The factory prepared lime saccharate by mixing 200 g/kg solution of CaO and 68 brix factory syrup ( $\sim 1:1$  ratio). Strategy 2: Filtered and heated mixed juice was limed with lime saccharate to specific pH followed by 1 h settling of the floc particles and collection of the supernatant for membrane treatment – designated partially clarified juice. Strategy 3: Clarified juice was collected from the ESJ tank. The defecation process at the factory

**Table 1**  
Material and module details of the membranes.

Item	Description	
	Membrane A	Membrane B
Manufacturer	Membrane Science and Technology research center, Nanjing University of Technology, China	Jiangsu Jiuwu HiTech, Nanjing, China
Membrane type	Tubular	Tubular
Membrane material	$\text{Al}_2\text{O}_3$	$\text{ZrO}_2$
Membrane support material	$\text{Al}_2\text{O}_3$	$\text{Al}_2\text{O}_3$
Pore size	20 nm	20 nm
Portable water flux	$230 \text{ L/m}^2\text{h}$ bar	$200 \text{ L/m}^2\text{h}$ bar
Length	500 mm	500 mm
Number of channels	19	19
Channel diameter	4 mm	4 mm
Surface area	$0.1193 \text{ m}^2$	$0.1193 \text{ m}^2$

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