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Accumulated flux and resistances in clarification of sugarcane juice by microfiltration – impact of operational parameters

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Abstract

Effects of operational parameters of microfiltration on permeate flux and individual resistances were evaluated. Assays were undertaken using ceramic membranes (pore size of 0.2 µm). Operational parameters – temperature and transmembrane pressure (TMP) – varied from 25.9 to 54.1°C, and from 1.8 to 3.2 bar, respectively. The main influence on time of processing was exerted by temperature (lowest values for highest temperatures, indicating a relationship to the viscosity). Resistance by irreversible fouling (R_{IF}) was the most representative, while by membrane (R_M) was the least. R_{IF} was influenced by temperature and TMP, although temperature had been the most important. Reversible fouling (R_{RF}) was also influenced by temperature and TMP, with the first presenting the highest impact. Mathematical models proposed for R_{IF} and RRF exhibits good fit to the experimental data, respectively, 95 and 93%. Results found suggest that: higher temperatures contribute for shorter periods of processing; higher TMP contribute for higher initial fluxes; the highest contribution of R_{IF} to the general total resistance occurred as a function of a considerable blockage of pores of membrane by suspended and colloidal particles; and models proposed for the resistances (R_{IF} and R_{RF}) can be applied to predict the experimental data with good accuracy.

Introduction

According to Cantarella *et al.* (2012), sugarcane is comprised by 75-82% of water and 18-25% of soluble solids, of which the greatest part is sucrose (15-24% of sugarcane juice). Authors also point sugarcane as an important source of food, feed, biofuels and bioelectricity. For Kulkarni (1996), sugar is used in a great amount in different industries: beverages, coffee, tea, confectionary.

Raw sugarcane juice is represented by a stable suspension that contains a large number of suspended particles. Soluble components include sucrose, glucose, fructose, proteins, oligosaccharides, polysaccharides, organic acids, amino acids and salts. Removal of suspended and colloidal particles and non-sucrose impurities, with minimal inversion of sucrose is of great importance, because this type of material affects juice filterability, heat transfer coefficient of evaporators, sucrose crystallization, and quality and quantity of sugar produced. Furthermore, influences on color, crystal morphology and quantity of raw sugar produced (Doherty, 2011).

The application of membrane processes, like microfiltration and ultrafiltration, in the sugar industry, aiming to obtain clarified sugarcane juice with good quality, as an alternative to the traditional clarification process (based on liming and heating), is to a great extent hindered by the occurrence of membrane fouling and concentration polarization caused by non-sucrose compounds (Gyura *et al.*, 2005). These phenomena impact directly on the membrane performance along the time, decreasing permeate flux of permeate through membrane.

Filtration processes (i.e. microfiltration) are strongly influenced by operational parameters, like transmembrane pressure, temperature, viscosity and density of fluid, and the tangential velocity. An increase in temperature, results in a decrease in fluid viscosity and increase in molecular mobility, that is in diffusivity, resulting in a higher permeate flux. Besides, an increase in transmembrane pressure contributes to a greater convective rate for the transport of the solute to the membrane surface, increasing its concentration at the surface, decreasing the permeate flow rate. Although the adjustment of process parameters to obtain high permeate flux rate is applied during microfiltration experiments, these do not guarantee constant high values, cause of phenomena like concentration polarization and fouling (Coutinho *et al*., 2009; El Rayess *et al*., 2011).

Modeling of the flux during filtration allows to obtain better identification of membrane fouling,

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providing predictive tools for successful scale up or scale down of microfiltration systems. Many empirical and theoretical models have been proposed to describe the membrane fouling phenomena. Among these models, Resistance-in-Series is highlighted as one of the simplest and most applied for juices and wine microfiltration. According to this model, flux is inversely proportional to the total resistance, being the later the sum of individual resistances - membrane, reversible (caused by cake layer deposition and concentration polarization) and irreversible (fouling) (El Rayess *et al*., 2011; Rezaei *et al*., 2014).

In literature, are found different works which have applied the model based on Resistance-in-Series to evaluate the membrane fouling phenomena during the filtration of juices from different vegetables: black mulberry (Hojjatpanah *et al*., 2011), Brazilian cherry (Ongaratto and Viotto, 2009), pineapple (Laorko *et al*., 2010), tamarind (Watanabe *et al*., 2006), umbu (Ushikubo *et al*., 2006).

This work aimed to evaluate the influence of operational parameters (temperature and transmembrane pressure) of microfiltration on the accumulated permeate flux and resistances in accordance to "Resistance-in-Series" model.

Materials and Methods

Sugarcane (*Saccharum officinarum* L.), variety SP 81-3250, obtained in the season of 2010 (in may), from a supplier of the region of Campinas (Sao Paolo State, Brazil), with appropriate conditions of cleaning and maturity, had its leaves and tips removed before be passed twice (without imbibition) through an electric laboratory mill (model B-722, MAQTRON, Brazil), which was previously sanitized with a solution of 10 ppm of free chloride. Juice obtained was called as raw sugarcane juice and was sieved in a 0.125 mm sieve, aiming to remove coarse impurities, before be put into polyethylene recipients and be stored under frozen conditions (-18 ± 2°C).

Raw sugarcane juice was unfrozen (until 25°C) and had its soluble solids content adjusted to 14 °Brix, using distilled water, before be submitted to microfiltration experiments, which were carried out during the second semester of 2010. The experiments of microfiltration were undertaken in a micro-pilot equipment (TIA, France), which was installed at the Department of Food Technology of Faculty of Food Engineering, in the State University of Campinas (Campinas, Brazil). The equipment is constituted by: feeding tank (5.0 L of volume), positive displacement motor pump, drainage and counter-pressure valves, manometers, tubular heat exchanger, digital thermometer and microfiltration modules. These modules are represented by four stainless steel cylinders, which are disposed in series, each one containing a tubular ceramic membrane.

The main characteristics of ceramic membranes are described as follows: diameter of pore of 0.2 µm; internal diameter of 7 mm; external diameter of 10 mm; length of each membrane 25 mm; permeation area of 0.005 m². Considering the four membranes installed into the equipment, the total permeation area is 0.02 m².

For each assay, approximately 5.0 L of raw sugarcane juice at 14 °Brix was placed into the feeding tank and was recirculated throughout the micro-pilot equipment, using a motor pump that had its frequency kept as the same for all experiments in 800 rpm. Membranes used for the assays were cleaned between the experiments using a clean-in-place (CIP) procedure, which was based on the utilization of warmed solutions of sodium hydroxide 0.1% (w/v) and nitric acid 0.1% (w/v) interspersed with rinsing water. Temperature and TMP values proposed in experimental design were obtained and kept using thermometer/heat exchanger, and manometers/counter-pressure valve, respectively. Juices were processed without backwashing procedure in the micro-pilot equipment.

Experiments were carried out until reach a concentration factor (ratio between mass of feeding and mass of retentate stream) (CF) of 2.0. The effective experiment started (t = 0 s) only after the conditions of temperature and TMP proposed were stabilized and 20 ml of permeate was collected and discharged. After proposed conditions were reached, permeate collected was weighted in intervals of 1 min to evaluate the decline of permeate flux along the time.

Combinations of temperature and TMP, which were the independent variables, were determined using a central composite rotational design of 2nd order for Response Surface Methodology (RSM), as proposed by Barros Neto *et al.* (2003). Combined effects of independent over dependent variables (responses) – irreversible and reversible fouling – were evaluated.

The experimental design used was a complete factorial 22 with 11 assays, being 4 factorials (combinations between levels -1 and +1), 4 axials (one variable in level + α and other in 0; and one in level - α and other in 0) and 3 centrals (the two independent variables in level 0). Temperature and TMP varied from 25.9 to 54.1°C and from 1.8 to 3.2 bar, respectively.

For determination of maximum and minimum values of each independent variable, were considered data obtained from prior tests in terms of rotation of positive displacement motor pump. Furthermore, were taken into account the manufacturer's recommended range of values for operation of membranes with respect to temperature and TMP. Experiments of microfiltration were carried out randomly.

Response surfaces were obtained using STATISTICA software, version 5.5 (StatSoft, USA). During the elaboration of the surfaces, both independent variables varied simultaneously in the region proposed by experimental design.

The TMP, which is calculated as described by Eq. (1), represents the difference between the arithmetic average values of inlet and outlet pressure in retentate stream, and pressure in permeate stream, being this last one normally considered as null, cause it is represented by atmospheric pressure (stream in contact with surrounding ambient) (Hojjatpanah *et al*., 2011; Muthukumarappan and Marella, 2012).

$$\mathbf{TMP} = \frac{P_{in} + P_{out}}{2} - P_p \quad (1)$$

Where:

TMP = transmembrane pressure (bar);
P_{in} = inlet retentate pressure (bar);
P_{out} = outlet retentate pressure (bar);
P_p = permeate pressure (bar).

Calculus of accumulated permeate flux of sugarcane juice for experiments of microfiltration was done in accordance to Eq. (2), as proposed by Muthukumarappan and Marella (2012).

$$J_a = \frac{m_p}{t \cdot A_p} \quad (2)$$

Where:

J_a = accumulated permeate flux (kg/h.m²);
m_p = mass of permeate (kg);
t = time (h);
A_p = area of permeation (m²).

The J_a represents the total mass of permeate obtained under the total time of experiment, which represents the period necessary to reach a CF of 2.0. Values determined for J_a for each one of assays proposed in experimental design were plotted in Figure 1, which relates J_a and time.

Individual resistances in microfiltration experiments, in accordance to "Resistance-in-Series" model was evaluated based on the methodology proposed by Laorko *et al.* (2010). This methodology defines that membrane filtration process can be generally described by Darcy's law, as showed in Eq. (3).

$$J = \frac{TMP}{\mu \cdot R_T} \quad (3)$$

Where:

J = permeation flux (m.s^{-1});
TMP = transmembrane pressure (Pa);
μ = viscosity of permeate (Pa.s);
R_T = total resistance (m^{-1}).

Total resistance (R_T) represents the sum, which is indicated by Eq. (4), of R_M (membrane resistance), R_{RF} (the resistance caused by reversible fouling) and R_{IF} (the resistance caused by irreversible fouling).

$$R_T = R_M + R_{RF} + R_{IF} \quad (4)$$

As described by Laorko *et al*. (2010), RRF was defined as the fouling which could be removed through water flushing, while R_{IF} would be the residual fouling (after flushing), being this type further removed by chemical cleaning. For the same authors R_T could be evaluated by the measurement of water flux during cleaning process; while R_M could be determined using the slope of cleaned water flux versus TMP and Eq. 3.

After the sugarcane microfiltration experiments, water was flushed through the membrane surface aiming to remove RRF, while permeate passages were restricted.

For water flushing, was used 30 L of cleaned water at 30°C. For this procedure, counter-pressure was kept totally open and water fed into the feeding tank was not recirculated, being discarded, as proposed by Clareto (2007). Besides, to verify the total removal of soluble solids, was collected a sample to be checked in a refractometer. For water flushing, motor pump was adjusted to work at 1,200 rpm.

In accordance to the methodology proposed by Laorko *et al.* (2010), after the first water flushing, the passages of permeate were opened, being the water flux measured to determine the residual fouling resistance (R_M + R_{IF}). A CIP procedure, was applied in the microfiltration system through the recirculation of solution of sodium hydroxide (1%) (w/v), added of sodium hypochlorite at 20 ppm of free chloride, at 60°C, for 60 min. After recirculation, the solution was discarded and the system was flushed out with 10 L of deionized water at 40°C. Afterwards, a solution of nitric acid (1%) (w/v), at 40°C, was also recirculated during 60 min, being the solution discarded after this period and the system flushed out with deionized water at 30°C. Cleaning procedure was undertaken with motor pump at 1,200 rpm. Following the cleaning procedure, water flux was measured to check the residual resistance (R_M + R_{IF}).

Table 1. Assays of the experimental design (levels in coded values and corresponding decoded or real values), times of processing and observed values for initial and final accumulated permeate flux

Assay	X_1	X_2	x_1	x_2	Time of processing (min)	J_0 (kg/h.m²)	J_F (kg/h.m²)
1	-1	-1	30.0	2.0	158	114.9	47.5
2	+1	-1	50.0	2.0	90	98.9	86.1
3	-1	+1	30.0	3.0	171	88.9	44.0
4	+1	+1	50.0	3.0	101	120.5	74.5
5	- α	0	25.9	2.5	240	61.9	32.0
6	+ α	0	54.1	2.5	83	151.5	93.9
7	0	-α	40.0	1.8	156	72.7	48.2
8	0	+α	40.0	3.2	130	119.2	58.3
9 (C)	0	0	40.0	2.5	146	80.2	51.5
10 (C)	0	0	40.0	2.5	155	80.4	48.9
11 (C)	0	0	40.0	2.5	150	74.6	50.1

X_1, x_1 = Temperature of processed juice (°C); X_2, x_2 = Transmembrane pressure (bar); (C) Central point.

All types of resistance were obtained with R_T, which was determined after sugarcane juice microfiltration, using Eq. (3) and combining the results from the cleaning procedure with Eq. (4).

Results and Discussion

Values obtained for initial and final accumulated permeate fluxes, J_0 and J_F, respectively; as well as times of processing of each one of assays, which were determined when CF = 2.0 was reached, are presented in Table 1. Curves of accumulated permeate flux for the assays proposed in the experimental design are shown in Figure 1.

According to data from Table 1, considering only values of JF for the eleven assays, was observed a variation from 32.0 to 93.9 kg/h.m² (assays 5 and 6, respectively). It was verified an important influence of temperature applied, because the lowest value of JF was observed in the experiment performed under the lowest temperature, while the highest JF was obtained for lowest temperature. This fact probably is related to the viscosity of feeding stream, which is lower for higher temperatures.

For J_0, as described for J_F, was observed that the lowest value was obtained for assay 5 (61.9 kg/h.m²) and the highest for assay 6 (151.6 kg/h.m²). This difference is also probably linked to the temperature influence. Times of processing were comprised between 83 and 240 min, determined, respectively, for assays 6 and 5. Therefore, can be suggested that higher temperatures contributed to decrease the viscosity in the feeding stream. This fact resulted in higher permeate fluxes and shorter periods of processing. It is important to emphasize that both experiments – 5 and 6 – were carried out under the same TMP.

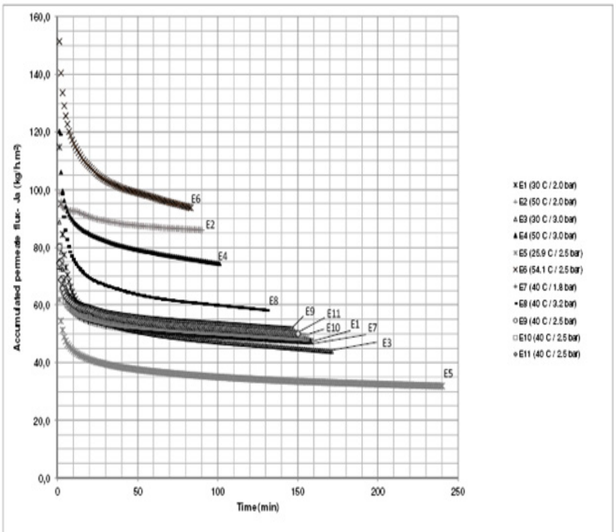

Figure 1. Accumulated permeate flux from microfiltration experiment. Letter E represents abbreviation of the word experiment

Based on values from Table 1 and curves presented in Figure 1, was observed that assays 2 and 4 also presented high J_0 and J_F, and reduced times of processing. These experiments were undertaken at 50°C, although under different TMP (2.0 and 3.0 bar, respectively). Assay 8, which was carried out at 40°C, representing the intermediate temperature of experimental design, and under the highest TMP

Table 2. Effect of conditions of processing on individual resistances

Assay (°C/bar)	R_M (m^{-1})	R_{IF} (m^{-1})	R_{RF} (m^{-1})	R_T (m^{-1})
1 (30 / 2.0)	7.43E+08 (12.2 %)	2.48E+09 (40.8%)	2.86E+09 (47.0%)	6.08E+09 (100.0%)
2 (50 / 2.0)	8.24E+08 (22.5%)	2.53E+09 (68.9%)	3.17E+08 (8.6%)	3.67E+09 (100.0%)
3 (30 / 3.0)	9.27E+08 (8.5%)	3.93E+09 (35.9%)	6.08E+09 (55.6%)	1.09E+10 (100.0%)
4 (50 / 3.0)	1.02E+09 (14.9%)	4.25E+09 (62.1%)	1.58E+09 (23.0%)	6.84E+09 (100.0%)
5 (25.9 / 2.5)	7.76E+08 (7.0%)	3.43E+09 (30.8%)	6.95E+09 (62.2%)	1.12E+10 (100.0%)
6 (54.1 / 2.5)	9.27E+08 (20.6%)	3.57E+09 (79.1%)	1.28E+07 (0.3%)	4.51E+09 (100.0%)
7 (40 / 1.8)	7.12E+08 (12.9%)	2.66E+09 (48.4%)	2.13E+09 (38.7%)	5.51E+09 (100.0%)
8 (40 / 3.2)	1.02E+09 (12.1%)	5.02E+09 (59.4%)	2.41E+09 (28.5%)	8.45E+09 (100.0%)
9 (40 / 2.5)	9.33E+08 (12.5%)	4.71E+09 (63.3%)	1.80E+09 (24.2%)	7.44E+09 (100.0%)
10 (40 / 2.5)	9.39E+08 (12.0%)	4.54E+09 (58.1%)	2.33E+09 (29.9%)	7.82E+09 (100.0%)
11 (40 / 2.5)	9.63E+08 (12.4%)	4.42E+09 (56.7%)	2.41E+09 (30.9%)	7.79E+09 (100.0%)

(3.2 bar), presented a relatively elevated value of J0 (119.2 kg/h.m²), but an intermediate, tending to low value of J_F (58.3 kg/h.m²). This behavior is in accordance to proposed by Ahmad *et al.* (2005) and Hong *et al*. (1997) that indicated that for high TMP, high initial fluxes are obtained, which fall rapidly as a consequence of solute mass transfer to membrane surface, contributing, therefore, for a fast polarization of concentration and formation of polarized gel layer, and for fouling occurrence.

Assays 1, 7, 9, 10 and 11 exhibited similar behaviors in terms of J0 and JF and times of processing. Exception for assay 1, experiments were performed under the same temperature (40°C), representing an intermediate condition. Comparing assays 3 and 4, which were carried out under the same TMP (3.0 bar) but at different temperatures (30 and 50°C), was observed that temperature was the factor that most influenced on the permeate flux, because values of J0 and J_F of assay 3 were significantly lower than those found for assay 4.

The values found for resistances: by membrane (R_M), by irreversible fouling (R_{IF}), by reversible fouling (R_{RF}) and total (R_T) are exhibited in Table 2. Furthermore, are showed the percentages of contribution of each resistance in relation to the total. According to data showed in Table 2, flux of permeate was influenced in higher extension by the resistance caused by irreversible fouling (R_{IF}), which varied from 30.8% (assay 5) to 79.1% (assay 6). Otherwise, in general, the lowest impact on total resistance was caused by membrane (R_M), that contributed between 7.0% (assay 5) and 22.5% (assay 2) in the total resistance. Moreover, resistance by reversible fouling (R_{RF}) varied widely (from 0.3 to 62.2%). As exhibited above, the main influences on total resistance were related to irreversible fouling (R_{IF}) and reversible fouling (R_{RF}). These resistances are directly influenced by operational parameters (i.e.: temperature, TMP and cross-flow velocity); while resistance by membrane depends on mainly of characteristics inherent to membrane (i.e.: diameter of pore, material, channels structure) (Rezaei *et al.*, 2014).

Flux decline during microfiltration has been attributed to the concentration polarization and membrane fouling, including deposition, pore plugging and protein adsorption in the pores of membrane. The formation of a deposit layer leads to additional resistance to permeate flow, while the pore clogging changes the effective membrane pore size and distribution. Even if these materials form only a very thin gel or cake-layer, the high fouling resistance results in an unacceptable filtration rate value. These phenomena influence the membrane performance in a way that the flux declines to a pseudo-steady state that can be one to three orders of magnitude lower than the initial value (Rezaei *et al*., 2014).

In membrane processes, an increase in pressure results in a greater convective rate for the transport of solute to the membrane surface, increasing its concentration on the interface, causing an increase in diffusivity of the solute in the opposite direction to that of the process, thus decreasing the permeate flow rate (Coutinho *et al*., 2009).

El Rayess *et al*. (2011) highlight that viscosity of fluid decreases with temperature, thus impacting on membrane filtration through its influence on permeate fluid viscosity. For authors, a rise in temperature affects positively the permeate flow. Furthermore, authors emphasize that R_M decrease linearly when temperature increases.

Considering the importance and contribution of resistances by irreversible and reversible fouling on RM, values obtained for experiments were treated using STATISTICA software, version 5.5 (StatSoft, USA), aiming to obtain coded mathematical models. Experimental values for resistance by irreversible fouling (R_{IF}) for each experiment proposed in experimental design are shown in Table 2.

The influence of independent variables on RIF can be represented by a mathematical model presented in Eq. (5), which must be used with coded values and includes only the significant regression coefficients at P≤0.10. Moreover, the model proposed was well adjusted, with $R^2 = 0.95$, especially considering such a complex process as microfiltration.

$$R_{IF} = 45.6 * 10^8 - 62.7 * 10^7 * T^2 + 81.3 * 10^7 * TMP - 45.1 * 10^7 * TMP^2 \quad (5)$$

Where:

R_{IF} = resistance by irreversible fouling (m^{-1});
T = temperature of processed juice (°C);
TMP = transmembrane pressure (bar).

The effects of microfiltration conditions on R_{IF} are exhibited in Figure 2.

According to the model represented by Eq. (5) and Figure 2, resistance by irreversible fouling (RIF) was influenced significantly by the two independent variables – temperature and TMP – cause of an increase in both resulted in higher values of resistance. However, it is emphasized that the most representative influence was occasioned by TMP, especially above 2.4 bar. Below this value, temperature exhibited a reduced influence, because an elevation in temperature resulted in minimal elevation of RIF.

The most significative influence by temperature was observed for the range between 30 and 48°C, since combined to TMP values higher than 2.4 bar. According to showed above and considering the proposed mathematical model, it is recommended to use TMP values lower than 2.4 bar, independently the temperature considered, aiming to reduce the resistance caused by R_{IF}. The experimental values obtained for resistance by reversible fouling (R_{RF}) for each assay proposed in experimental design are

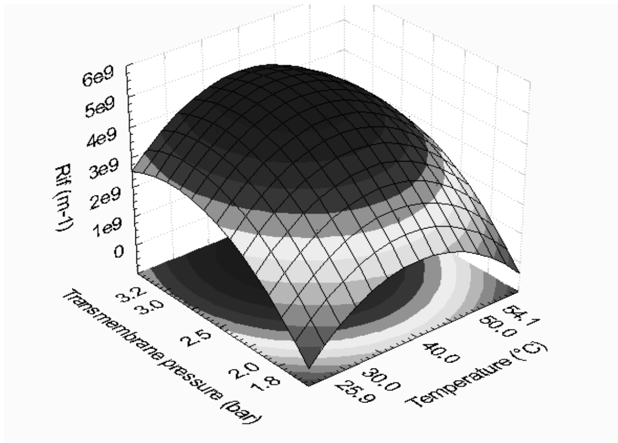

Figure 2. Response surface for resistance by irreversible fouling (RIF)

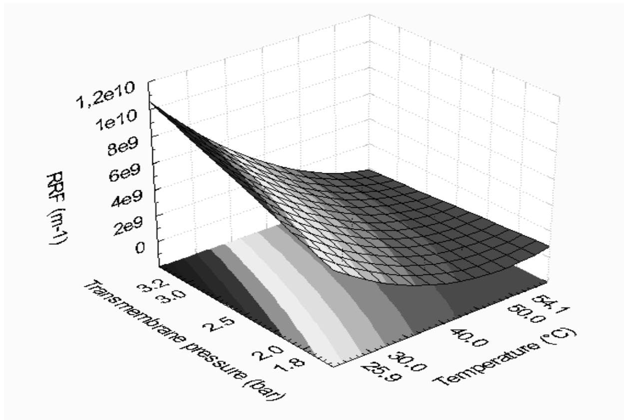

Figure 3. Response surface for resistance by reversible fouling (R_{RF})

shown in Table 2.

Equation 6 represents a coded mathematical model, which is related to the influence of independent variables on RRF. This model, which must be used with coded values, includes only the significant regression coefficients at P≤0.10. As indicated for resistance by irreversible fouling (R_{IF}), the model proposed for resistance by reversible fouling (R_{RF}) also presented a good adjustment to the experimental data ($R^2 = 0.93$).

$$R_{IF} = 45.6 * 10^8 - 62.7 * 10^7 * T^2 + 81.3 * 10^7 * TMP - 45.1 * 10^7 * TMP^2 \quad (6)$$

Where:

R_{RF} = resistance by reversible fouling (m^{-1});
T = temperature of processed juice (°C);
TMP = transmembrane pressure (bar).

In Figure 3, are showed the effects of microfiltration conditions on RRF. Based on Figure 3 and respective coded mathematical model (Eq. 6), is observed that R_{RF}, which is directly related to polarization of concentration and polarized gel layer, was significantly influenced by both independent variables – temperature and TMP – once that an increase in temperature results in lower values of RRF, while higher values of TMP contribute to higher RRF values.

Although both – temperature and TMP – had been significant, was verified that the highest influence in such resistance was related to temperature, because a variation in TMP resulted in minimal variation of values of R_{RF}, with exception for temperatures below 30°C, where an increase in pressure resulted in higher values of this resistance. According to Figure 3, is observed a tendency to obtain minimal resistance by RRF in higher temperatures and lower TMP.

Conclusions

Operational parameters of processing – temperature and TMP – did not present significant influence on physicochemical features evaluated, indicating that the size of pores of microfiltration membrane was fundamental to ease the passage of small molecules through it (i.e.: sugars, salts and ions), independently of values of operational parameters applied.

Higher temperatures contributed for shorter periods of processing, being related to lower viscosity and higher diffusivity. Furthermore, the lowest values of J_0 and J_F were obtained for the highest temperatures. Higher TMP contributed for higher initial fluxes, although fast drop of flux was observed as a consequence of solute mass transfer to membrane surface, contributing, therefore, for a fast polarization of concentration and formation of polarized gel layer, and for fouling occurrence.

The highest contribution of RIF to the general total resistance indicates that there was a considerable blockage of pores of membrane by suspended and colloidal particles. Models proposed for the resistances – R_{IF} and R_{RF} – can be applied to predict the experimental data with good accuracy, since be used with values in the range proposed in this work for temperature and TMP, using ceramic membranes.

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