# Pretreatment of Aqueous Pectin Solution by Cross-Flow Microfiltration: Study on Fouling Mechanism

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***Abstract*—This research was carried out to determine the predominant fouling mechanism during the pretreatment of an aqueous pectin solution by cross-flow microfiltration with membrane with nominal size of 0.44 μm, at different values of transmembrane pressure, temperature, and pectin concentration. To evaluate the predominant resistive mechanism was used series resistance and permeate flux model analysis. The rejection coefficient for pectin varied from 93.4 to 97.9%. The maximum flux observed was 238.69 ±6.48 kg $m^{-2}$ $h^{-1}$ at transmembrane pressure of 0.12 MPa, temperature of 50 ℃ and initial pectin of 1.0 g $kg^{-1}$. The dominant restrictive mechanism observed was the cake layer formation, for all assay evaluated.**

***Index Terms*—Pectin, microfiltration, permeate flux, fouling.**

## I. Introduction

Pectin is a family of complex polysaccharides constituted of galacturonic acid units linked by $\alpha(1\rightarrow4)$ glycoside bonds. It also presents esterified regions with methylic groups and branches constituted of neutral sugars. The structure of the lateral chain and the degree of esterification characterize the capacity of gelling, solubilization, and aggregation of pectin in solutions. Due to its gelling characteristic, pectin is a very important raw material in the jelly, sweet, and preserve industries [1]-[3]. In the production of pectin, the most costly step is the purification process, which requires large amounts of ethanol to precipitate pectin from the extraction solution. The ethanol solution is later evaporated by vacuum [4], [5].

The porous membrane separation process has been widely used in the food industry, especially in the clarification of juices. The traditional macromolecular fractioning processes that are frequently used in the industry can be optimized with the implementation of membrane separation steps [6]-[8]. According with Moresi [9], the implementation of a microfiltration step before the precipitation with ethanol results in the successful concentration of the extraction solution, reducing the volume of ethanol required to precipitate pectin, and consequently, reducing the energy spent in the evaporation step. Cho *et al.* [10] incorporated a system of cross-flow microfiltration with a 0.2-μm cellulose membrane to a purification step in the extraction of pectin. With the microfiltration system, was achieved a reduction of 75% of the volume of ethanol required.

However, the reduction in the permeate flow is a restriction of membrane processes. Pectin is undesirable in the fruit and vegetable juice clarification process, as the typical concentrations of 1 % mass precipitate on the membrane surface as a viscous gel, increasing the resistance to permeation [11]-[13]. The polarization layer is the concentration boundary layer adjacent to the membrane surface, formed in the beginning of process, resulting in a sharp flux decrease, which is stabilized by the renewal surface effect promoted by the crossflow flux [14]. However, a gradual decrease in flux is also observed due to physical and chemical interactions of pectin with the membrane, known as fouling. Fouling limits the flux by continuous blocking of membrane, thus gradually reducing the permeate flux [15].

Fouling is an intrinsic problem associated to all kind of membrane process and the extent to which fouling can be controlled is proportional to the understanding of the mechanisms that govern this process. The identification of these phenomena associated with the operating conditions allows to assessment the technical viability of process. This study evaluated the effect of operational parameters in the permeate flux and fouling resistance and estimated the major blocking mechanism present in the concentration of pectin solutions.

## II. Materials and Methods

### A. Experimental Procedure

This study was performed in a crossflow microfiltration pilot unit used in previous studies [16], [17]. This system was made with stainless steel for use an alumina ceramic membrane (Fairey Ceramics Inc™, London, England) with a nominal pore size of 0.44 μm, surface area of 0.06 m², length of 60 cm and external diameter of 2.0 cm. The feed flow adopted was 1.0 $m^3$ $h^{-1}$ and the temperature of the solution was adjusted through a serpentine immersed in a heating bath (Quimis, model Q215, São Paulo, Brazil). The pectin solutions were prepared by slowly dispersion of commercial pectin (CP Kelco, Limeira, Brazil) in deionized water with mixing (Q-250M at 500 rpm, QUIMIS™). The permeate flux was determined by gravimetry by (1).

$$J_P = \frac{m_{PER}}{t \cdot S} \quad (1)$$

where $J_P$ is the permeate flux (kg $m^{-2}$ $h^{-1}$), $m_{PER}$ is the permeate mass (kg), $t$ is the filtration time (h), and $S$ is the membrane surface area ($m^2$).

### B. Experimental Design

The experiments were performed with factorial design $2^3$ evaluating: temperature (30 ℃ and 50 ℃), transmembrane

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pressure (0.04 MPa and 0.12 MPa) and initial pectin concentration (1.0 g/L and 2.0 g/L) with central point (40 ℃, 0.08 MPa and 1.5 g/L). The initial solution quantity used in each experiment was 6 kg with a fixed time of 1 hour. All experiments were made in triplicate. Analysis of variance was performed for the treatments with Tukey's test to compare means with 5.0 % of significance level.

The system was operated with feed flux of 1.0 m $^3$h$^{-1}$ and mean surface velocity of 3.82 m s$^{-1}$. As a result, the Reynolds number varied between 5,400 and 12,000 for the experiments, ensuring a turbulence condition in all tests.

The cleaning process was made after each replicate of assays, started with the washing with deionized water at boiling point. After the cleaning system, the membrane was removed of the system and immersed in ultrasonic bath (Ultrasonic cleaner USC 1400 – UNIQUE$^{TM}$) with successive cycles of 10 minutes of cleaning with solutions of NaOH (1.0 % m/V), deionized water and Citric Acid (0.8%). All chemical products used were analytical standard.

### C. Sample Analysis

Dynamic viscosities of the pectin solutions were measured in a capillary viscometer (Schott TM CT 52). Laser granulometry (1064 CILAS liquid) and turbidity (DLM DEL 2000B LAB) analyses were performed to characterize the distribution of pectin aggregates in the dispersed portion of solution.

The pectin concentration was determined by spectrophotometry (Pro-analise series 1000 UV/VIS spectrophotometer, São Paulo, Brazil) at 300 nm using deionized water as blank [18]. The pectin rejection coefficient was calculated with (2) [19].

$$CR = \left(1 - \frac{C_P}{C_F}\right) \cdot 100 \tag{2}$$

where $CR$ is the rejection coefficient (%), $C_P$ is the pectin concentration in the permeate flow (g kg$^{-1}$), and $C_F$ is the feed concentration (g kg$^{-1}$).

### D. Fouling Resistance

The fouling resistance effects were estimated using the fouling resistance model [19], [20]. This model describes the effects that may interfere with the permeate flux as a function of the total resistance $R_T$. The permeate flux can be described as (3):

$$J_P = \frac{\Delta P}{\mu \cdot R_T} \tag{3}$$

where $\Delta P$ is the transmembrane pressure, $\mu$ is the permeate viscosity.

Fouling resistance $R_F$ is obtained by difference between the total resistance $R_T$ of pectin solution permeation and the hydraulic membrane resistance (4):

$$R_F = R_T - R_M \tag{4}$$

The hydraulic resistance membrane $R_M$ was measured by (5) with deionized water.

$$R_M = \frac{\Delta P}{\mu_W \cdot J_W} \tag{5}$$

where $J_W'$ is the water permeate flux through the cleaning membrane and $\mu_W$ is the water viscosity.

### E. Mathematical Models of the Permeate Flux

The pore blocking models depend on the interaction between the membrane and the solute. In this study, the pore blocking models and the classical filtration theory at constant pressure were used. The classical filtration model adopted [12], [13], [20], expressed as permeate flux, follows (6):

$$\frac{1}{J_P(t)} = R_{ME} \cdot \frac{\mu}{\Delta P} + m_{PER}(t) \cdot \frac{\mu \cdot C_F \cdot \alpha}{2 \cdot S \cdot \Delta P} \tag{6}$$

where $R_{ME}$ represents the total resistance of the membrane, $m_{PER}(t)$ is the mass of permeate accumulated up to instant $t$ and $\alpha$ is the resistance of the surface cake.

The pore blocking models is an empirical model proposed by Hermia [21], and adapted for cross-flow filtration system [22]-[24]. The general pore blocking model can be described by (7):

$$\frac{dJ_P}{dt} = -k_n \cdot \left(J_P - J^*\right) \cdot J_P{}^{2-n} \tag{7}$$

where $k_N$ and $n$ represent the phenomenological coefficient and the general fouling index, respectively, and $J^*$ is the ideal critical flux for which fouling does not occur. The integration of (7) gave the distinct pore-blocking mechanisms as a function of the index $n$.

For $n$ = 2.0, the complete pore blocking model is defined in (8). In this model, the particles are bigger than the pore opening, a part of the membrane surface covered by the particles is sealed, and permeation is prevented by the reduction of the surface area.

$$J_p(t) = J_{LIM} + \left(J_O - J_{LIM}\right) \cdot \exp\left[-k_{2.0} \cdot t\right] \tag{8}$$

where $Jp(t)$ is the permeate flux value at time $t$, $Jo$ is the permeate flux value at the initial time and $J_{LIM}$ is the steady-state value of permeate flux.

The value $n$ = 1.5 gives the internal pore blocking model. This model, (9), considers that the particle size is smaller than the pore opening and that the particles tend to be retained inside the pores due to adsorption on the pore walls or by deposition on the internal cavities. As the blocking occurs internally, the reduction of the pore opening by blocking becomes independent of the flux conditions, that is, the flux decays to a null value.

$$J_P(t) = \frac{Jo}{\left[Jo + Jo^{0.5} \cdot k_{1.5} \cdot t\right]^2} \tag{9}$$

The value $n$ = 1.0 gives the partial pore blocking model, (10). In this case, the particle size is close to the pore size and they tend to agglutinate in specific regions of the pore, without closing it.

$$J_P(t) = \frac{Jo}{J_{LIM} - Jo \cdot (\exp[k_{1.0} \cdot J_{LIM} \cdot t] - 1)} \quad (10)$$

For $n$ = 0, the resulting model corresponds to the cake filtration, (12). Considering the effect of larger particles on the pores and that they agglomerate on the membrane surface forming a filter cake, they constitute an additional resistance to the process.

$$k_0 \cdot t = \frac{1}{J_{LIM}^{\ 2}} \cdot \ln\left[\frac{J_P(t)}{Jo} \cdot \frac{J_O - J_{LIM}}{J_P(t) - J_{LIM}} - J_{LIM} \cdot \left(\frac{1}{J_P(t)} - \frac{1}{J_O}\right)\right] \quad (12)$$

The models were submitted to non-linear regression by optimization by Simplex algorithm. The sum of the squares residues *SSR* and coefficient of determination $R^2$ were determined as the evaluation criteria of the models.

The mean experimental values were adopted for parameters $J_{LIM}$ and *Jo*, adjusting only the $k_n$ parameter.

## III. Results and Discussions

### A. Fouling Resistance

A high retention of pectin was observed in all assays, as showed in Table I. The rejection coefficient was within 93.4 ±0.7 % and 97.8 ±0.5 %. Much of the pectin in solution was found dispersed in the solution, with average turbidity values between 21.62 and 41.89 NTU, and this conditions, pectin take the form of aggregates groups with size between 500 mm and 0.01 μm, as Fig. 1, formed from Van der Waals bonds and hydrogen bridges between available hydroxyl. Due these aggregates, the cross-flow microfiltration showed high values of pectin rejection. Lower values were observed at the highest experimental temperature (50 ℃), where the solubility of pectin is increased and the solution viscosity is reduced, favoring the mass transfer and the permeation, especially of the neutral sugar and bound residues in pectin.

In previous works [16], [17] the authors observed that the permeate flux has strong dependence of the temperature, pectin concentration and transmembrane pressure. Table I showed the values of the permeate flux at the final of operation and fouling resistance. The hydraulic membrane showed means between 3.49 ± 0.28 and 3.91 ± 0.28, statistically equal for all assays, what indicated the efficiency of the cleaning procedure proposed.

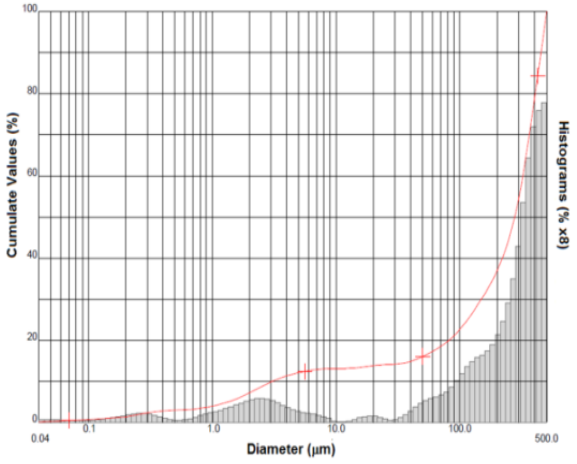


Fig. 1. Granulometric distribution of dispersed pectin.

Temperature is favorable for the viscosity and diffusion coefficient pectin, thus high temperature is favorable for water permeation; while the initial pectin concentration interferes on the resistance mechanisms, particularly with the respect to the polarization layer and gelling surface layer. The transmembrane pressure is the driving force, so its increase is favorable to the permeation until the limit, that the equilibrium between the surface renewal and resistive mechanisms.

This behavior was similar that observed by Wang [20], and can be explained by solubilization of pectin, reducing it to particle aggregates formation and its surface deposition.

TABLE I: Analyses of Steady-State Permeate Flux, Rejection Coefficient and Fouling Resistance

| Assays* | CR (%) | *Jp* (kg/m ħ) | $R_F \times 10^{-8}$ (m ⁇kg) |
|---|---|---|---|
| 0.04/30/1.0 | 97.6 ±0.2$^{AB}$ | 143.64 ±10.06$^{cd}$ | 7.01 ±0.78C |
| 0.12/30/1.0 | 97.9 ±0.5$^{A}$ | 185.42 ±6.15$^{bc}$ | 22.22 ±3.12B |
| 0.04/50/1.0 | 94.5 ±1.2$^{CD}$ | 214.40 ±8.15$^{ab}$ | 5.74 ±1.17C |
| 0.12/50/1.0 | 95.3 ±0.8$^{BCD}$ | 238.69 ±6.48$^{a}$ | 20.03 ±0.42B |
| 0.04/30/2.0 | 97.7 ±0.8$^{AB}$ | 112.30 ±4.51$^{d}$ | 9.34 ±1.13C |
| 0.12/30/2.0 | 97.9 ±0.3$^{A}$ | 122.95 ±5.77$^{d}$ | 30.57 ±4.36A |
| 0.04/50/2.0 | 93.8 ±0.9$^{D}$ | 148.30 ±3.26$^{cd}$ | 7.35 ±1.15C |
| 0.12/50/1.0 | 93.4 ±0.7$^{D}$ | 175.33 ±10.55$^{bc}$ | 23.73 ±3.74B |
| 0.08/40/1.5 | 96.4 ±0.6$^{ABC}$ | 164.85 ±13.70$^{c}$ | 19.73 ±1.61B |

* The same letters in each column represent statistically equal values according to Tukey's test at the 5% significance level.
* Experimental conditions: Transmembrane pressure (MPa) / temperature (℃) / concentration (g $kg^{-1}$).

According to Sulaiman [18], the linear and branched structure of the pectin molecule allows for mobility and deformation of the chain under the action of an external force, favoring its insertion into the membrane pores. As higher tension on the molecules greater force necessary to pass them, increasing obstruction of the pores and resistance to fouling. Additionally, the increase in transmembrane pressure has a compacting effect on the gel layer on the pectin surface, raising the filtering cake resistance.

The increase in the concentration leads to an increase in the fouling resistance, which was greater for the assays with the highest transmembrane pressure (0.12 MPa). The fouling resistance found in assay 2 (0.12 MPa, 30 ℃ and 1.0 g $kg^{-1}$) was (20.03 ±0.42) × $1.0^8$ $m^2$ $kg^{-1}$, while that found in assay 6 (0.12 MPa, 30 ℃ and 2.0 g $kg^{-1}$) had a value of (30.57 ±4.36) x $1.0^8$ $m^2$ $kg^{-1}$. The larger amount of pectin in the system leads to an accumulation of larger mass on the surface, favoring an increase in the filtration cake, which in this case was intensified by the compaction effect exerted by the pressure of 0.12 MPa.

### B. Behavior of Permeate Flux

The behavior of the permeate flux initially had a sharp decay, followed by leveling. Fig. 2(a) and Fig. 2(b) display the permeate flux values observed in 1 h assays in continuous mode at 1.0 g $kg^{-1}$ and 2.0 g $kg^{-1}$, respectively. Assay 4 (0.12 MPa, 50 ℃ and 1.0 g $kg^{-1}$) gave the highest flux after 1 h (238.69 ±6.48 kg $m^{-2}$ $h^{-1}$).

TABLE II: PERMEATE FLUX DATA MODELLING

| Models* | Parameters | 1 | 2 | 3 | 4 | 5 | 6 | 7 | 8 | 9 |
|---|---|---|---|---|---|---|---|---|---|---|
| Complete Blocking (n = 2.0) | $k_{2.0}$ | 9.06 | 9.60 | 53.28 | 7.86 | 12.96 | 4.74 | 9.00 | 11.52 | 8.76 |
| | $R^2$ (%) | 93.5 | 87.9 | 79.5 | 95.2 | 91.7 | 89.4 | 94.2 | 89.4 | 93.5 |
| | SSR | 6550 | 14567 | 33745 | 12929 | 3207 | 14972 | 7449 | 12522 | 8105 |
| Internal blocking (n = 1.5) | $k_{1.5}$ x $10^2$ | 2.99 | 3.42 | 4.62 | 1.93 | 2.95 | 2.60 | 2.84 | 3.10 | 2.80 |
| | $R^2$ (%) | 80.6 | 89.6 | 77.4 | 79.5 | 73.6 | 82.2 | 70.7 | 76.4 | 81.2 |
| | SSR | 38736 | 94295 | 261222 | 90679 | 23563 | 46033 | 61573 | 69802 | 47170 |
| Partial Blocking (n = 1) | $k_{1.0}$ x $10^2$ | 4.46 | 3.62 | 9.24 | 2.42 | 8.10 | 2.78 | 4.30 | 4.91 | 3.81 |
| | $R^2$ (%) | 95.0 | 90.4 | 84.7 | 96.4 | 93.4 | 90.6 | 95.5 | 91.4 | 95.0 |
| | SSR | 4876 | 11378 | 104524 | 9521 | 2562 | 11381 | 5509 | 9966 | 6062 |
| Cake filtration (n = 0) | $k_0$ x $10^4$ | 1.56 | 1.06 | 2.21 | 0.65 | 4.34 | 1.70 | 2.24 | 1.30 | 1.51 |
| | $R^2$ (%) | 96.4 | 94.3 | 96.3 | 97.4 | 95.1 | 91.6 | 96.9 | 93.0 | 96.4 |
| | SSR | 4624 | 6251 | 8285 | 7364 | 2027 | 8132 | 3359 | 11417 | 4192 |
| Classic filtration model | x $10^5$ | 0.252 | 0.456 | 8.40 | 0.354 | 6.31 | 0.329 | 0.130 | 0.570 | 0.245 |
| | $R_{ME}$ x $10^{-9}$ | 0.781 | 1.77 | 0.725 | 1.70 | 0.976 | 2.94 | 0.809 | 1.74 | 1.46 |
| | $R^2$ (%) | 80.5 | 89.0 | 66.6 | 81.3 | 70.0 | 82.3 | 70.2 | 80.6 | 80.7 |
| | SSR | 5802 | 5970 | 39096 | 15775 | 10336 | 57532 | 16828 | 40385 | 7273 |

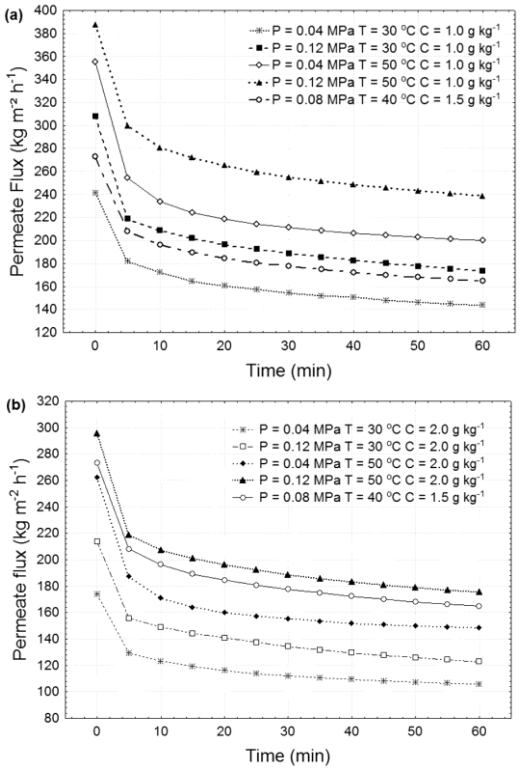


Fig. 2. Permeate flux behavior: (a) C = 1.0 g $kg^{-1}$, (b) C = 2.0 g $kg^{-1}$.

To identify the mechanism of fouling during pectin microfiltration, it was adopted the comparative study between experimental values of permeate flux and different mathematical models for restrictive flux, like other studies [23], [24]. The coefficient of determination ($R^2$) and the sum of the square of residuals (SSR) between numerical predictions and experimental data were the criterion used to choice the best fit model for each assay evaluated.

For all assays the cake filtration model ($n$ = 0) derived of the pore blocking model showed the best fitting. The cake filtration model had SSR values between 2,020 and 11,500 and the best $R^2$ observed, as showed in Table II, while the other models had high values of SSR between 2,500 and 262,000 and, therefore, they do not describe the pectin-membrane interaction phenomena. For all models considered in this study, independent of the operational conditions, the precision in the fitted results at the beginning is poor due these models evaluated only the fouling aspects, and the initial of the process the resistive mechanisms are not fully formed, specially the dynamic resistances, like the polarized layer.

The classical filtration model has significant deviations (see Table II) due its restrictions. This model does not estimate the initial flux value and does not take into account the effect of surface renewal promoted by the cross-flow flux. Fig. 3 presents the experimental flux values and the values estimated by all models used for the maximum flux observed in total recycling mode (0.12 MPa, 50 ℃ and 1.0 g $kg^{-1}$).

Assays in the total recycling mode, or in process with lower values of concentrate factor, the blocking model can be successful to describe the fouling phenomena, despite the deviations observed in the initial region due the limitation of the pore blockage models in estimating the initial decay (Fig. 3). However, they predict the behavior in the region where the fouling effects predominate. In contrast, the classical filtration model is not able to describe the balance relationship between the renewal effect and the interactions due the surface accumulation and the polarization layer, which justifies the high deviations in the initial and final moments of the process.

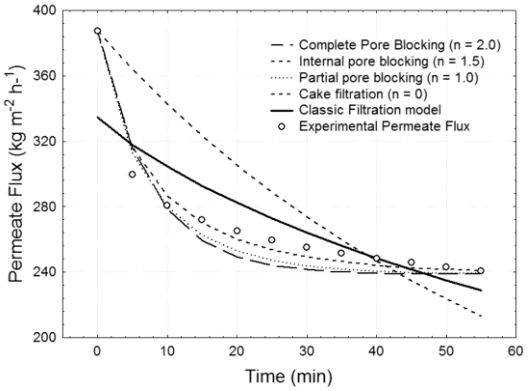


Fig. 3. Fitting of the pore blockage and the classical filtration models (C = 1.0 g $kg^{-1}$, T = 50 ℃ and P = 0.12 MPa).

Based on the fitting described for these models, was observed that the fouling profile suggests a surface

interaction between the pectin and the pore caused by the pore opening sealing due the deposition of pectin on surface in the form of gel, as proposed by Rai [25]. This restrictive phenomenon was expected, because pectin is a long macromolecule and also because of characteristics interactions between the molecules, which results gelling properties to pectin in the presence of sugars. The formation of gel on the surface is presumed, due the high pectin aggregation capacity (see Fig. 1), which allows the retention of pectin in the membranes of the microfiltration process.

Barros [23] observed the predominant blocking mechanism by cake filtration in ultrafiltration of pineapple juice (with large amount of pectin, between 1% at 3 %), especially after the first minutes of operation, due the high interaction between the colloidal materials and membrane surface. According with Jiraratananon [26], glycoside molecules have the tendency to form aggregates with other macromolecules by hydrogen bounding, and form cake on the surface. In this case, the convective flux, direct form the bulk solution toward the membrane, prevails on the rate of back diffusion of colloidal materials reject by surface membrane.

## IV. Conclusion

This study evidenced the potential application of crossflow microfiltration processes to the treatment of pectin with rejection coefficients of up to 97.8%.

The highest steady-state permeate flux obtained was 238.69 kg $m^{-2}$ $h^{-1}$ at 0.12 MPa, 50 ℃ and 1.0 g $L^{-1}$, while the greatest fouling resistance observed was the order of 30.57 × $10^8$ $m^2$ $kg^{-1}$ for experimental conditions of 0.12 MPa, 30 ℃ and 2.0 g $kg^{-1}$.

From the analysis of the blocking models, we conclude that the dominant restriction mechanisms are the surface effects, notably the filtration cake due the formation of gel layer on the membrane surface.

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