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Pretreatment with ceramic membrane microfiltration in the clarification process of sugarcane juice by ultrafiltration

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ABSTRACT. In the present study, the sugar cane juice from COCAFE Mill, was clarified using tubular ceramic membranes (α-Al_2O_3/TiO_2) with pore size of 0.1 and 0.3 μm, and membrane area of 0.005 m^2. Experiments were performed in batch with sugar cane juice, in a pilot unit of micro and ultrafiltration using the principle of tangential filtration. The sugar cane juice was settled for one hour and the supernatant was treated by microfiltration. After that, the MF permeate was ultrafiltered. The experiments of micro and ultrafiltration were carried out at 65ºC and 1 bar. The ceramic membranes were able to remove the colloidal particles, producing a limpid permeated juice with color reduction. The clarification process with micro- followed by ultrafiltration produced a good result with an average purity rise of 2.74 units, 99.4% lower turbidity and 44.8% lighter color in the permeate.

Keywords: pretreatment, ceramic membrane, sugar cane juice, clarification.

Efeito do pré-tratamento por microfiltração com membrana cerâmica no processo de clarificação do caldo de cana por ultrafiltração

RESUMO. No presente trabalho, o caldo de cana, proveniente da usina COCAFE, foi clarificado utilizando membranas cerâmicas tubulares (TiO_2/ α-Al_2O_3), com diâmetros de poros de 0,1 e 0,3 μm e área de filtração de 0,005 m^2. Os experimentos com caldo de cana-de-açúcar foram realizados em batelada em uma unidade piloto de micro e ultrafiltração, que utiliza o princípio de filtração tangencial. O caldo de cana-de-açúcar foi sedimentado durante uma hora e o caldo sobrenadante foi tratado por microfiltração e, em seguida, o permeado obtido deste processo foi ultrafiltrado. Os experimentos de micro e ultrafiltração foram realizados à temperatura de 65ºC e pressão transmembrana de 1 bar. Os resultados mostraram que as membranas cerâmicas foram capazes de remover as partículas coloidais, produzindo um caldo permeado límpido e com redução de cor. O processo de clarificação com micro seguido de ultrafiltração apresentou bons resultados, promovendo um aumento de pureza de 2,74 unidades, com redução de turbidez de 99,4% e com redução 44,8% de cor.

Palavras-chave: pré-tratamento, membrana cerâmica, caldo de cana, clarificação.

Introduction

Sugarcane belongs to the family of grasses (*Saccharum officinarum*) from Asia. The sugarcane juice is a colloidal suspension whose color varies from dark green to brown. This color results from the presence of substance such as chlorophyll, anthocyanin, xanthophyll, and carotene. The opacity is caused by colloids, proteins and inorganic salts (MARAFANTE, 1993).

In the conventional process of white sugar production, the sugarcane is sulfated in an SO_2 column. Then the juice undergoes an alkalization process with lime milk, reaching a pH from 6.8 to 7.2. After sulfation and alkalization, the suice is heated to about 105ºC aiming to speed up and ease the coagulation and flocculation by sedimentation.

One disadvantage of conventional clarification of the sugar cane is the high amount of inputs used as sulfur and lime. Besides, the conventional production of white sugar features inefficient removal of substances like starch, silica, ash, colloids, during the clarification, which in turn affect the color of the final product (BHATTACHARYA et al., 2001).

An alternative to the conventional process is the use of separation by membranes which has several advantages: energy saving, selectivity, ease of operation, and reduced consumption of chemicals.

According to Saha et al. (2006) the ultrafiltration permeate is characterized by high clarity, low viscosity, and color reduction, which eventually results in a higher quality sugar. However, its use is limited by a significant flow reduction due to the combined concentration polarization and fouling.

This study aimed to evaluate the influence of pretreatment with microfiltration in the clarification process of sugarcane juice by ultrafiltration.

Material and methods

Sugarcane juice (mixed juice) used in the clarification tests was furnished by the COCAFE alcohol producing unit in 2007. The juice was placed in 5 liters-drums and stored at -10ºC.

The experiments of membrane filtration were performed in the module of micro/ultrafiltration that uses the principles of tangential filtration with membranes, as shown in Figure 1.

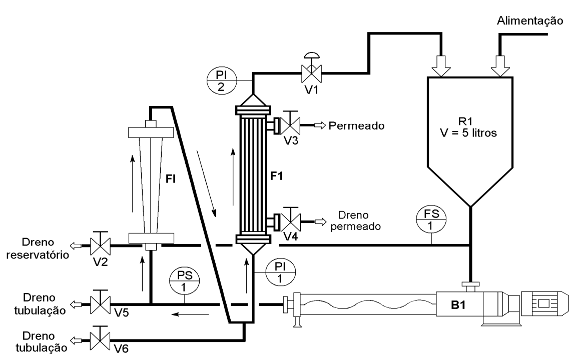

Figure 1. Schematic drawing of the micro/ultrafiltration module. F1) Membrane module; B1) Peristaltic pump; R1) Feed Tank; V2, V3, V4, V5, V6) Drain valves; FI) Rotameter; V1) Pressure Regulating Valve; PS1, PS2) Gauges; FS) Flow sensor;PS) Pressure sensor.

For the clarification tests, it was used TiO_2/α-$Al2O_3$ ceramic membranes with pore diameters of 0.3 and 0.1 μm and area of 0.005 m^2, provided by NETZCH.

For each experiment, the juice was thawed and subjected to natural sedimentation for one hour, without adding chemicals. Then the trials were performed as follow:

• Method A: microfiltration (pretreatment) followed by ultrafiltration.

• Method B: ultrafiltration.

For the method A, the supernatant was fed to the module and microfiltered. The achieved permeate was then submitted to ultrafiltration. In the method B, the supernatant juice was directly ultrafiltered. In the trials of micro/ultrafiltration, we evaluated the flow, percentage of fouling, and the permeate quality at 1 bar and 65°C. The module was operated in batch with complete recycle of the retentate.

The permeate flow was obtained through collecting permeate mass over time, measured in a semi-analytical scale (BG 4000 - Gehaka), and calculated by the Equation 1.

$$J_{perm} = \frac{m_p}{A.t} \quad (1)$$

where:

J_{perm} is the permeate flow (kg h^{-1} m^2);

m_p is the mass (kg) gathered in the time interval;

t is the time interval (hours);

A is the membrane permeation area (m^2).

The main analytical methods used in the analyses of the fed juice (supernatant juice) and permeate juice, were based on the methods recommended by ICUMSA (International Commission for Uniform Methods of Sugar Analysis), such as:

- Brix (total dissolved solids), using a refractometer (Shimadzu; ABBE-3L);
- Pol (a measure of the total polarizing substances, primarily sucrose), using an automatic digital polarimeter (ACATEC; PDA 8300);
- Juice purity (sucrose fraction in dissolved solids), according to Equation 2;

$$\text{Purity (\%)} = (\text{Pol / Brix}) * 100 \quad (2)$$

- ICUMSA Color (absorbance index of a sugar solution, at 420 nm multiplied by 1000), using a Shimadzu spectrophotometer (UV-1203);
- pH, using a Digimed pHmeter (DM-20).
- Turbidity (reduction in transparency of a liquid caused by the presence of undissolved solids) using a Shimadzu spectrophotometer (UV-1203);

The membrane performance in the micro/ultrafiltration of sugar cane was evaluated by the purity rise, sugar, non-sugar, and brix rejection, color reduction, and fouling index, which were calculated according to Equations 3, 4, 5, 6, 7 and 8, respectively.

$$\text{Purity rise} = Purity_p - Purity_a \quad (3)$$

$$\text{Sugar Rej.(\%)} = \left[1 - \left(\frac{Pol_{p.}}{Pol_{a.}}\right)\right] * 100 \quad (4)$$

$$\text{Non-sugar Rej.(\%)} = \left[1 - \left(\frac{(Brix - Pol)_p}{(Brix - Pol)_a}\right)\right] * 100 \quad (5)$$

$$\text{Brix Rej.(\%)} = \left[1 - \left(\frac{Brix_p}{Brix_a}\right)\right] * 100 \quad (6)$$

$$\text{Fouling } (\%) = \left(\frac{PAP_{limpa} - PAP_{suja}}{PAP_{limpa}} \right) * 100 \qquad (7)$$

$$\text{Color Reduction } (\%) = \left(\frac{Cor_a - Cor_p}{Cor_a} \right) * 100 \qquad (8)$$

where:
PAP is the pure water permeability, the lower index 'a' and 'p' refer respectively to fed and permeate juice.

The cleaning of the membranes was performed in ultrasonic bath with NaOH 1% at 70ºC. After each regeneration cycle, it was measured the flow with deionized water, ensuring the reproducibility of the experiments.

Results and discussion

The characteristics of the sugar cane juice used in the clarification tests are listed in Table 1.

Table 1. Characteristics of the juice.

Property	Value
pH	5.27
Brix (%)	13.3
Pol (%)	9.3
ICUMSA Color	6,810.0

In the Figure 2 is presented the permeation curve of the sugar cane juice using the microfiltration as a pretreatment at 1bar and 65ºC.

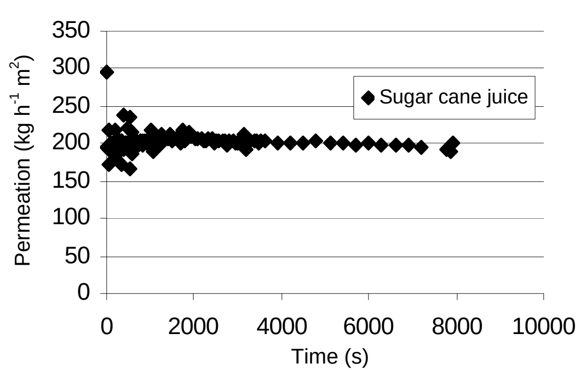

Figure 2. Permeation curve of the sugar cane juice over time (65ºC, 1 bar).

The steady flow of 0.3 μm-membrane, at 1 bar and 65ºC, is 198.38 kg h^{-1} m^{2}.

The permeation curves in the ultrafiltration of the pretreated juice and the supernatant juice at 1bar and 65ºC are shown in Figure 3.

In the first minutes, there was a rapid decay of the flow, typically observed in the separation by membranes, indicating the formation of a secondary filtration layers on the membrane surface, which provides an additional resistance to the permeate flow. Then the flow decreased slowly until stabilizing around 253.62 kg h^{-1} m^{2} for the pretreated juice and around 179.8 kg h^{-1} m^{2} for the supernatant juice.

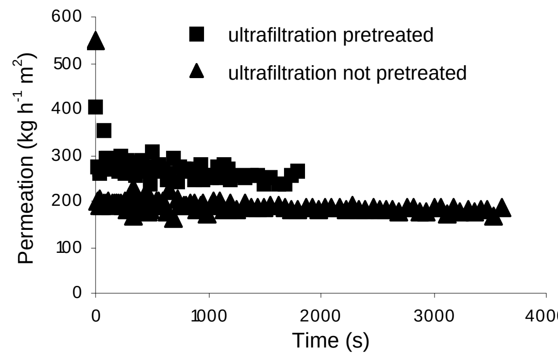

Figure 3. Permeation curve of the sugar cane juice over time (65ºC, 1 bar).

The characteristics of the fed and permeate juices of the method A and B are listed in the Table 2. There was a reduction for both methods, indicating that both method A and method B was effective to remove impurities.

Table 2. Characteristics of the fed and permeate sugar cane juice.

		Method A 0,3		Method B
		0.3 μm	0.1 μm	0.1 μm
Fed	pH	5.19	5.23	5.34
	Brix (%)	14.2	13.3	12.4
	Pol (%)	9.42	8.85	9.20
	Purity	66.3	66.54	74.20
	ICUMSA Cor	5,320	1,830	8,300
	Turbidity	3,240	19	3,393
Permeate	pH	5.21	5.24	5.37
	Brix (%)	13.6	13.2	11.9
	Pol (%)	9.35	8.82	9.13
	Purity	68.75	66.84	76.69
	ICUMSA Color	3,460	1,650	5,300
	Turbidity	19	6	8

The Table 3 shows the performance of the Methods A and B in the clarification process of the sugar cane juice. It was observed 44.8% reduction in the INCUMSA color, and a purity rise of 2.74 units for the juice of the method A.

Table 3. Membrane performance.

	Method A	Method B
Purity rise	2.74	2.49
Sugar rejection (%)	1.01	0.81
Non-sugar rejection	12.80	13.29
Brix Rejection (%)	4.98	4.03
Color reduction (%)	44.80	36.14
Steady flow (kg h^{-1} m^{-2})	253.62	179.8
Fouling (%)	57.64	81.27

For the juice treated by microfiltration, there was an increase of 29.01% in the steady flow and a fouling reduction of 29.07%. According to Habert et al. (2006), in the microfiltration, the solvent and all soluble material permeate the membrane and only the suspended material is retained. As the mixed juice has suspended materials, the juice pretreated by microfiltration minimized the fouling effects and increased the flow in the ultrafiltration step.

According to the Table 3, the rejection of sugar was lower than 1.01% and the rejection of non-sugar was above to 12.8, evidencing that ceramic membranes are able to retain colloidal materials and dissolved impurities (non-sugar) with little change in sugar levels.

The Figure 4 shows the performance of the Methods A and B. The percentage of the values was proportionally calculated.

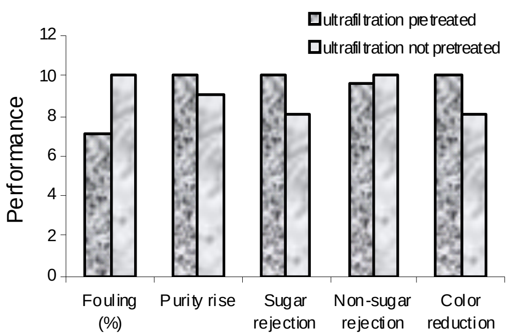

Figure 4. Performance of the methods A and B.

In the Figure 4 it was possible to observe a reduction of *fouling* and increase of purity in the juice pretreated with microfiltration.

The Figure 5 shows the performance index of the methods. It was calculated by summing the obtained values for each evaluated parameter.

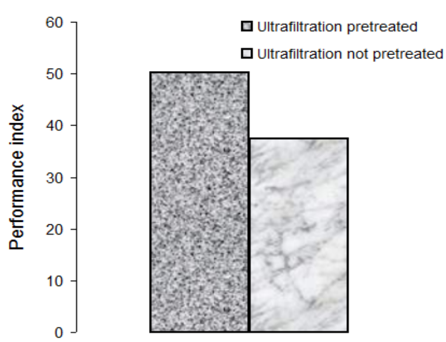

Figure 5. Performance index of the methods A and B.

The performance indices were 50.21 and 37.47 for the methods A and B, respectively. The method A, which employed the microfiltration as a pretreatment, had the highest index.

Conclusion

The clarification trials for the sugar cane juice showed that the ceramic membrane of micro/ultrafiltration was able to remove colloidal particles, producing a limpid permeated juice with a purity increase of up to 2.74 units.

The pretreatment with microfiltration, using a 0.3 μm-ceramic membrane, was able to remove suspended impurities, promoting an increase of 29.01% in steady flow and fouling reduction of 29.07% in the ultrafiltration step. The method A, which uses the microfiltration as a pretreatment, featured the highest performance index, by decreasing the fouling effect and increasing the flow in the ultrafiltration step of the clarification process of sugar cane juice.

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