

Acta Scientiarum. Technology

ISSN: 1806-2563

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Universidade Estadual de Maringá

Brasil

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Acta Scientiarum. Technology, vol. 34, núm. 4, octubre-diciembre, 2012, pp. 413-419

Universidade Estadual de Maringá

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## Comparison between microfiltration and addition of coagulating agents in the clarification of sugar cane juice

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**ABSTRACT.** This study accomplished a comparison between microfiltration and addition of coagulating agents to clarify sugar cane juice. Microfiltration tests were carried out using ceramic tubular membranes made with  $\text{TiO}_2/\alpha\text{-Al}_2\text{O}_3$ , with pore diameter of 0.2; 0.4 and 0.6  $\mu\text{m}$ . The transmembrane pressures applied were 1.0, 2.0 and 3.0 bar, and temperature was kept constant at 20°C. Clarification test with addition of coagulating agents were performed with PAC and  $\text{Ca}(\text{OH})_2$  at 65°C. The highest permeate flow was 76  $\text{kg h}^{-1} \text{m}^{-2}$  at 1.0 bar with 0.6  $\mu\text{m}$ -membrane. The clarification process with membranes achieved a reduction of turbidity and color superior to 92 and 16%, respectively. In the clarification by adding coagulating agents we verified a reduction superior to 78 and 46% to turbidity and color, respectively.

**Keywords:** microfiltration, coagulating agents, clarification, sugar cane.

## Comparaç o entre microfiltra o e adi o de agentes coagulantes na clarifica o do caldo de cana

**RESUMO.** Neste trabalho foi realizada a compara o entre a microfiltra o e adi o de agentes coagulantes na clarifica o do caldo de cana. Os testes de microfiltra o foram realizados com membranas tubulares cer micas feitas de  $\text{TiO}_2/\alpha\text{-Al}_2\text{O}_3$  e di metro de poros iguais a 0,2; 0,4 e 0,6  $\mu\text{m}$ . As press es transmembranas aplicadas foram de 1,0; 2,0 e 3,0 bar e a temperatura foi mantida constante e igual a 20°C. A clarifica o com adi o de agentes coagulantes foi feita com adi o de policloreto de alum nio e  $\text{Ca}(\text{OH})_2$  a 65°C. O maior fluxo de permeado foi de 76  $\text{kg h}^{-1} \text{m}^{-2}$  a 1,0 bar com a membrana de 0,6  $\mu\text{m}$ . O processo de clarifica o com membranas alcan ou redu o de cor e turbidez superiores a 16 e 92%, respectivamente. Na clarifica o com adi o de agentes coagulantes foi obtida redu o de cor e turbidez superior a 46 e 78%, respectivamente.

**Palavras-chave:** microfiltra o, agentes coagulantes, clarifica o, cana de a u ar.

### Introduction

Currently sugar cane juice arouses the interest of researchers since it is a popular drink that can be consumed alone or with added fruit juice. Moreover, the drink is able to replace the energy rapidly because of its sugars. According to Soccol et al. (1990) the nutritionists affirms that the sugar cane juice has an excellent hydrating capacity, rich in carbohydrates, iron and vitamins, ideal to be consumed after practicing exercises to replenish minerals and water.

The industrialization of sugar cane juice has proven to be a lucrative business for both domestic market and export. But for this, it is required to obtain a process aiming its stabilization, since it is an extremely perishable product because of its high sugar content. Just minutes after extraction, the sugar cane juice gets very dark color by the oxidation

of its components (chlorophyll and polyphenols), providing an unpleasant aspect for the possible consumers.

One of the processes indicated to enable the sugar cane juice industrialization is the clarification, which can be achieved through two methods: the conventional adapted to consumption, and by membrane separation (PRATI et al., 2005).

In Brazil, two models of conventional clarification are predominantly used: the addition of quicklime which employs lime and heat to obtain raw sugar, and the sulphodefecation – which, prior the treatment with lime and heat, adds  $\text{SO}_2$  to the juice to get white crystal sugar. Prati et al. (2005) states that it was tested a new coagulant developed in Japan, the polyaluminum chloride (PAC) that has some advantages in relation to the usually employed coagulants: promotes flocculation in any pH range;

it is more effective than aluminum sulfate in removing colloids, with less expenditure of reagents; it generates less aluminum residue in the final product.

The other method used in the clarification process of sugar cane juice is the microfiltration, widely used in food industry for clarification and sterilization, replacing conventional techniques. The microfiltration is the separation process with membranes, similar to classical filtration, being the indicated process to retain material in suspension and emulsion, besides sterilizing the product at room temperature. This process has several advantages, such as: energy efficiency, selectivity, simplicity of operation, and reduced consumption of chemicals. As the microfiltration is a physical process, its advantage is the achievement of a natural juice, without adding chemicals.

The clarification of sugar cane juice through microfiltration and ultrafiltration has been extensively examined in laboratories for sugar production (BHATTACHARYA et al., 2001; DORNIER et al., 1995; GHOSH; BALAKRISHNAN, 2003; SAHA et al., 2007). These authors assure that the permeate in the tests of micro- and ultrafiltration produces a clarified sugar cane juice, with low viscosity and excellent color reduction.

In this way, the goals of this study were: (i) to analyze the clarification of sugar cane juice using microfiltration and conventional clarification using PAC; (ii) to proceed physical, chemical and microbiological comparisons of the sugar cane juice clarified by these techniques.

## Material and methods

### Raw material

The sugar cane juice used in the clarification tests was purchased from a street vendor in Maringá city, Paraná State. The juice was separated in 4 L - containers and stored at  $-10^{\circ}\text{C}$ . For each test we defrosted only the amount necessary for the experiment.

### Clarification by microfiltration

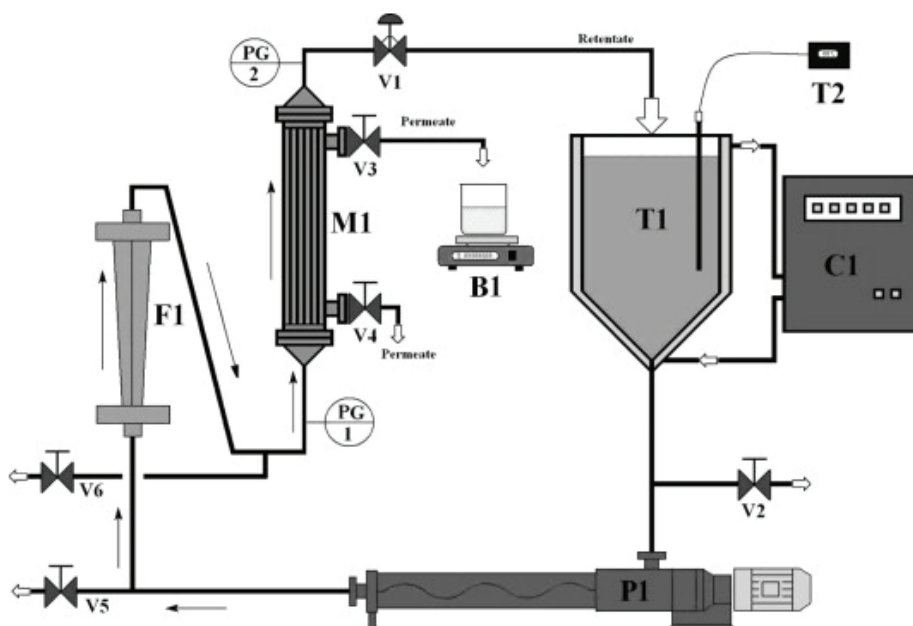
The experimental design of the tests performed in the first step of microfiltration is listed in Table 1.

**Table 1.** Experimental design for the tests of microfiltration.

Factor	Specifications	Levels
Transmembrane pressure (bar)	1.0, 2.0 and 3.0	3
Membrane pore diameter ( $\mu\text{m}$ )	0.2, 0.4 and 0.6	3
Temperature ( $^{\circ}\text{C}$ )	20	1
Total of tests per microfiltration	-	9

For each experiment, 4 L of sugar cane juice were fed to filter unit and subjected to the tests with different membranes and transmembrane pressures. The experimental procedure adopted in this stage consisted of fixing one variable and evaluate the effect of variation of the others.

The filtration tests with membranes were performed in the microfiltration unit, which uses the principles of tangential filtration, as shown in Figure 1. In this unit, it was adapted a tubular module with ceramic membranes ( $\text{TiO}_2/\alpha\text{-Al}_2\text{O}_3$ ), filtration area of  $0.005\text{ m}^2$ , internal diameter of 7 mm and average pore diameter of 0.2, 0.4 and  $0.6\ \mu\text{m}$ .



**Figure 1.** Schematic drawing of the microfiltration pilot unit.

where:

- M1 - membrane module;
- P1 - screw pump;
- T1 - reservoir of juice;
- F1 - rotmeter;
- V1 - pressure regulating valve;
- V2, V3, V4, V5, V6 - drain valves;
- T2 - thermocouple;
- B1 - scale;
- PG1, PG2 - gauges;
- FS - flow sensor;
- PS - pressuremeter;
- C1 - thermostatic bath.

For all the tests, the microfiltration unit was operated in batch with the maximum recycled flow supplied by the pump (800 L h<sup>-1</sup>), with total recycle of the juice retained while the permeate was continuously removed, in this way, the juice in the feeding tank has become increasingly concentrate.

#### Clarification with coagulant agent

For the clarification-stabilization of the juice, it was used the process as established by Prati et al. (2005). The juice was heated in a water bath (65°C 50 min.<sup>-1</sup>), alkalized with Ca(OH)<sub>2</sub> to pH 8.0, supplemented with 60 ppm of polyaluminum sulfate and decanted for 45 min.

#### Physical, chemical and microbiological analyses

The permeate, from microfiltration, and the supernatant, from clarification with coagulant agent, were subjected to physical, chemical and microbiological analyses. The microbiological analyses included: yeast and mold count, standard count and MPN for total and fecal coliforms, according to *American Health Association* (VANDERSANT; SPLITSTOESSER, 1992). In physical and chemical analyses we employed the methods adopted by FERMENTEC (AMORIM, 1996), based on methods recommended by Icumsa (1994): °Brix, Pol, color, pH, reducing sugars and turbidity.

Physical and chemical analyses, of the stage of the sugar cane juice microfiltration, are presented by means of the rejection (R) of the parameters evaluated, according to Equation 1.

$$R(\%) = \left[ 1 - \left( \frac{\text{Solute concentration in the permeate}}{\text{Solute concentration in the fed}} \right) \right] \times 100 \quad (1)$$

The choice of the best operating condition for the microfiltration tests was based on the quality of each permeate produced. To facilitate the decision,

it was developed the PQI – Permeate Quality Index – based on Pinto et al. (2008), according to the Equation (2).

$$PQI = \left[ \frac{5 \times (\%R_{\text{turbidity}}) - 3 \times (\%R_{\text{color}}) - 2 \times (\%R_{\text{reducing sugar}}) - 2 \times (\%R_{\text{pol}}) - 2 \times (\%R_{\text{Brix}}) - 4 (\%R_{\text{pH}})}{18} \right] \quad (2)$$

#### Assessment of the effect of temperature on the permeate flow in the microfiltration

The effect of temperature was assessed for the membrane with the highest PQI, i.e., chosen the membrane and transmembrane pressure that provided the highest QCI (quality control index) it was undertaken the experimental test of microfiltration at 35°C.

## Results and discussion

### Raw material

Physical and chemical characteristics of the sugar cane juice are listed in Table 2. The comparison with other studies must be carefully made given that the experimental conditions and products can be quite different. The average values obtained for °Brix and pol were 22.8 e 21.5, respectively, indicating that the sugar cane was not dry when the juice was extracted.

**Table 2.** Physical and chemical characteristics of the sugar cane juice.

Variables	Values
pH	5.47
Soluble Solids (°Brix)	22.8
Turbidity (FAU)	2,863
Reducing sugar (g L <sup>-1</sup> )	5.35
Pol (%)	21.5
Color	1,172

### Permeate flow performance in the microfiltration

Three different membranes (0.2, 0.4 and 0.6 µm) were tested to determine which one will produce the best clarified juice in terms of productivity.

As the sugar cane juice is a colloidal solution of highly complex natural products, in the first minutes there was a drop in the permeate flow that stabilized afterward. This reduction was between 15 and 48% in relation to initial flow. This large reduction in the values of permeate flow is caused by the formation of a boundary layer on the membrane surface, which acts as a secondary membrane. The effects of this secondary membrane added to the retained concentrate and the pore clogging cause a slow and continuous decrease in the permeate flow.

The colloidal materials present in the sugar cane juice have size distribution between 0.1 and 0.5  $\mu\text{m}$ . The Table 3 shows the stabilized flows of the sugar cane juice at 20°C.

**Table 3.** Average flows of the permeate ( $\text{kg h}^{-1} \text{m}^{-2}$ ) at 20°C

Pressure	Membrane 0.2 $\mu\text{m}$	Membrane 0.4 $\mu\text{m}$	Membrane 0.6 $\mu\text{m}$
1.0 bar	56	59	76
2.0 bar	64	64	69
3.0 bar	69	71	75

The increase of transmembrane pressure from 1.0 to 2.0 bar and from 0.2 and 0.4  $\mu\text{m}$  caused increase of 14.3 and 8.5% in the permeate flow, respectively. An antagonistic trend was verified for the 0.6  $\mu\text{m}$  membrane, with decrease in the permeate flow of 9.2% with increase of transmembrane pressure from 1.0 to 2.0 bar. Also, the increase transmembrane pressure from 1.0 to 3.0 bar produced increase of 23 and 20% in the permeate flow, respectively, for the membranes 0.2 and 0.4  $\mu\text{m}$ ; otherwise for the 0.6  $\mu\text{m}$ -membrane, the increase was not significant.

The increase of transmembrane pressure had no significant effect on the permeate flow, because the increase in transmembrane pressure causes an increase of the membrane fouling and, on the contrary of predicted by Darcy's Law, the gain in the permeate flow is not proportional to the applied transmembrane pressure. According to Saha et al. (2007), this trend is expected due to the presence of macromolecules and soluble inorganic compounds responsible for the increased fouling during the micro- and ultrafiltration tests.

As for the pore diameter, considering the pressure of 1.0 bar, there were no expressive increase in stabilized permeate flow between the membranes 0.2 and 0.4  $\mu\text{m}$ , but there was an increase of about 36% when comparing 0.2 and 0.6  $\mu\text{m}$ -membranes. For the other conditions, this increase was lower than 9% in relation to the lowest stabilized flow.

Ghosh and Balakrishnan (2003) used spiral wound membrane module of polyethersulfone with cutting diameter of 32, 40 and 80 kDa in the ultrafiltration tests of sugar cane juice, in the temperature range of 50-53°C. The authors report that the highest permeate flow was 58  $\text{L h}^{-1} \text{m}^{-2}$  at

2.0 atm pressure for the 40 kDa-membrane, being this flow twice higher than obtained with the 80 kDa-membrane. They also observed that under pressure lower than 3.0 atm there is a slight increase in the permeate flow, and with pressure higher than 3.0 atm, there is a reduction in the stabilized permeate flow.

Nogueira and Venturini Filho (2007) have clarified sugar cane juice using ceramic membranes of micro- and ultrafiltration, 0.14  $\mu\text{m}$  and 15 kDa, respectively. Their results show that due to the larger pore size, the microfiltration membrane had average flow of permeate 125% greater than the ultrafiltration one: 50.14 against 22.31  $\text{L h}^{-1} \text{m}^{-2}$ . It is observed that the flows were higher than 56  $\text{L h}^{-1} \text{m}^{-2}$ , being superior to those obtained by Nogueira and Venturini Filho (2007).

### Characterization of the microfiltered juice

The performance of the membranes, according to equation (2), in the microfiltration of the sugar cane juice is listed in Table 4.

In all experimental conditions, the Brix rejection was higher than 15% and the highest occurred for the 0.6  $\mu\text{m}$ -membrane, at 2.0 and 3.0 bar. The greatest rejection of reducing sugar was observed for the 0.2  $\mu\text{m}$ -membrane, at 2.0 bar, whereas the highest rejection of sugar (Pol), for the 0.6  $\mu\text{m}$ -membrane, at 3.0 bar.

The color reduction in the clarified juice is related to the removal of suspended particles that give color to the product, as reported by Dziezak (1990). Significant reductions of color were achieved, the highest of 66.7% for the 0.2  $\mu\text{m}$ -membrane, at 2.0 bar of pressure. According to Collado-Fernández et al. (2000), the turbidity is especially caused by colloidal-size particles in the liquid phase. In this study, the turbidity reductions, acquired with microfiltration, were higher than 94% for all conditions. Nogueira and Venturini Filho (2007) registered turbidity removal of 99.98 and 99.99%, respectively for the membranes of 0.14  $\mu\text{m}$  and 15 kDa.

The Table 5 presents the results relative to PQI for the microfiltration. For the assessment of the best membrane, it was used the PQI, calculated by the equation (2).

**Table 4.** Percentage of rejection of the sugar cane juice clarified by microfiltration.

Membrane	0.2 $\mu\text{m}$			0.4 $\mu\text{m}$			0.6 $\mu\text{m}$		
	1.0	2.0	3.0	1.0	2.0	3.0	1.0	2.0	3.0
Pressure (bar)	1.0	2.0	3.0	1.0	2.0	3.0	1.0	2.0	3.0
Reduction of pH (%)	20.3	22.7	24.5	26.1	20.4	26.6	26.1	24.0	24.9
Rejection of Brix (%)	24.4	22.8	15.2	15.3	17.0	19.0	16.2	25.4	25.4
Reduction of Turbidity (%)	95.7	98.1	95.2	96.0	96.3	99.3	96.3	92.9	94.0
Rejection of reducing sugar (%)	15.7	42.7	21.3	5.7	11.1	16.6	41.1	3.37	14.8
Rejection of sugar (Pol) (%)	17.4	23.0	20.2	22.8	20.7	23.5	24.8	33.4	35.4
Reduction of color (%)	36.0	66.7	21.6	42.7	28.3	25.3	16.4	27.9	28.7

**Table 5.** Permeate quality index (PQI).

Membrane	0.2 $\mu\text{m}$			0.4 $\mu\text{m}$			0.6 $\mu\text{m}$		
	1	2	3	1	2	3	1	2	3
Pressure									
PQI	9.65	1.24	11.09	8.86	12.09	10.87	9.11	8.90	7.35

The membrane with pore diameter 0.4  $\mu\text{m}$ , at 2.0 bar of pressure, presented the highest PQI (Table 5). Importantly, the permeate flow under this condition was 64  $\text{kg h}^{-1} \text{m}^{-2}$ .

#### Effect of temperature on the flow of microfiltered permeate

The microfiltration of the sugar cane juice, using 0.4  $\mu\text{m}$  membrane, at 2.0 bar pressure and 20°C, had the best PQI. In order to evaluate the temperature effect on the permeate flow we proceeded the microfiltration using 0.4  $\mu\text{m}$  membrane, at 2.0 bar pressure, in the temperature of 35°C. The stabilized permeate flow obtained at 35°C was 133  $\text{kg h}^{-1} \text{m}^{-2}$ , which corresponds to a 108% increase, in relation to the permeate flow obtained with the same membrane and pressure, but at 20°C.

Kishihara et al. (1981) observed that a temperature increase from 30 to 60°C doubled the permeate flow. This is attributed to the decreasing viscosity of the juice with increasing temperature. Nielse et al. (1982) also obtained greater flows of permeate at high temperatures.

In the Table 6 are summarized the percentages of reduction for the 0.4  $\mu\text{m}$ -membrane and 2.0 bar in different temperatures. The reduction of color and turbidity were greater at 35°C, but of the other parameters, the reduction is lower than that at 20°C. As the purpose is to reduce turbidity and color, maintaining unchanged the other physical and chemical parameters the temperature increase favors this condition.

In Table 7 is presented the PQI of the clarified juice for the 0.4  $\mu\text{m}$ -membrane and 2.0 bar, at 20°C and 35°C, where one can observe that the PQI values at 35°C were higher than at 20°C.

**Table 6.** Percentage of rejection of the sugar cane juice clarified by microfiltration at different temperatures.

Parameters	Temperature	
	20°C	35°C
Reduction of pH (%)	20.4	20.4
Rejection of Brix (%)	17.0	10.5
Reduction of Turbidity (%)	96.3	98.1
Rejection of reducing sugar (%)	11.0	4.10
Rejection of sugar (Pol) (%)	20.7	7.20
Reduction of color (%)	28.3	41.5

The rejection of sugar can range from 2.8 to 9.6% for polymeric membranes (GHOSH et al., 2000; VERMA et al., 1996).

**Table 7.** PQI values for the 0.4  $\mu\text{m}$ -membrane and 2 bar pressure at different temperatures.

Membrane	0.4 $\mu\text{m}$	
Temperature	20°C	35°C
PQI	12.1	13.4

Thus, the membrane selected to compare with the conventional method is 0.4  $\mu\text{m}$  of average pore diameter, working at 2.0 bar transmembrane pressure and at 35°C.

#### Characterization of the sugar cane juice clarified with coagulant

The clarification performance of the sugar cane juice by adding coagulant agent, according to Equation (1), is presented in Table 8.

**Table 8.** Percentage off rejection of the sugar cane juice clarified by adding coagulant.

Parameter	Value
Reduction of pH (%)	25.2
Rejection of Brix (%)	27.6
Reduction of Turbidity (%)	78.3
Rejection of reducing sugar (%)	0.96
Rejection of sugar (Pol) (%)	55.3
Reduction of color (%)	46.7

The process of clarification of sugar cane juice with coagulant agents resulted in a product whose rejection of turbidity and color was 78.3 and 46.7%, respectively. Comparing these values with those obtained in the microfiltration process, it is verified that the clarification through microfiltration was superior in rejecting the turbidity under the studied conditions Nogueira and Venturini Filho (2007), Hervé et al. (1995) and Kishihara et al. (1981) reported similar results.

#### Microbiological analysis

The Table 9 shows the results of the microbiological tests with sugar cane juice clarified using the two techniques investigated in this study.

**Table 9.** Microbiological analysis of the sugar cane juice clarified by microfiltration and by coagulant agent.

Microbiological Test	Conventional	Membrane
Mesophilic Bacteria (CFU $\text{mL}^{-1}$ )	$9.5 \times 10^2$	$9.8 \times 10^1$
Mold (UFC $\text{mL}^{-1}$ )	$3 \times 10^1$	$4.5 \times 10^1$
Yeast (UFC $\text{mL}^{-1}$ )	<10	$4.1 \times 10^2$
Total Coliforms (MPN $\text{mL}^{-1}$ )	<0.3	<0.3
Fecal Coliforms (MPN $\text{mL}^{-1}$ )	<0.3	<0.3

For both juices clarified by adding PAC and by microfiltration, the count of total coliforms pointed values lower than 0.03 MPN  $\text{mL}^{-1}$  of product, within the RDC standard number 12 (BRASIL, 2001), for the sugar cane juice

pasteurized, alone, or mixed, established as 10 MPN mL<sup>-1</sup> of product. The standard count (mesophilic bacteria) ranged from 102 to 104 CFU mL<sup>-1</sup> of product. The count of yeast and mold had microbial load in the order of 101 and 102 CFU mL<sup>-1</sup> of product. These values indicate that the products are not spoiled. Moreover, in Table 9, it is verified that the sugar cane juice clarified by the conventional method had better microbiological quality than that processed by membrane. However, both are within the standard established by the RDC Resolution no. 12 (BRASIL, 2001) for the sugar cane juice in natura, alone or mixed, which sets a maximum of 10<sup>2</sup> MPN mL<sup>-1</sup>.

### Conclusion

The results of the clarification tests showed that both the method using ceramic membranes and the conventional method with addition of anticoagulant agents were able to remove colloidal particles, producing a clarified juice of good acceptability.

In the comparison of the clarification methods, it was observed that the physical and chemical results are similar for both methods; the only differing parameters were the reducing sugar and pol, which had higher values in the membrane method. It was concluded that the membrane permeates more sugar than the conventional method.

### Acknowledgements

The authors express their gratitude to CAPES for financial support.

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*Received on November 30, 2009.*

*Accepted on October 26, 2010.*

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