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Microfiltration of passion fruit juice using hollow fibre membranes and evaluation of fouling mechanisms

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ABSTRACT

This work evaluated the microfiltration process for the clarification of passion fruit juice. Moreover, the influence of some pretreatments (centrifugation, enzymatic liquefaction and chitosan coagulation) before passion fruit juice microfiltration was analyzed. Enzymatic treatment reduced the juice viscosity, and centrifugation step was important for colour and turbidity reductions. Chitosan addition was the most promising pretreatment, since it provides the highest reductions of colour and turbidity, enabling the highest permeate flux in the microfiltration process of pretreated passion fruit juice. The microfiltration process with hollow fibre membranes resulted in a clean passion fruit juice, almost free of turbidity. The applied pretreatment did not influence the characteristics of the obtained permeate. According to the obtained results, the predominant fouling mechanism depends on the applied pretreatment. In centrifuged and enzymatic treated samples, cake formation was found to be the major fouling factor, while internal pore blocking occurred during the filtration of the chitosan pretreated sample.

1. Introduction

Yellow passion fruit (*Passiflora edulis var. flavicarpa*) is a tropical fruit, recognized for its unique attractive flavour. Its distinct aroma makes passion fruit a desirable ingredient for many formulated beverages and food products. Nevertheless, the flavour of passion fruit is extremely sensitive to change as a result of heat processing. Conventional stabilization methods, such as thermal pasteurization, induce to large losses of flavour volatile compounds, and to specific changes in aromatic compounds, even when short-duration procedures are applied (Vaillant et al., 1999; Yu and Chiang, 1986). Sensorial properties and nutritional compounds such as vitamins of fresh juices can be degraded at temperatures higher than 50 °C (Cisse et al., 2005; Shaw et al., 2001; Vaillant et al., 2001a). Beverage producers are looking for innovations, aiming to obtain products based on natural fruits, richer in vitamins, minerals, sugar and antioxidants.

Some fruit juices present natural degrees of turbidity due the presence of insoluble matter such as pectin, starch, cells from the juice, among others. Depending on the application of the fruit juice, a clarification process is required. Clarified fruit juices are needed for the production of sparkling clear beverages (soft drinks, clear juice cocktails, natural aromatic waters, mineralized water, alcoholic beverages, cold teas with clear juice), candies (melting products), pastries (natural essences, translucent fruit sauce), uniformly juice fruit juice blends (cocktails, ice-creams, among others), natural translucent jelly products (fruit jellies, gelatins, among others) (Vaillant et al., 2001b). Brazil is the largest world's producer and consumer of passion fruit. These pieces of information show that several market opportunities exist not only for passion fruit applications, but for many clarified juices made from fruits or vegetables with originally high juice content.

Conventional clarification processes usually involve some sequential batch operated steps, such as enzymatic pretreatment, clarification with bentonite, gelatin or diatomaceous earth and pasteurization (Cheryan and Alvarez, 1995). Membrane processes have been applied for the clarification and stabilization processes of fruit juices (Carneiro et al., 2002; Cheryan and Alvarez, 1995; Matta et al., 2004), since it provides a sterile product with high sensorial quality.

Currently, produced inorganic membranes are usually in the form of either finite sized tubes with diameters of at least several millimetres or flat discs and, consequently, have low surface area/volume ratios. These low area/volume ratios compare unfavourably with polymeric hollow fibre modules where area/volume ratios of several thousand are obtainable.

The applied pretreatment plays an important role for juice filtration. Low permeate fluxes can be observed during juice filtration due to its relative high content of pectin, lignin and hemicellulose

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(Vaillant et al., 1999). In order to control and reduce fouling occurrences in pressure driven processes such as microfiltration and ultrafiltration, pretreatments are usually used prior to the filtration, aiming to decrease the content of macromolecules which are able to be accumulated in the membrane surface. These pretreatments include mainly the application of pectolic enzymes (Domingues et al., 2011; Jiraratananon and Chanachai, 1996; Vaillant et al., 2001b). Alternative materials, such as chitosan, were also reported in the literature (Chatterjee et al., 2004; Domingues et al., 2012; Oszmianski and Wojdylo, 2007).

Membrane fouling in cross-flow membrane separation is a key factor, affecting the economical and commercial viability of a membrane process which essentially depends on the obtained permeate fluxes (Cheryan and Alvarez, 1995; Fane and Fell, 1987; Orsello et al., 2006). A better comprehension of fouling occurrences during juice filtration can elucidate alternative ways to control the flux decline. There are many available mathematical models in the open literature that describe the transport mechanisms throughout porous membranes, although these contributions are not completely satisfactory to identify accurately permeate flux nor the nature of fouling formation (Aliaa et al., 2010; de Bruijn et al., 2002; Reis et al., 2009), since most of these models are semi-empirical and require a large number of experimental data, besides being able to be applied under certain restriction and necessary correlations. Arnot et al. (2000) applied some models to the experimental data of crossflow ultrafiltrations of water–oil emulsions and reported that the model proposed by Field et al. (1995) was the most accurate for predicting the permeate flux behaviour. Barros et al. (2002) investigated the fouling mechanism in pineapple juice ultrafiltration based on the mathematical model proposed by Field et al. (1995). Similar analyses were carried out by Cassano et al. (2008) for kiwi fruit juice ultrafiltration. A recent re-examination of the model proposed by Field et al. (1995) better elucidated the concept of critical flux when complete pore blocking occurs (Field and Wu, 2011).

This work proposes the application of different pretreatments (centrifugation, enzymatic liquefaction and chitosan clarification) for the microfiltration of passion fruit juice, and the evaluation of the influence of these pretreatments on the physical–chemical characteristics of the obtained clarified juice, as well as in the permeate flux. Moreover, the mathematical model proposed by Field et al. (1995) was applied in order to describe the obtained experimental fluxes, elucidating the major fouling occurrences.

2. Materials and methods

2.1. Passion fruit juice

The passion fruit juice was purchased from a local juice industry (Minas Gerais – Brazil). The juice was stored at −16 °C and it was defrosted to room temperature before its use. The passion fruit juice was used in this work without any dilution.

2.2. Pretreatments in passion fruit juice

2.2.1. Centrifugation

Centrifugations of passion fruit juice samples were carried out in a Beckman Coulter Avanti J-25 centrifuge. Samples were centrifuged for 5 min at two different centrifugal forces (6000 and 14,000 g). The supernatant was used for further procedures and analyses.

2.2.2. Enzymatic treatment

Enzymatic treatment was conducted with the enzymatic complex Pectinex 3 XL (Novozymes) in passion fruit juice samples placed in 250 mL conic flasks at constant agitation and temperature for 90 min. Reaction temperature and enzyme concentration were set at 44 °C and 1 mL L^{-1}, respectively, as indicated in a previous study (Domingues et al., 2011). After enzymatic liquefaction, samples were heated at 90 °C for 5 min, in order to interrupt the reaction.

2.2.3. Clarification with chitosan

Chitosan from shrimp shells was purchased by Sigma–Aldrich (Iceland). A stock solution was prepared by hydrolysing shrimp shell chitosan with a 5% v/v solution of acetic acid (Dinamica, Brazil). Stock solutions at concentrations of 0.1, 0.05 and 0.01 kg L^{-1} of chitosan were evaluated in preliminary tests, but due to the high viscosity of the first two stock solutions, the mixing of the chitosan solution with the passion fruit juice was not satisfactory since a thick mass was formed in the centre of the sample inducing to an unsatisfactory coagulation process. Thus, the concentration of stock solution was chosen to be 0.01 kg L^{-1}.

The coagulation/flocculation process of passion fruit juice with chitosan was carried out in a Jar test apparatus at the optimized conditions reported by Domingues et al. (2012). A previous mild centrifugation of the samples at 6000 g was carried out. Samples of 200 mL of centrifuged passion fruit juice were put in glass beakers and chitosan solution was added for a concentration of 300 ppm of chitosan. Jar test parameters were set as follows: 150 rpm of fast agitation for 3 min and 50 rpm of slow agitation for 12 min. Samples were decanted during 90 min.

2.2.4. Combination of pretreatments

A combination of centrifugation, enzymatic treatment and clarification with chitosan as pretreatments for passion fruit juice was carried out, resulting in different samples after each step, in a total of 7 samples (S1–S7), as described below.

Passion fruit juice as received (S1) was treated by the enzymatic treatment (S2) and by the centrifugation processes at 14,000 g (S3) and 6000 g (S5). The samples S3 and S5 received a sequential enzymatic treatment (resulting in the samples S4 and S6). The sample S5 was also sequentially treated by chitosan coagulation/flocculation process, resulting in the sample S7. All these samples were further microfiltrated, resulting in a number of seven microfiltrated samples.

2.3. Microfiltration process

Microfiltration experiments were made in a pilot-scale microfiltration module purchased by PAM (Brazil). A simplified scheme of the microfiltration system is shown in Fig 1. Hollow fibre mem-

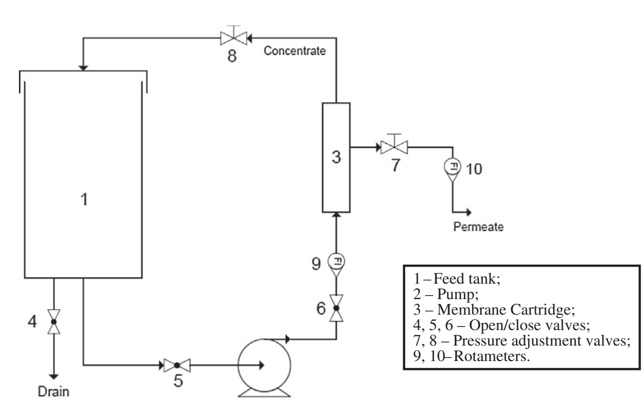

Fig. 1. Simplified scheme of the microfiltration module.

branes of polieterimide with an average pore diameter of 0.40 μm and with a filtration area of 0.056 m^2 were used.

Experiments were carried out at room temperature, with 2.5 L of the passion fruit sample in the feed tank. Transmembrane pressure was adjusted to 1 bar and the microfiltrations were carried out until the permeation flux remained almost constant.

2.3.1. Membrane cleaning procedures

Permeate flux of distilled water was measured at 1 bar of transmembrane pressure at the beginning of each microfiltration run. These values were taken as a reference to guarantee membrane cleaning after the experiments with passion fruit juice. The cleaning procedure was done by the following steps: rinsing the external surface of the membranes with water; recirculation of a 1% v/v NaOH solution for 60 min through the external membrane surface; recirculation of a 5% v/v nitric acid through the external membrane surface and finally recirculation of a 1 ppm solution of pectinase from *Aspergillus niger* (Sigma Aldrich) at 50 °C for 60 min. After each step, permeate flux of distilled water was measured, and the procedure was stopped if the permeate flux was recovered at a minimum of 90%.

2.4. Physico-chemical analysis

Passion fruit juice, before and after pretreatments and microfiltration processes, were analysed for turbidity, colour, total soluble solids (TSS) and viscosity. Turbidity was measured with a Nova Organica HD 114 turbidimeter (Brazil). Colour was measured as absorbance at 540 nm in a Shimadzu UV mini 1240 spectrometer (Japan), as suggested by Rai and De (2009). Total soluble solids were measured with a Hanna Instruments HI 96801 refractometer (USA), expressed as °Brix. Viscosities were measured using a Brookfield LVDV-III digital rheometer (USA) at 25 °C at a shear rate value of 83 s^{-1}, since some samples presented non Newtonian behaviour.

2.5. Mathematical modelling

The mathematical model proposed by Field et al. (1995) was applied to describe the fouling mechanisms during the crossflow microfiltration of passion fruit juice. The characteristic equation proposed by the model is described in Eq. (1).

$$-\frac{dJ}{dt} = k_n(J - J^*)J^{2-n} \quad (1)$$

where J^* is the steady-state flux and k_n is an adjusted parameter. The parameter n is set according to the predominant fouling mechanism: $n = 0$ for cake filtration, $n = 2$ for complete pore blocking; $n = 1$ for partial pore blocking and $n = 1.5$ for internal pore blocking.

The Levenberg–Marquardt method was used to solve the nonlinear least squares curve-fitting problem, applying an integration step of 10^{-3} with precision of 10^{-8}. The Levenberg–Marquardt method was programmed in Fortran language, combining the Gauss and the Steepest Descent methods (Press et al., 1996). The sum of quadratic differences (the residual) between the experimental flux data and the flux data computed with the Field model was minimized, obtaining the best adjusted parameter (k_n). This procedure was carried out for each n value in the Field equation. Thus, the best equation to describe the fouling occurrence will be that with the lowest residual value, since this will be the best equation to describe the experimental data.

3. Results and discussion

3.1. Comparison of pretreatments on passion fruit juice

Pretreated passion fruit samples were characterized by turbidity, colour, viscosity and total soluble solids. Table 1 shows the physico-chemical characterization results of the pretreated samples of passion fruit juice. The expressed values are average results of triplicates followed by their respective standard deviations (in brackets).

Data reported in Table 1 suggests that TSS was not affected by the applied pretreatments. Enzymatic treatment reduced the viscosity of raw passion fruit pulp. If the pairs of samples S1/S2, S3/S4 and S5/S6 are considered individually, it can be observed a significant reduction between the averages of the values of viscosity of these samples (p values equal to 0.0108, 0.0059 and 0.00008, respectively). Moreover, the combination of centrifugation and enzymatic treatment did not affect viscosity result, as it can be observed by the comparison between the pairs of samples S3/S5 ($p = 0.2006$) and S4/S6 ($p = 0.4714$). However, the increase in the centrifugation speed induced higher reductions of turbidity and colour.

A reduction in turbidity results was observed after the enzymatic treatment, but only when the enzymatic treatment was carried out in the raw pulp, as can be observed comparing the pair of samples S1/S2 ($p = 0.0012$). The enzymatic treatment did not affect the colour of treated passion fruit juice. de Oliveira et al. (2012) treated diluted passion fruit juice with the Pectinex Ultra SP-L enzyme at a lower concentration (0.3 mL/L) and reported values of turbidity and colour similar to those achieved in this work.

The chitosan pretreatment was efficient in the reduction of turbidity, colour and viscosity, with 99%, 96% and 94% of reduction of these parameters related to the values of turbidity, colour and viscosity of raw passion fruit juice, respectively. These were the highest decreases for turbidity and colour, among the proposed pretreatments.

Fig. 2 shows the percentage reductions in the evaluated physico-chemical parameters in comparison to the raw juice ones.

In an overview of the pretreatment effects, it is possible to conclude that the enzymatic treatment was efficient on viscosity reduction and the centrifugation is necessary to separate suspended solids from the passion fruit juice, which can be observed by turbidity and colour reductions. The combined treatment of centrifugation at 14,000 g and enzyme addition (S4) was the most efficient for viscosity reduction. The action of enzymes on viscosity reduction of fruit juices is widely reported in the scientific literature. ‘Aliaa et al. (2010), Jiraratananon and Chanachai (1996) and Lee et al. (2006) reported decreases in the viscosity of pitaya, passion fruit, and banana juices, respectively, after enzymatic liquefaction.

According to the obtained results, the combination of a mild centrifugation (6000 g) and chitosan treatment (S7) is a suitable alternative for passion fruit juice pretreatment, since this treatment was the most efficient for turbidity and colour reductions, besides reducing the viscosity of passion fruit juice. Moreover, the addition of chitosan as a substitute of the enzymatic treatment will probable represent a cost reduction in the passion fruit processing, since chitosan is added to the juice in a small concentration (300 ppm) and it is cheaper than pectolytic enzymes. Besides, chitosan treatment is carried out at room temperature while the enzymatic treatment requires a temperature of 60 °C.

3.2. Effect of pretreatments on the permeate flux during microfiltration process

Before microfiltration tests with passion fruit juice, the permeate flux of the clean membrane was measured, in order to establish

Table 1
Physico-chemical characteristics of passion fruit pulp samples according to the proposed pretreatments.

Sample	Turbidity (NTU)	TSS (°Brix)	Color (Abs 540 nm)	Viscosity (cP)
S1	3982 (127.95)a	12.60 (0.98)a	3.8353 (0.0026)a	46.74 (6.62)a
S2	3220 (96.44)b	12.47 (1.25)a	3.6475 (0.2583)a	27.58 (3.26)b
S3	633 (22.12)c	12.23 (0.84)a	1.3076 (0.5142)b	3.71 (0.60)c
S4	672 (47.98)c	12.27 (0.81)a	1.4383 (0.2933)b	1.82 (0.14)d
S5	1054 (54.52)d	11.50 (0.53)a	2.7551 (0.1763)c	4.28 (0.24)c
S6	965 (42.50)d	11.83 (0.49)a	2.6540 (0.1426)c	1.90 (0.10)d
S7	28 (14.21)e	11.00 (1.74)a	0.1518 (0.0786)d	2.45 (0.12)e

Indexes "a", "b", "c", "d" and "e" represent significance between treatments in an analysis of variance (ANOVA) at $p < 0.05$. Same letters mean that samples did not differ statistically and different ones mean that they differ at 95% of confidence level. The expressed values are average results of triplicates followed by their respective standard deviations (in brackets).

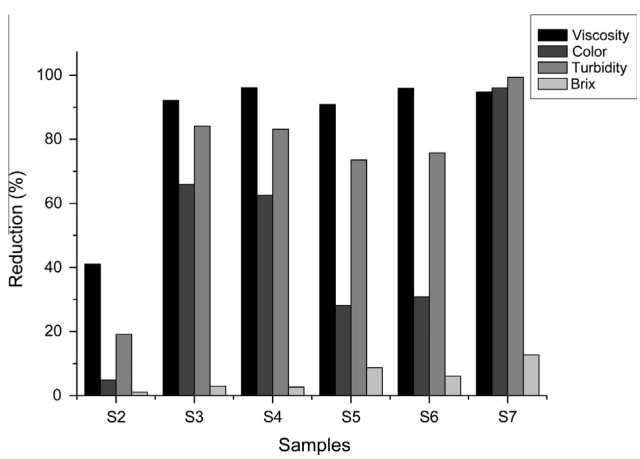

Fig. 2. Reductions in physico-chemical parameters of raw passion fruit juice after the applied pretreatments.

a reference value of flux for future cleaning procedures. It was observed a value of 932.14 L h^{-1} m^{-2} for permeate flux of distilled water at 1 bar of transmembrane pressure. After experiments and cleaning procedures, a flux of 863.93 L h^{-1} m^{-2} (90% of the flux of the clean membrane) was adopted as acceptable for stopping the cleaning process and initiate further microfiltrations.

Microfiltrations of the proposed samples were carried out at transmembrane pressure of 1 bar. Fig. 3 shows the permeate flux behaviour during the microfiltrations of all the considered samples.

The achieved permeate fluxes are probably related with the physico-chemical characteristics of the sample to be filtrated. The samples S1 and S2 presented the lowest flux values since they presented relative high values of colour, turbidity and viscosity (see Table 1).

The enzymatic treatment increased the permeate flux of raw juice. This can be associated with the viscosity reduction observed in the sample S2. Echavarria et al. (2011), Rai and De (2009) and Vaillant et al. (1999) also reported the positive effect of depectinization on permeate flux in membrane filtration processes. Although there are not significant differences between colour and turbidity values of the pair of samples S3/S4 and S5/S6, the addition of the enzyme reduced significantly the viscosity values of the same pairs of samples. Thus, it could be expected that the observed flux of the sample S3 was greater than of the sample S4 and, at the same way, the flux of the sample S5 would be greater that the flux of the sample S6. Moreover, greater rotations in the centrifugation process enable greater turbidity, colour and viscosity reductions. In this way, the observed flux of the samples S3 and S4 would be greater than of the samples S5 and S6. However, the results presented in Fig. 3 show that the observed fluxes for the samples centrifuged and enzymatic treated are quite similar. Comparing these samples, the treatment by centrifugation at 14,000 g plus the enzyme addition (S4) presented the highest permeate fluxes. However, as can be seen in Fig. 3 the treatment with chitosan was the most promising to get higher fluxes.

For a better overview, Fig. 4 shows the values of the final permeate fluxes obtained in all the experiments. de Oliveira et al. (2012) filtrated diluted passion fruit juice pretreated by centrifugation (17,000 rpm) and enzyme addition (300 ppm) with polyamide hollow fibre membranes of 0.3 μm of pore size, reporting a established flux value of 19.5 kg h^{-1} m^{-2} for the filtration at 1 bar. This value reported by de Oliveira et al. (2012) is a slightly higher than the flux obtained in this work when the juice was treated with chitosan (sample S7, 15.2 L h^{-1} m^{-2}). Many filtration operation conditions, as well as the quality of the juice fed in the system,

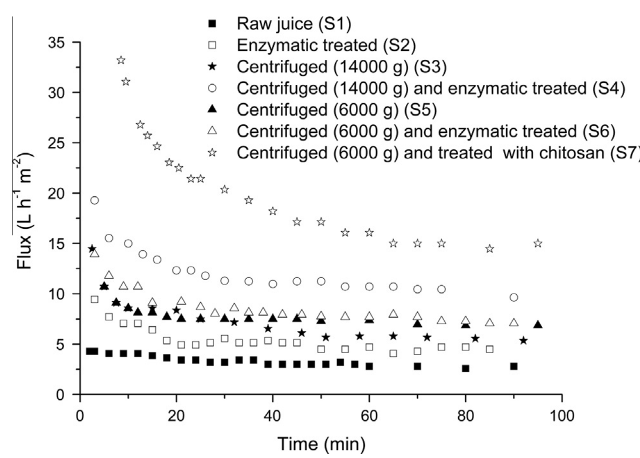

Fig. 3. Permeate flux during microfiltration at 1 bar of all the considered samples.

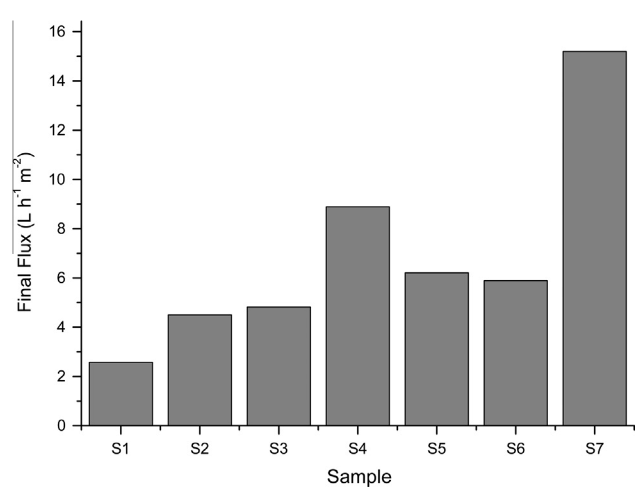

Fig. 4. Final fluxes of all the considered samples in the microfiltration process at 1 bar.

can have influenced the observed permeate fluxes. Since the operation conditions of our system is similar to those used by de Oliveira et al. (2012), we believe that the main factor that influenced the obtained results was the quality of the juice used as feedstock. de Oliveira et al. (2012) filtrated juice with an initial soluble solid content of 7.8 °Brix, while our treated juice presented a initial soluble content of 11 °Brix. The lower soluble content obtained by de Oliveira et al. (2012) is probable related with the applied dilution for the raw passion fruit pulp and the higher centrifugation speed used in the juice pretreatment.

3.3. Physico-chemical analyses of permeate samples

All microfiltration experiments resulted in a yellow colour permeate with very similar visual characteristics.

Fig. 5 presents the characteristics of permeate samples according to the filtration time for the microfiltration of the sample S4. Fig. 5 shows that the data is randomly distributed around its averages, indicating that there are no trends or systematic errors during the data collecting procedures. Moreover, the analysed physico-chemical parameters do not change significantly according to the filtration time, since the variances of each parameter are small in comparison with the respective average values (Table 2). This behaviour was observed for all the other samples.

Table 3 presents the physico-chemical characteristics of permeate samples at some filtration times (15, 60 and 180 min).

Results presented in Table 3 show that the microfiltration process was able to reduce turbidity, colour and viscosity of raw or pretreated passion fruit juice samples. Microfiltration process was especially efficient for the reduction of turbidity and colour of the passion fruit juice. Percentage reductions of turbidity and colour of the sample S7 were equal to 95% and 74%, respectively, since the applied pretreatment (chitosan) already had reduced these parameters. Considering a medium value for all the samples,

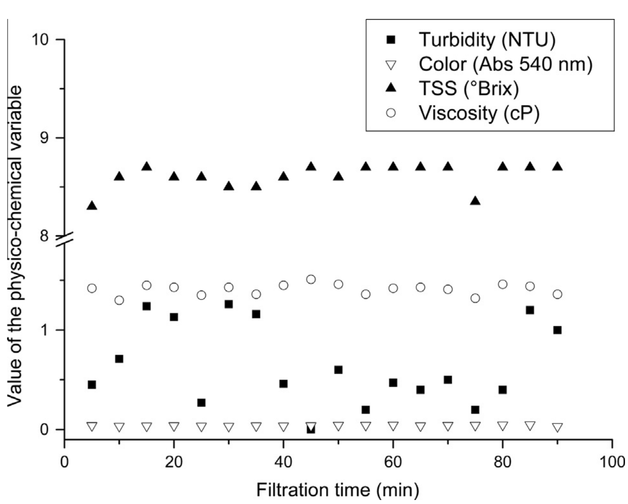

Fig. 5. Physico-chemical characteristics of permeate samples according to the filtration time for microfiltration of the sample S4 at 1 bar.

the microfiltration process reduced TSS content and viscosity at 22% and 57%, respectively.

An analysis of variance (ANOVA) with 95% of significance was carried out in order to check the variability of the physico-chemical characteristics of the microfiltrated passion fruit juice samples according to the filtration time and the applied pretreatment. Table 4 presents the obtained *p*-values analyzing the influence of the filtration time and the applied pretreatment on the physico-chemical variables of the microfiltrated passion fruit.

The analysis of the ANOVA table (Table 4) shows that there are no significant differences between the characteristics of the permeate samples according to the filtration time. Analysis of variance indicated that there is no significant difference between the physical chemical characteristics of the microfiltrated juice regarding to the elapsed time of microfiltrations, indicating that the quality of the permeate is constant from the beginning to the end of the microfiltration procedure. Moreover, the applied pretreatment did no influence turbidity, colour and viscosity of the permeate samples. A significant difference regarding to the applied pretreatment was detected only for the variable TSS. The TSS value of the permeate obtained by the filtration of the sample S1 is slightly inferior of the other averages. This value probably influenced the *p*-value of the variable TSS regarding to the applied pretreatment. The filtration of a sample without any treatment for suspended solids removal and depectinization may have resulted in the formation of gel layer in the membrane surface, decreasing the porosity of the selective layer, retaining some components like soluble sugars and vitamin C (Rai and De, 2009).

3.4. Mathematical modelling of fouling

The equations proposed by Field et al. (1995) were adjusted to the experimental data of permeate fluxes of each microfiltration run. Table 5 presents the values of the root-mean-square deviation (RMSD) between calculated and experimental flux data for each fouling model.

Results presented in Table 5 indicate that the predominant fouling mechanism was complete pore blocking ($n = 2$) in the microfiltration of the sample S1. However, for this filtration, RMSD values are quite closed, indicating that all fouling mechanisms may have occurred during the filtration of the sample S1. For all the centrifuged and enzymatic treated samples (S2–S6), the best adjustment was for $n = 0$, indicating that cake formation was the major fouling factor. Similar results for depectinized passion fruit juice was achieved by Jiraratananon and Chanachai (1996). This result was also observed by de Oliveira et al. (2012) working with passion fruit juice treated by centrifugation and enzyme addition. Nandi et al. (2012) applied the Hermia model to describe the fouling occurrences during the filtration of centrifuged and enzymatic treated orange juice obtaining the best linear adjust to the cake filtration mechanism. Barros et al. (2002) reported that cake formation ($n = 0$) was the major fouling factor during the ultrafiltration of enzyme treated pineapple juice with polysulfone hollow fibre membranes. Rai et al. (2007) also associated the flux decline due to the formation of a gel layer during the crossflow ultrafiltration of depectinized mosambi juice. The large content of soluble solids

Table 2
Mean values and variances of the physico-chemical characteristics of the sample S4 according to the filtration time.

	Variable			
	Turbidity (NTU)	TSS (°Brix)	Color (Abs 540 nm)	Viscosity (cP)
Mean value	0.65	0.0403	8.61	1.41
Variance	0.169	0.00002	0.015	0.003

Table 3
Physico-chemical characteristics of microfiltrated passion fruit juice samples.

Feed sample	Time (min)	Turbidity (NTU)	TSS (°Brix)	Color (Abs 540 nm)	Viscosity (cP)
S1	15	2.30	7.40	0.0480	1.38
	60	1.22	7.50	0.0464	1.40
	180	2.30	6.80	0.0543	1.37
	Average	1.94	7.23	0.0496	1.38
S2	15	2.10	9.00	0.0182	1.40
	60	3.40	8.90	0.0348	1.49
	180	3.10	9.20	0.0364	1.45
	Average	2.87	9.03	0.0298	1.45
S3	15	1.07	9.40	0.0474	1.49
	60	2.58	9.30	0.0449	1.34
	180	1.29	9.20	0.0914	1.64
	Average	1.65	9.30	0.0612	1.49
S4	15	1.21	9.30	0.0558	1.56
	60	2.45	9.40	0.0507	1.47
	180	1.40	9.50	0.0465	1.43
	Average	1.69	9.40	0.0510	1.49
S5	15	2.91	11.20	0.0507	1.34
	60	1.09	10.30	0.0533	1.47
	180	1.67	9.70	0.0532	1.47
	Average	1.89	10.40	0.0524	1.43
S6	15	1.25	9.50	0.0344	1.34
	60	1.43	10.00	0.0347	1.47
	180	0.51	9.90	0.0302	1.47
	Average	1.06	9.80	0.0331	1.43
S7	15	1.71	10.30	0.0567	1.53
	60	1.10	8.50	0.0504	1.63
	180	1.06	11.00	0.0740	1.49
	Average	1.29	9.93	0.0604	1.55

Table 4
p-Values obtained from the analysis of variance (ANOVA) of the physico-chemical variables of the microfiltrated passion fruit according to the filtration time and the applied pretreatment.

Factor	Variable			
	Turbidity	TSS	Color	Viscosity
Filtration time	0.8523	0.6422	0.3446	0.6479
Pretreatment	0.1585	0.0012*	0.4318	0.3404

* Significant at $p < 0.05$.

Table 5
Root-mean-square deviation (RMSD) between calculated and experimental flux data for each fouling model.

Sample	Fouling model			
	Cake filtration ($n = 0$)	Intermediary pore blocking ($n = 1$)	Internal pore blocking ($n = 1.5$)	Complete pore blocking ($n = 2$)
S1	0.1365	0.1246	0.1203	0.1174[a]
S2	0.3712[a]	0.5890	0.7328	0.8765
S3	0.4990[a]	0.7455	0.8821	1.0182
S4	0.9782[a]	1.4906	1.7398	1.9702
S5	0.4392[a]	0.5341	0.5847	0.6351
S6	0.9788[a]	1.4877	1.7017	1.8762
S7	2.4662	1.7716	1.6230[a]	1.7003

[a] The smallest RMSD for each experimental data block.

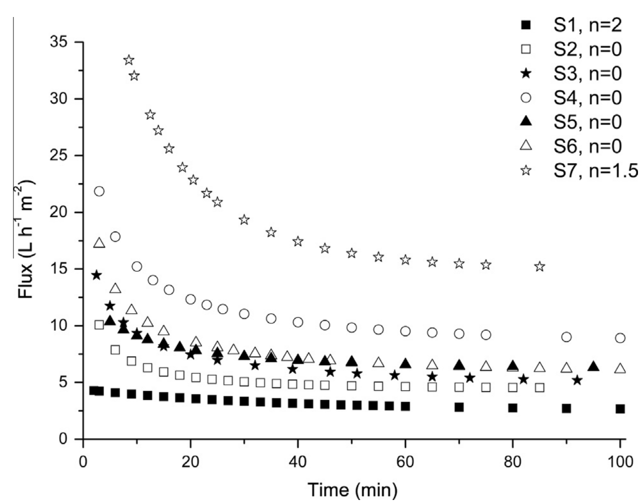

Fig. 6. Predicted fluxes according to the best adjusted fouling model.

in fruit juices essentially contributes to the cake formation during micro or ultrafiltration.

For the sample S7, the best adjustment was get when $n = 1.5$ (internal pore blocking), while the worst adjustment was for $n = 0$ (cake formation). The chitosan treatment reduced turbidity and colour of raw juice at greater levels. This removal probably reduced the amount of large particles, minimizing fouling occurrences due to cake formation with the predominant incidence of internal pore blocking.

Fig. 6 presents the calculated flux points for each sample when the best fouling model is applied. Comparison between Figs. 3 and 6 shows that experimental and calculated flux data are similar.

4. Conclusion

The chitosan addition showed to be a promising alternative for the pretreatment of passion fruit juice, achieving the highest reductions of colour and turbidity among all the evaluated pretreatments. Although enzyme addition is the most suitable process for viscosity reduction, the chitosan addition also promoted the reduction of the viscosity of passion fruit juice. The microfiltration of the sample treated with chitosan presented the highest permeate flux. However, all the proposed pretreatments presented a positive effect on the permeate flux for microfiltrations.

The applied pretreatment did not influence the quality of the obtained clarified juice, except for total soluble solids. The microfiltration process was able to reduce colour and turbidity of the fed juice, resulting in a visually clean product.

The Field model was suitable to describe major fouling mechanisms during the microfiltration of passion fruit juice. A slight predominance of total pore blocking was attributed to raw juice microfiltration. In centrifuged and enzymatic treated samples, the best adjustment was observed for cake formation. For the chitosan pretreated sample, internal pore blocking was found to be the major fouling factor. This affirms that pretreatment with chitosan removes large particles and minimizes the cake and gel layer formation during the microfiltration of passion fruit juice.

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