

Influence of the pectin and cellulose on the performance of cross-flow microfiltration

Influencia de la pectina y celulosa en el desempeño de la microfiltración de flujo tangencial

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ABSTRACT: The cross-flow microfiltration (CFM) of fruit juices allows obtaining products of high quality by conserving their organoleptic characteristics and microbiological stability during storage. The effect of the main macro-compounds and the transmembrane pressure (TMP) on the process performance with model fluids was evaluated. The model fluids concentration varied between 0.25 and 0.75% for pectin and 0.04 and 0.08% for cellulose. To study the influence of transmembrane pressure (TMP) and concentration on the hydrodynamic properties of the fouling layer (K, C_g), diffusivity (D) and the boundary layer thickness (δ_b) the Box-Behnken design with three replicas in the center was used. It was found that the CFM process is efficient and commercially feasible when working at a constant TMP of 1.93 bar and at concentrations of pectin and cellulose of 0.25% and 0.0513%, respectively.

RESUMEN: La microfiltración de flujo tangencial (MFT) de jugos de frutas permite obtener productos de buena calidad al conservar sus características organolépticas y estabilidad microbiológica durante el almacenamiento. En este trabajo se evaluó el efecto de los principales macro-compuestos y de la presión transmembrana (PTM) sobre el desempeño del proceso con fluidos modelo. Las concentraciones de los fluidos modelo, variaron entre 0,25 y 0,75% para pectina y 0,04 y 0,08% para celulosa. Se utilizó el diseño Box-Behnken con tres réplicas en el centro para estudiar la influencia de la presión transmembrana (PTM) y concentración sobre las propiedades hidrodinámicas de la capa de colmatación (K, C_g), difusividad (D) y espesor de la capa límite (δ_b). Se encontró que el proceso de MFT es eficiente y comercialmente factible cuando se trabaja a una PTM constante de 1,93 bar y a concentraciones de pectina y celulosa de 0,25% y 0,0513% respectivamente.

1. Introduction

In 2019, Colombia was consolidated as the main exporter of tropical fruits in Latin America and the sixth in the world [1]. According to the Fontagro report in 2020, it is estimated that 30% of all fruits and vegetables grown in the world are

sold as processed products. Exports of processed fruits and vegetables have increased by 8% from 2015 to 2019, with a value of 63.667 million dollars in the last year, where 42.141 million dollars correspond to processed fruits of which 36% correspond to the industry of the juices [2, 3].

Tropical fruits can present large post-harvest losses as total loss or decrease in their organoleptic quality caused by mechanical, physiological, or by pest and

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disease damage; this is due to the lack of appropriate storage and conditioning systems and deficiency of accessible technological offers to allow growers to offer competitive and sustainable alternatives that will help them to increase their commercialization. Therefore, membrane technology is presented as an implementation alternative to reduce losses and produce additive-free and high-quality fruit extract with a natural fresh taste [2], [4-8].

Membrane filtration can be a continuous and automatic process, which results in savings in labor and energy input, since a phase change does not occur. Also, membranes can retain all undesirable microorganisms on the membrane surface, making the fruit juice totally stable microbiologically after membrane filtration and a high-quality final product [4].

Tropical fruit juices contain bioactive compounds and large amounts of polysaccharides such as pectin, cellulose, hemicellulose, lignin, and tannins, which in most cases, block the pores of the membranes while reducing the permeate flow during filtration processes. Permeate flow reduction limits the scaling of the cross-flow microfiltration (CFM) at the industrial level in the fruit juice industry; consequently, the process can become a bit unfeasible technically [4].

Studies with membrane technologies have been conducted in small equipment with total recycling to reach a steady process state rapidly. The industrial-scale filtration of fruit juice should be performed in concentration mode, with continuous feeding and constant collection of the permeate without retentate [5]. In membrane technologies, the supplied transmembrane pressure allows the rejection of solids dissolved in the juices, which are rejected and removed from the filtration system [6].

Membrane technology (MT) is a method of filtration of solutes dissolved in a fluid, whose principle is the separation by the difference of the weights molecular and particle size [9-11]. Membrane processes are classified according to the type of compound or microorganism to retain in: inverse osmosis (RO), which prevents the passage of bacteria, protozoa, algae, viruses, dissolved organic matter, and divalent and monovalent ions. RO works on size exclusion and solution diffusion through a semipermeable membrane. Unlike RO membranes, nanofiltration (NF), with a pore size of approximately 0.001 μm , does not retain monovalent species such as sodium and chlorine. Ultrafiltration (UF) with a pore size (in the range 2 - 100 nm) and the molecular weight of the separating compounds (1000 - 100000 Da), which is an energy-intensive process, works at low transmembrane pressures for the removal of dissolved and colloidal

materials [12].

During the intermediate process between UF and RO, the molecular weights of the separating compounds range between 200 Da and 1000 Da with pore diameters varying from 1 nm to 2 nm-; -MF (microfiltration) with a pore size of 0.1 μm , which differs from the membranes previously described, only allow the removal of bacteria and suspended solids. MF separates particles with an intermediate size between 0.05 and 10 μm , at sensitive pressures between 0.5 and 3 bars. Generally used to retain solids in suspension, it completely eliminates bacteria and spores (size: $\sim 0.4\text{a}2\mu\text{m}$) [6, 12, 13].

Membrane processes have been consolidated in various productive sectors due to their selectivity, the ability to operate at room temperature, low energy consumption, and the ability to retain nutritional compounds [6, 9]. However, during cross-flow microfiltration, the membranes tend to foul by the presence of macro-compounds such as pectin, cellulose, lignin, and hemicellulose that cause the fouling of the membranes and, therefore, decrease process performance [5, 9, 13]. In order to determine the process performance, and increase separation and selectivity yields, it is necessary to study the influence of main macro-compounds contained in tropical fruit juices (pectin and cellulose) on CFM performance, which will allow to control and improve its application at an industrial level.

2. Materials and Methods

The following materials were used for preparing the model fluids: citric pectin of esterification degree of 58%, type 121 of slow fastening at low temperatures (GENU®, CP Kelco product, file No. 0001064-01). This pectin has high contents of esters extracted from citrus peels and is standardized by the addition of sucrose. The texture was a free-flowing powder texture, with a particle size of 0.2011 mm and pH (1% s/n) 2.9-3.6. Insoluble Cellulose with a white fibrous powder appearance of purity greater than 97%, pH between 5.0 and 7.5 (10% s/n), and a water retention capacity of 1:5 (Tecnas, PT-823).

2.1 Preparation of the model solutions

Model solutions of pectin and cellulose were prepared based on the content found in tropical fruit juice reported in the literature [9, 10] [13-23]. Different mixtures of these solutions were used, varying the concentration for pectin between 0.25 and 1.5% (%w/w) and for cellulose between 0.04 and 0.15%. Pectin was first mixed in the dry form with sugar, which was solubilized in deionized water at 30 °C; after ensuring the complete dissolution of the pectin, cellulose was added with constant stirring following

the procedure for the adjustment of soluble solids and $pH(40.2)$ with sodium bicarbonate or citric acid as the case may be. The content of soluble solids was adjusted to 10 ± 0.5 °Brix with a refractometer.

2.2 Physicochemical parameters evaluated

The physicochemical parameters were evaluated in the feed, permeating retentate streams to obtain the values of pH, density, viscosity, and soluble solids, according to classical procedures [24]. An Abbe refractometer (Atago® model 1T, Japan, measurement accuracy of brix of 0.5%) was used to determine the soluble solids. The pH of the samples was determined with a previously calibrated Schott Geräte model CG 820 pHmeter.

Turbidity (TU) was measured in nephelometric turbidity units (NTU), using a turbidimeter (model 2100AN, Hach Company, Loveland, CO, USA). The turbidimeter was calibrated for every measurement series, using different formazine standard solutions, from 0 to 7500 NTU. All turbidity measurements were performed in triplicate, using 25 ml sample cells that were stoppered and gently inverted twice to ensure even mixing. A reading was taken exactly 8 seconds afterwards. The final reading was the average of the measurements recorded by the turbidimeter.

The viscosity (μ) of the samples was determined at 251 °C using a Cannon-Fenske® viscometer and at 25 °C with a Brookfield LV DV-I Prime® viscometer. A spindle R.00 at 50 rpm with torque at 10 N·m, was used for the feed and retentate. The content of suspended insoluble solids (SIS) in the feed stream was determined by centrifuging 25 ml of a sample previously homogenized at 8000 x g for 20 min; the results were expressed in g/l. The samples were analyzed in triplicate.

2.3 Microfiltration

The partially automated CFM equipment (TIA. Applied Industrial Techniques) at the Universidad del Valle (Cali - Colombia), consisted of two multichannel profile tubular ceramic membranes (Membralox®, model 1P19-40, Pall-Exekia, Bazet, France) with a $0.48m^2$ total effective filtration area and a mean pore diameter of $0.2\mu m$, two pumps (one of supply and one of circulation) and a tubular heat exchanger regulated with a proportional controller (PI) featuring an ON/OFF combination and a pulse width modulator (Figure 1).

The outlet exchanger temperature was measured with an electrical sensor RTD PT-100 (class B), with a temperature range from 0 °C to 100 °C. In order to measure the input and output pressures of the filtration

module, two pressure transmitters (PT2 and PT1) of piezo - resistive type from 0.5 bar to 10 bar and a proportional valve VP1 with Proportional Integral Derivative Controller (PID) were used. The communication signals coming from pressure and flow transmitters and sent to the proportional valve (VP1), were in the standard (4 to 20) mA. The filtration unit has a 50 L feed tank. The experimental unit varied between 40 and 80 liters of solution per run, depending on the concentration used.

The start-up process was set according to the procedure suggested by Dornier *et al.* [25]; the permeate valve was opened 30 minutes after adjusting the velocity to 5 m·s⁻¹ and the transmembrane pressure (TMP) between 1.8 and 3.3 bar. The CFM of each model fluid was analyzed by continuous feeding and constant collection of permeate without removal of the retentate (concentration mode). Permeate fluxes (J_p) were measured every minute using graduated containers of 2-L and the equipment's flow meter. All trials were conducted for approximately 2 hours. The cleaning procedure was carried out as described by Gallego-Ocampo H-L [17]. The volumetric concentration ratio (VCR) was calculated for the area (A), as according to Equation (1)

$$VCR_t = \frac{V_d + \int_0^t J_p A dt}{V_d} = \frac{V_d}{V_d - V_p} \quad (1)$$

where V_d is the dead volume of the circuit in (L), J_p is permeate flux ($L \cdot h^{-1} \cdot m^{-2}$), A is filtration area (m^2), V_p is the permeate volume in (L).

The hydrodynamic characteristics of the fouling layer (K, C_g, D, δ_b) were determined from the adjustment of the experimental data to the mathematical models described in the gel polarization model, Equation (2); mechanistic model, Equation (3), and thickness of gel layer, Equation (4), using the software SigmaPlot v.10.01

$$J_p = K \cdot \ln \left(\frac{C_g}{VCR \cdot C_0} \right) \quad (2)$$

where K is the global mass transfer coefficient (m/h), $VCR \cdot C_0$ is the volumetric concentration of particles in the bulk (retentate), C_g is the volumetric concentration of particles in the gel or cake layer (% w/w), determined by extrapolation of the regression curve with the intercept of the x-axis when $J = 0$, C_0 is the volumetric concentration of particles in the initial feed juice (% w/w) (Figure 2). Representing K as $K = D/\delta$, where D is the diffusion coefficient, δ is the thickness of concentration boundary layer in (m), l is the filter channel length, γ shear stress (s^{-1}) estimated as $\gamma = 2v/d = 2180 s^{-1}$ where v is the average cross-flow velocity, the Equation (2) would be like:

$$J = \left[\left(\frac{3}{2} \right)^2 \left(\frac{D^2 \gamma}{l} \right) \left(\frac{C_g}{VCR \cdot C_0} - 1 \right) \right]^{\frac{1}{3}} \quad (3)$$

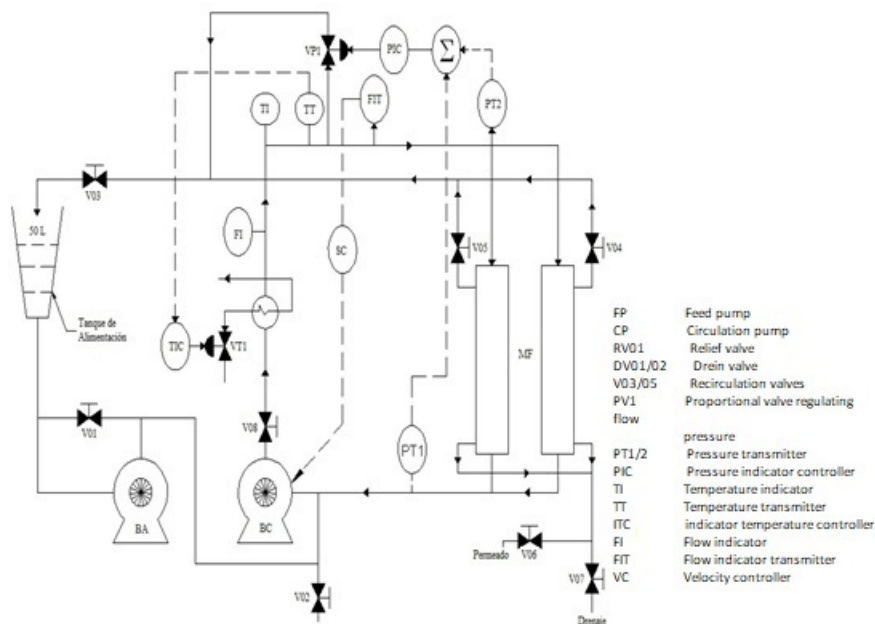


Figure 1 Schematic view of a cross-flow microfiltration equipment (CFM) partially automated at Universidad del Valle (Cali – Colombia) [17]

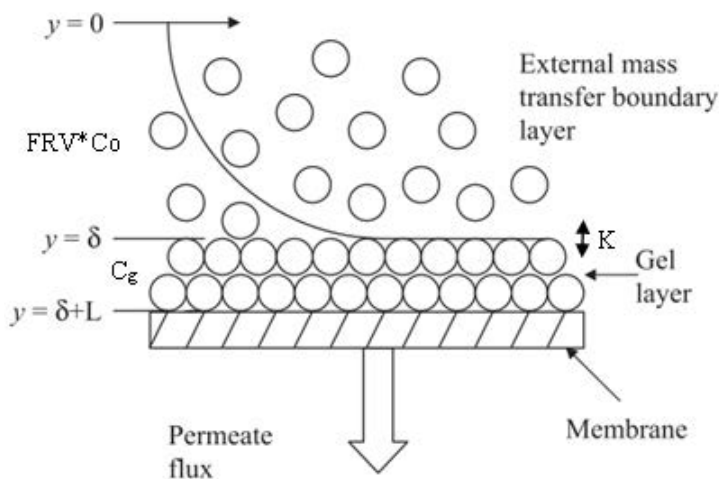


Figure 2 Schematic of the formation of external concentration boundary layer and gel layer over the membrane surface. Mechanism of the fouling layer during cross-flow microfiltration [26].

The thickness of the gel layer on the membrane (L) is calculated as:

$$L(t) = \frac{1}{C_g} \int_0^t J_p \cdot C_0 dt \quad (4)$$

$VCR \cdot C_0$ and δ parameters correspond to the external area of mass transfer in the boundary layer (Figure 2), L and C_g to the polarization gel layer; K and D to the mass transfer and diffusivity of the particles from the area of the boundary layer toward the gel layer on the membrane. All these hydrodynamic parameters will allow understanding the effect of the evaluated factors on the performance

parameters of the process.

2.4 Experimental design and data analysis

The variation of the TMP (1.8, 2.55, and 3.3 bar), the pectin concentration (C_p) [0.25, 0.5 and 0.75%] and cellulose concentration (C_c) [0.04, 0.06, and 0.08% w/w] allow determining the effect of the factors on each response variables. ($K, C_g, VCR \cdot C_0, D$ y δ_b). All trials were carried out at a fixed cross-flow velocity of $5 \text{ m} \cdot \text{s}^{-1}$. The Box-Behnken design was used for different mixtures of the macro-compounds, in this case, the 2^k factorial

design was combined with incomplete blocks to reduce the number of experiments to 15 with three replicates in the center. The statistical analysis was made based on the response surface implemented in Minitab 18 software. In order to obtain the conditions of concentration and TMP that represent the best behavior of each response, a simultaneous optimization was performed in the same software.

3. Results and Discussion

3.1 Physicochemical analysis

The results of the physicochemical analysis carried out on the model solutions indicated that there is a direct relationship between the pair: SIS and the concentration of cellulose, TU_0 and the concentration of pectin, viscosity and C_p , since if one of these variables increases, the other also increases. In the retentate flow, it can be observed that as the cellulose concentration increases, the values of SIS and TU are increased (Table 1), which may indicate a total rejection of such particles, possibly promoted by the layer of pectin gel formed on the membrane and by the particle size of the cellulose molecules, which have a size between 15 and 40 nm, in addition to the insoluble nature of cellulose in water [12].

On the other hand, the physicochemical analysis carried out on the permeate stream indicated that there is no effect of the TMP, the pectin concentration, and the cellulose concentration on the viscosity, pH, soluble solids (SS), SIS and TU_0 ($Pvalue > 0.05$). However, a change in the behavior of these parameters between the permeate and retained stream with respect to the feed stream was observed. In the retentate stream, pH values between 3.65 and 3.99 were recorded, below the pH of the feed (4 ± 0.2) (Table 1), this reduction is attributed to the retention of pectin molecules on the surface of the membrane whose characteristic pH varied between 2.9 and 3.6. This could be the same reason why SS was higher in the retentate (9.9-11.9 °Brix) due to the retention of pectin [27]. For the permeate stream, the pH varied between 4.28 and 4.69 and the SS varied between 9.0 – 10.1 °Brix. The recovery of sucrose in the permeate stream observed in this experiment was due to the permeation of sucrose through the gel layer and through the pores of the membrane.

3.2 Influence of the transmembrane pressure and the composition of the feed on the flux of permeate

The increase in the TMP only decreased the initial values of the permeate flow (Figure 3) without affecting its behavior ($P < 0.05$), which may be due to the range of TMP worked,

which was above 1 bar, and because the concentration of the feed was above 0.5% [28, 29].

In all cases evaluated, the pectin registered the lowest values of J_p and $VCR \cdot C_0$, while cellulose had the highest values at pectin concentrations between 0.25 and 0.75% and of cellulose at 0.06 % (weight percentage) at 1.8 bar (Figure 3).

According to Jiraratananon *et al.* [30], at high pressures, the flux becomes independent of the system pressure and is controlled by mass transfer, where the concentration polarization layer reaches a limit concentration at the surface of the membrane. In addition, the velocity and the convective flow of the large particles towards the surface of the membrane are favored, while the small particles are forced to pass through the gel layer, which contributes to a slight decrease in the resistance of the gel layer as the fluid passes through the membrane [17], [31–34].

The presence of pectin adds considerable resistance to the flow of permeate, while cellulose at small concentrations facilitates obtaining high permeate flows (J_p). As the microfiltration process progresses, the acid-galacturonic-hydrogen bond contained in the pectin collapses and tends to form gels that are then deposited on the surface of the membrane, generating a layer of the filter cake that, as the process goes on, increases the thickness of the cake and the resistance to the passage of the fluid [4], [35–40]. Conversely, cellulose acted as a dispersing agent of the gel layer that forms the pectin. Several authors [26, 27, 41] also found that pectin is the main fouling agent of the membrane and that it directly affects the performance of the CFM process.

3.3 CFM Performance Assessment

Microfiltration technology can be technically feasible in an industrial application if permeate fluxes are greater than $50 \text{ L/h} \cdot \text{m}^2$ [36]. Based on the above, the feasible time was established as the processing time in which a limit flux of $65 \text{ L/h} \cdot \text{m}^2$ was reached, considered as an indication of the moment in which the retention valves must be opened to perform the sweep of the fouling layer generated on the surface on the membrane, which contributes to improving the performance of the CFM process.

In Table 2, it is observed that at low concentrations of pectin and cellulose, the processing time and the final permeate flux are greater; however, when the concentration of pectin is 0.75%, the concentration of cellulose does not affect the processing time ($p < 0,05$). According to Urošević *et al.* [9], a greater quantity of pectin and cellulose particles could represent a less porous and more compact layer on the surface of the membrane that hinders the filtration process.

Table 1 Main physicochemical properties of each flow streams at different pressures

Cp (%w/w)	Cc (%w/w)	TMP (bar)	Feed			Permeate				Retentate		
			SIS (g/L)	μ (cP)	TU (NTU)	pH	SS (°Brix)	μ (cP)	pH	SS (°Brix)	SIS (g/L)	TU (NTU)
0.25	0.04	2.55	6.08	2825	81	4.52	10.1	1.70	3.81	11.85	49.9	882
0.25	0.06	1.8	10.53	2783	97	4.62	10.0	1.67	3.85	10.90	74.6	1465
0.25	0.06	3.3	10.53	2783	97	4.53	10.0	1.67	3.79	11.90	82.1	1911
0.25	0.08	2.55	11.21	2897	118	4.28	9.0	1.48	3.43	9.90	57.9	878
0.5	0.04	1.8	9.86	5117	130	4.40	10.0	1.67	3.76	10.95	44.8	658
0.5	0.04	3.3	9.86	5117	130	4.52	9.6	1.60	3.68	10.60	23.9	581
0.5	0.06	2.55	12.48	4763	146	4.54	9.8	1.64	3.79	11.43	68.9	1164
0.5	0.08	1.8	9.06	5315	164	4.47	10.0	1.67	3.58	11.10	74.4	1416
0.5	0.08	3.3	9.06	5315	164	4.53	9.3	1.55	3.65	10.95	57.7	890
0.75	0.04	2.55	8.28	8886	156	4.68	10.0	1.67	3.99	11.50	58.4	645
0.75	0.06	1.8	14.86	8488	186	3.65	9.8	1.64	3.66	10.45	58.3	1234
0.75	0.06	3.3	14.86	8488	186	4.69	9.0	1.48	3.87	11.75	77.2	1129
0.75	0.08	2.55	13.21	8116	205	4.33	10.0	1.67	3.68	11.90	72.9	2909

Cp: pectin concentration; Cc: cellulose concentration

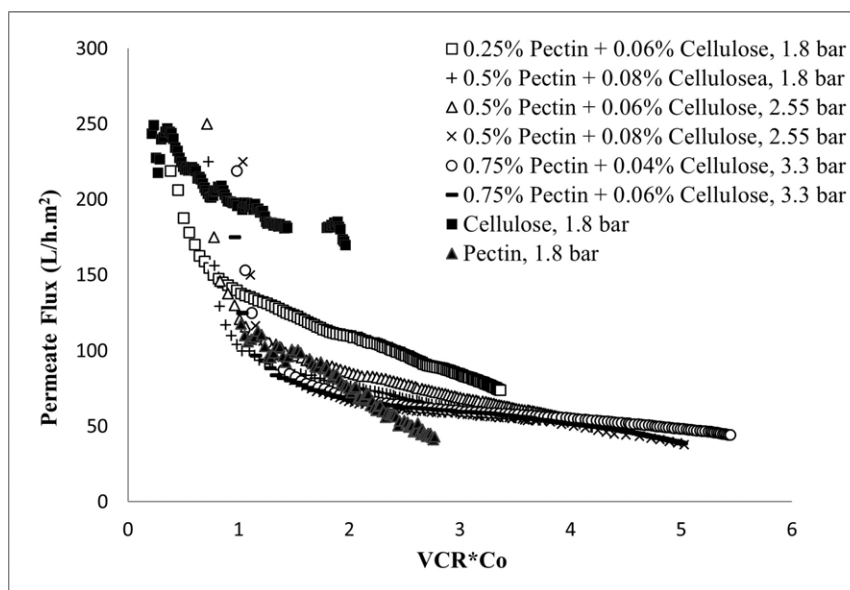


Figure 3 Microfiltration of model solutions at different concentrations of pectin and cellulose, and at different PTM, at constant temperature and cross-flow velocity ($T = 35 \pm 2^\circ\text{C}$; $v = 4.5 \text{ ms}^{-1}$).

The feed flow inside the pipe of the CFM equipment behaves like turbulent flow before entering the channels of the membrane thanks to the action that exerts the high pressures and the high velocities worked, which decreases the probability of deposition of pectin particles on the surface of the membranes when the concentration of this in the feed is low; while the flow behavior changes when going through the channels of the membranes (from turbulent to laminar), therefore the gel layer would take a longer time to form resulting in a longer processing time [12, 42].

Although at high TMP, there was no significant effect on the performance parameters, it can be observed that the effect of the TMP on these is minimal at concentrations of pectin and cellulose higher than 0.5% and 0.08%, respectively. In general, at C_p 0.5% and low TMP, the formation of the gel layer is more likely, which makes it difficult to reach quickly the 65 L/hm^2 established so that the process at the industrial level is feasible.

High concentrations of pectin significantly influenced the decrease in permeation ($P < 0.05$) [Table 2]. Taking into account that the permeation is directly related to

Table 2 CFM performance parameters.

C_p [% w/w]	C_c [% w/w]	PTM (bar)	Final flux (L/m^2h)	Feasible time (min)	Permeation (L/m^2kWh)
0.25	0.04	2.55	70.63	190	39.24
0.25	0.06	1.8	73.91	180	41.06
0.25	0.06	3.3	65.47	120	36.37
0.25	0.08	2.55	64.06	116	35.59
0.5	0.04	1.8	49.58	56	27.55
0.5	0.04	3.3	51.98	73	28.88
0.5	0.06	2.55	48.27	58	26.82
0.5	0.08	1.8	47.66	50	26.48
0.5	0.08	3.3	50.57	53	28.10
0.75	0.04	2.55	44.17	27	24.54
0.75	0.06	1.8	37.5	15	20.83
0.75	0.06	3.3	38.85	25	21.59
0.75	0.08	2.55	37.92	20	21.06

the volume of permeate and inversely to the area of the membrane and the power of the pump, it could be said that controlling the concentration of pectin would obtain a greater amount of permeate with lower energy consumption, without taking into account the effect of the cellulose concentration and the TMP. This behavior is strongly related to the contribution of pectin in the polarization phenomena of the membrane, also reported by da Silva & Scheer [43], who worked with pectin solutions between 0.2 and 2% (up to 2%) on ceramic membranes from 0.44 μm to TMP between 0.4 and 1.2 bar. However, it is necessary to analyze the hydrodynamic behavior of the fouling layer in order to ensure that these factors do not affect the performance of the process.

3.4 Hydrodynamic characteristics of the fouling layer

The experimental values of permeate flux vs $VCR \cdot C_0$ were adjusted to gel and mechanistic polarization models. The results showed that both models, the gel polarization and the mechanistic adjusted very well to the behavior of the permeate flow ($R^2 > 0.9$). The variation of cellulose and pectin concentration affected the values obtained from the boundary layer and the gel layer ($P < 0.05$) whereas the C_p influenced only the values of $VCR \cdot C_0$, where increasing the C_p reduces the concentration of particles in the retentate, since they tend to deposit more easily on the surface of the membrane. The low concentration of pectin used (0.25 %) and the operating conditions facilitate the turbulence of the solution at the entrance of the membranes facilitate.

Given the operating conditions that favor the turbulence of the solution at the entrance of the membranes and the low concentration of pectin used (0.25 %), it facilitates the

removal of the pectin particles and the incorporation of these into the volume of the solution, resulting in higher values of $VCR \cdot C_0$. According to Schafer and Hamachi [44, 45], when the solutions contain high concentrations of pectin, the deposition of particles and the formation of the gel layer are more easily promoted.

When studying the interaction between the C_p and the C_c , it can be observed that the thickness of the boundary layer is lower to low C_c and high C_p , lowering the possibility of conformation of aggregates between the pectin and cellulose molecules via hydrogen bonds [35]. At low concentrations of C_p and intermediate concentrations of C_c (0.06%), high values of K and D are obtained. As mentioned in section 2.2, cellulose could be acting as a dispersing agent and as a turbulence promoter, which helps to remove the pectin particles that may be deposited on the membrane surface and to reduce concentration polarization in the boundary layer of the membrane, improving mass transfer and process performance represented by the K values reported in Table 3 [46].

Taking into account the effect of the interaction between the C_p and the TMP on K and δ , it can be observed according to Table 3 that at high C_p and low TMP the values of these parameters decrease, predominating in this case the deposition of pectin on the membrane as mentioned above. The decrease of K and δ can be attributed to the increase in the viscosity of the retentate, which favors the formation of dense aggregates that impede the fluid flow [12], which agrees with Lee and Clark [47] who attributed the decrease of K to the decrease in the feeding velocity caused by the increase in viscosity.

The results obtained showed that both the thickness

of the gel layer (L) and the particle concentration (C_g) depend on the C_p and C_c and which are independent of the TMP in the range of evaluated pressures. The thickness was affected mainly by the C_c where at low C_c and C_p the lowest values were obtained, favorable conditions to improve the performance of the process. However, with the increase in C_c the effect of C_p becomes more noticeable presenting a maximum in the thickness at C_c of 0.06% and C_p of 0.5%. This behavior could be related to the change in the trend of the performance parameters K and δ to $C_p > 0.5\%$ presented in Figure 4, which could indicate that under these concentrations, the formation of strongly bound aggregates is favored, forming a less permeable and a denser layer.

3.5 Optimization of the process performance

The optimization results represented by the response surface models and the contour plots (Figures 5 - 7) reiterated that the concentrations of pectin and cellulose are the main variables that influence on K , C_g and δ , being more remarkable the influence of cellulose concentration on these parameters. The explanation of the phenomena that occurred for these responses has been previously discussed and is mainly due to the fouling of pectin and dispersing effect of the cellulose. The response surface models obtained have a high degree of reliability since the correlation coefficients are above 0.8, and the lack of adjustment is not significant ($P > 0.05$).

The conditions (C_p , C_c and TMP) in which the maximum results of process time, permeation and K , and minimum of C_g were found are: pectin concentration 0.25%, cellulose concentration 0.0631% and TMP 1.8 bar with a desirability of 0.9091 which guarantees that 90.91% of the results will obtain the expected value. In Figure 8, the predicted values can be observed under the global optimum condition, showing desirability levels greater than 0.87 for each of the optimized parameters.

The lower values of C_g were shown at low concentrations of pectin and intermediate concentrations of cellulose (Figure 6) so it is inferred once again that cellulose has a dispersing effect on the gel layer that pectin forms when low concentrations of cellulose are used.

In summary, the results of the global optimization indicated that the concentration of pectin in the range studied had a fouling effect on the membranes while the concentration of cellulose had a dispersing effect, conditions that managed to improve the overall performance of the process. On the other hand, response variables were optimal at low TMP, different other results were reported in the literature that showed maximum values at intermediate TMP [41].

4. Conclusions

Employing model fluids ensures the reproducibility of the data, and allows predicting the behavior of the permeate flux for tropical fruits that contain the same concentration of pectin and cellulose used in this research. In addition, model fluids are useful to understand how permeate flux is affected by juice characteristics, operating variables, the morphology of fouling compounds such as pectin and cellulose, and their interaction with membrane materials.

The main factors influencing the performance of the process and the hydrodynamic characteristics were the concentration of pectin and cellulose, while the TMP did not have a significant effect. However, the step-wise methodology allowed establishing the effect of the TMP on the fouling resistance and the resistance specific to the cake. The cellulose present in the model fluid exerted a dispersing effect on the gel formed, which improved the mass transfer through the membrane at intermediate concentrations (0.06%).

The overall optimum condition was found at 0.25% pectin, 0.0516% cellulose, and 1.936 bar with desirability in results of 78.5%, in which long processing periods were achieved with good mass transfer coefficient (K), controlled gel layer over the membrane (C_g), and good yield permeation that ensures a greater amount of permeate with low energy consumption and high efficiency. In this way, both, the overall performance of the process is ensured as well as the useful life of the membranes, especially when tropical fruit juices are used, which are highly desired for their bioactive compounds, but which have high contents of pectin and cellulose. In this case, the fouling effect of pectin and the dispersant of cellulose was corroborated.

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6. Declaration of competing interest

We declare that we have no significant competing interest, including financial or non-financial, professional, or personal interest interfering with the full and objective presentation of the work described in this manuscript.

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Table 3 Hydrodynamic characteristics of gel and boundary layers

C_p (% w/w)	C_c (% w/w)	PTM (bar)	Gel polarization model			Mechanistic model			L (mm)	δ_b (μ m)	VCR*Co
			K (10^{-3} m h $^{-1}$)	C_g (%p/p)	R^2	D (10^{-6} m 2 h $^{-1}$)	C_g (%w/w)	R^2			
0.25	0.04	2.55	41.0(0.9) ^b	20.9(1.08) ^b	0.951	3.08(0.292) ^b	12.5(2.02) ^b	0.933	2.11	75.1	0.4-3.02
0.25	0.06	1.8	54.6(0.8) ^b	14.1(0.40) ^b	0.977	5.69(0.126) ^b	6.27(0.19) ^b	0.982	3.37	104.1	0.39-3.36
0.25	0.06	3.3	40.7(0.6) ^b	17.5(0.58) ^b	0.976	3.59(0.137) ^b	8.05(0.47) ^b	0.970	2.40	88.2	0.52-3.02
0.25	0.08	2.55	43.8(0.3) ^b	13.5(0.17) ^b	0.995	4.22(0.048) ^b	5.80(0.09) ^b	0.994	2.99	96.3	0.63-3.15
0.5	0.04	1.8	29.9(0.3) ^b	23.1(0.55) ^b	0.987	2.26(0.059) ^b	10.7(0.42) ^b	0.985	2.34	75.4	0.74-4.11
0.5	0.04	3.3	32.5(0.3) ^b	23.4(0.54) ^b	0.988	2.53(0.074) ^b	10.9(0.48) ^b	0.982	2.53	77.9	0.61-4.28
0.5	0.06	2.55	42.7(0.6) ^b	13.4(0.31) ^b	0.981	3.92(0.063) ^b	6.01(0.11) ^b	0.982	3.62 (0.54) ^c	9.15 (0.34) ^c	0.87-4.08
0.5	0.08	1.8	37.5(0.5) ^b	15.9(0.39) ^b	0.979	3.10(0.028) ^b	7.39(0.08) ^b	0.995	3.30	87.1	0.83-4.27
0.5	0.08	3.3	38.7(0.3) ^b	15.5(0.20) ^b	0.995	2.33(0.035) ^b	6.80(0.08) ^b	0.994	3.34	89.1	0.93-4.13
0.75	0.04	2.55	25.8(0.4) ^b	32.3(1.24) ^b	0.968	1.82(0.064) ^b	14.8(0.79) ^b	0.974	1.96	70.7	1.35-5.44
0.75	0.06	1.8	27.4(0.5) ^b	20.6(0.74) ^b	0.961	2.13(0.055) ^b	8.61(0.28) ^b	0.964	2.85	77.8	1.13-4.86
0.75	0.06	3.3	30.2(0.5) ^b	20.8(0.68) ^b	0.966	2.50(0.056) ^b	8.57(0.23) ^b	0.969	3.00	82.6	1.29-5.01
0.75	0.08	2.5	28.9(0.8) ^b	21.9(1.18) ^b	0.957	2.33(0.087) ^b	9.00(0.45) ^b	0.960	2.31	80.7	1.45-5.03

^aFitted Adjustment parameters calculated for shear stress (γ) estimated as $\gamma = 2v/d = 2180 \text{ s}^{-1}$ where v is the average crossflow rate.

^bIn the brackets, the standard error of estimation.

^cStandard deviation of repetitions (only central point).

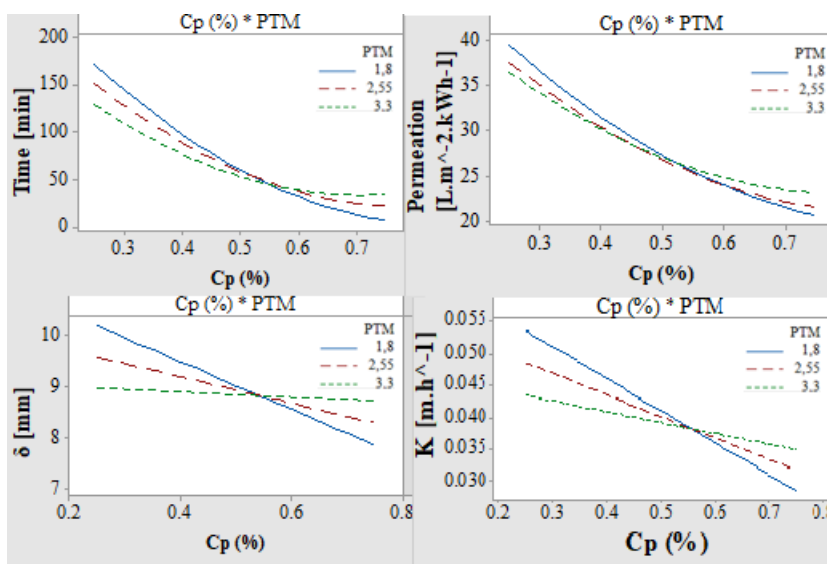


Figure 4 Interaction plot for pectin concentration and transmembrane pressure effect on process time, permeation, thickness of the boundary layer, and mass transfer coefficient.

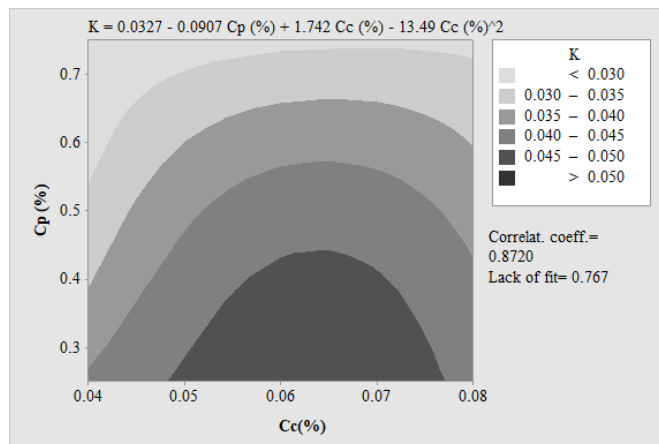


Figure 5 Contour plot of K, effect of pectin, and cellulose concentration.

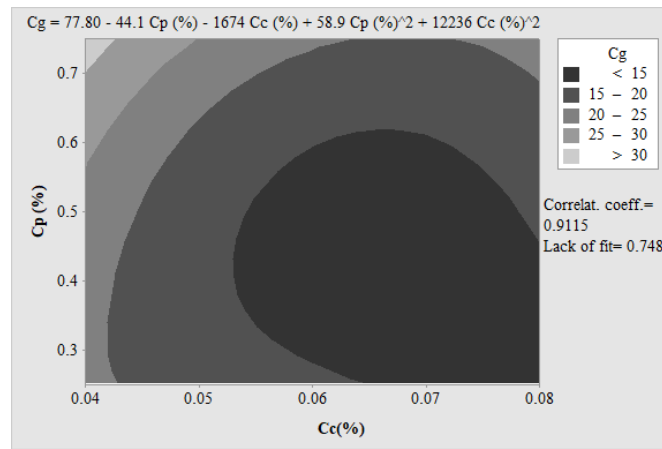


Figure 6 Contour plot of C_g , effect of pectin, and cellulose concentration.

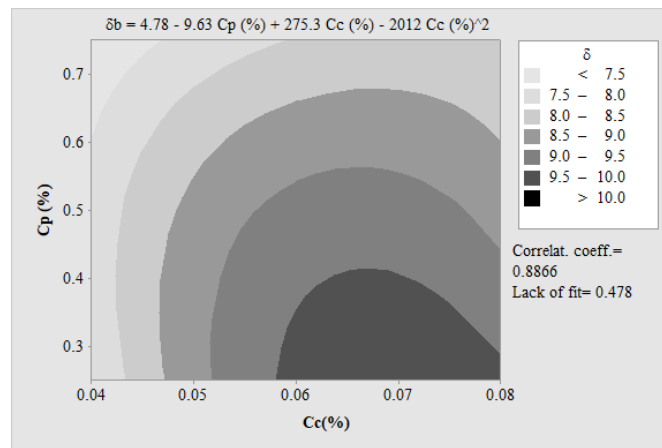


Figure 7 Contour plot of the thickness of boundary layer, effect of pectin, and cellulose concentration.

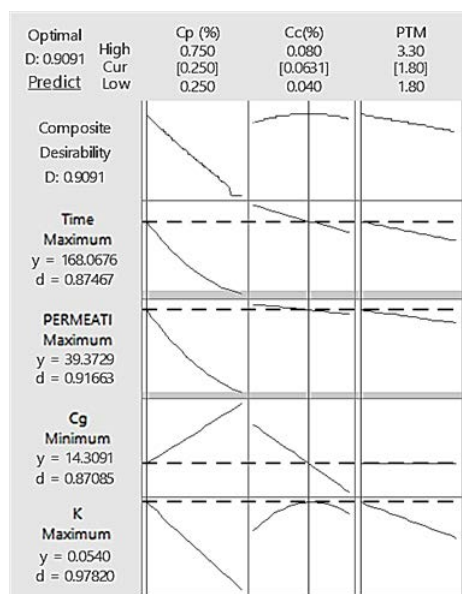


Figure 8 Simultaneous optimization of process time, permeation, C_g and K, regarding C_p , C_c and PTM.

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8. Author contributions

PhD. Heidy Lorena Gallego-Ocampo, PhD candidate Karen Johana Ortega-Villalba obtained the experimental data. All the authors worked on the analysis of the experimental data for this study, writing and compilation of the paper.

9. Data availability statement

The authors confirm that the data supporting the findings of this study are available within the article [and/or] its supplementary materials.

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