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Original

Clarification of pineapple juice by microfiltration

Clarificação de suco de abacaxi por microfiltração

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Abstract

In the present work, pineapple juice was first hydrolyzed with a commercial pectinase (Ultrazym 100 G) and then clarified by microfiltration. A tubular polyethersulfone membrane with an average cut-off of 0.3 μm and a total effective filtration area of 0.05 m^2 was applied. The transmembrane pressures were 1.5 and 3.0 bar, respectively, and the processes was conducted at room temperature. The results showed that the pineapple juice permeate fluxes were of 57.77 L/m^2/hours (1.5 bar) and 46.85 L/m^2/hours (3.0 bar). Concentration polarization and possibly fouling occurred during the processes. The best clarified juice fluxes were obtained when low transmembrane pressures (1.5 bar) were applied.
Keywords: *fruit juice; enzymatic hydrolysis; membrane processes.*

Resumo

No presente trabalho, suco de abacaxi foi inicialmente hidrolisado com enzima comercial (Ultrazym 100 G) e clarificado por microlfitração. Utilizou-se membrana tubular de polietersulfona com tamanho médio de poro de 0,3 μm e área total de filtração de 0,05 m^2. Foram aplicadas pressões de transmembrana de 1,5 e 3,0 bar, respectivamente, e os processos foram conduzidos à temperatura ambiente. Os resultados revelaram que os fluxos de suco permeado foram de 57,77 L/m^2/horas (1,5 bar) e 46,85 L/m^2/horas (3,0 bar). O fenômeno de polarização de concentração e o fouling foram observados durante os processos. Os melhores fluxos de suco clarificado foram obtidos utilizando-se a pressão de transmembrana menos elevada (1,5 bar).
Palavras-chave: *suco de fruta; hidrólise enzimática; processos com membranas.*

1 Introduction

The conventional fruit juices clarification process involves several steps to obtain a limpid juice. On the other hand, tangential filtration or crossflow filtration such as Microfiltration (MF) and Ultrafiltration (UF) can substitute this process. Microporous membranes of different materials, configuration, and cut-off (pore size) can be used. The feeding solution is applied in parallel to the membrane surface, and pressure is the principal driving force.

These processes have many advantages such as the possibility of operating at room temperature and, in addition, promoting cold commercial sterilization (CARVALHO et al., 2002, 2003; GIRARDI; FUKUMOTO, 2000; DRIOLI; ROMANO, 2004; YOUN et al., 2004). The limiting factors that affect tangential filtration are concentration polarization and the fouling that occur during the process (GEHLERT; LUQUE; BELFORT, 1998; JIRARATANANON; UTTAPAP; TANGAMORNSIKSUN, 1997; RIEDL et al., 1996; TODISCO et al., 1996; LOSANO et al., 1996; KIM; HUR; CHOI, 1995; FANE, 1994; SU; LIU; WILEY, 1993; PADILLA-ZACOUR; McLELLAN, 1989; SAURA et al., 1991; ITOUA-GASSAYE et al., 1991; CARNEIRO et al., 2002; VLADISAVLJEVI; VUKOSAVLJEVI; BUKVI, 2003; LÓPEZ et al., 2005; CASSANO; MARCHIO; DRIOLI, 2007; CASSANO et al., 2008). To minimize these problems, the enzymatic hydrolysis is recommended by many authors as a pretreatment for membrane processes (VAILLANT et al., 2001; PELEGRINE; VIDAL; GASPARETTO, 2000; TODISCO et al, 1996; PADILLA-ZACOUR; McLELLAN, 1993; HERNANDEZ et al., 1992; STUTZ, 1993; HASHIZUME; LATTIMER, 1974).

However, the use of certain enzymes such as lacase can confer negative characteristics such as turbidity increase (GOKMEN; BORNEMAN; NIJHUIS, 1998; STUTZ, 1993) after membrane processes. The UF and MF processes are used in juice clarification, but there are still unexplored clusters in the consumer market that can absorb them. Many authors have been studying the nutritional and sensorial characteristics as well as the microbiological quality of the juices before and after enzymatic hydrolysis (CARVALHO et al., 2006, CARVALHO; SILVA; PIERUCCI, 1998; JUAREZ; PAREDES-LOPEZ, 1994; MIETTON-PEUCHOT et al., 1990; PADILLA-ZACOUR; MCLELLAN, 1989; DUXBURY, 1986; HEATHERBELL; SHORT; STRUBI, 1977; MICHAELS, 1981).

Many volatile compounds can be lost during the process or remain in the retentate juice as well as in the concentration polarization layer of the membrane surface (CARVALHO; SILVA; ABADIO, 2002; 2008; CHAO; WEN; FANG, 1992; ITOUA-GASSAYE et al., 1991; RAO et al., 1987). The removal

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of compounds responsible for turbidity, such as starch, pectin, tannins (responsible for juice astringency) and others by UF/MF still promotes nutrients decrease in the permeated juices. Losses are normally verified according to the membrane cut-off and material, temperature, and transmembrane pressure. The purpose of this paper was to investigate the medium flux behavior of hydrolyzed pineapple juice using a 0.3 µm microfiltration polyethersulfone membrane in a tubular system.

2 Materials and methods

Pineapple (*Ananas comosus* L. Merril cv. Pearl) cultivated in the city of São Francisco de Itabapoama, Campos, in the State of Rio de Janeiro was used in this experiment.

The pasteurized pineapple juice was processed at CETEC-Food & Beverages, Vassouras, State of Rio de Janeiro. The crown was removed manually. The fruit was dipped in chlorinated water (15 ppm) for 10 minutes to remove dirtiness. The peeling was performed using previously cleaned steel knives, and the blanching was carried out in a crusher (Alfa Laval - Richmond, USA). The enzymatic inactivation (blanching) was performed at 85-90 °C for 2 minutes and the juice was filtered through sieves of 28; 20; 0.149, and 0.059 mm (Alfa Laval – finisher). Pasteurization was carried out at 85 °C for 2 minutes (retention time) and cooled at 45 °C. The juice was packed in 50; 5.0; 1.0, and 0.5 L PVC containers and stored at –20 °C.

The final yield of the pasteurized juice was calculated by the difference between the weight of the whole fruit (crown and peel) before the process and the weight of the final juice obtained.

Preliminary tests with 100 ppm of Ultrazym 100 G, a commercial pectinase from Novozymes (Zurich, Switzerland), for 30 minutes at 40 °C under constant shaking (100 rpm) and using Ultrazym 100 G (100 ppm/30 minutes) combined with 0.5% of Celluclast enzyme were performed to choose the best viscosity reduction (Tables 1 and 2).

Table 1. Viscosity (30 °C) of pasteurized pineapple juice and after enzymatic hydrolysis with Ultrazym 100 G.

Sample	Viscosity (mPa/second)	Viscosity decrease (%)
Pasteurized juice	9.5	-
30` 100 ppm	6.7	27.6
30` 300 ppm	7.4	22.1
90` 100 ppm	7.3	23.1
90` 300 ppm	8.8	7.5
75` 200 ppm	9.2	1.5

Table 2. Viscosity (30 °C) of pasteurized pineapple juice and after enzymatic hydrolysis with Ultrazym 100 G + Celluclast.

Sample	Viscosity (mPa/second)	Viscosity decrease (%)
Pasteurized juice	9.5	-
30` 100 ppm	7.5	21.1
30` 300 ppm	8.0	15.3
90` 100 ppm	7.7	19.2
90` 300 ppm	8.9	6.3
75` 200 ppm	9.1	3.8

The enzymatic hydrolysis of the pasteurized pineapple juice was carried out for each process to obtain enough mass once the feeding tank maximum capacity is 25 L.

An Armfield-multipurpose processing vessel Model-FF 40 (Armfield, Jackson, New Jersey, USA) homogenizer tank with juice at 40 °C was used under shaking, to which 100 ppm Ultrazym 100 G was added for 30 minutes. Next, the juice was frozen and stored at –20 °C.

The 20 L stainless steel vessels and the tank used for hydrolysis were first washed and sanitized with NaOH at 20%, chlorinated water at 200 ppm for 10 minutes, and rinsed with industrial water at 3 ppm.

The particle size reduction evaluation and its frequency were obtained by laser diffraction and reflection optical method in a particle analyzer (model Analysette 22, Fritsch GmbH, Klar-Oberstein, Germany). This method was used to verify the pasteurized pineapple juice particle reduction after hydrolysis. All experiments were performed in quadruplicate.

Before each alkaline/acid/alkaline microfiltration process, cleaning steps were applied to recover the membrane water flux. Distilled water was recirculated into the system to remove pulp and residues. Next, a chlorine-alkaline solution 1% (pH = 11) at 45 °C for 30 minutes and 1.5 bar transmembrane pressure was recirculated in the membrane system until rescovering of the initial water permeability The membrane was disinfected with hydrogen peroxide 1% followed by distilled water plus 1% formic aldehyde washing. At the end of the washing process, distilled water was applied again.

A tubular membrane (Koch Membrane Systems,Wichita, USA) with 0.3 µm pore size (polyethersulfone) and the effective filtration area of 0.05 m^2 in a pilot system were used. Transmembrane pressures of 1.5 and 3.0 bar were applied and the processes were conducted at room temperature (± 25 °C). Five microfiltration experiments in the pilot system were conducted for each transmembrane pressure applied in the same operational conditions.

The membrane water flux was obtained at room temperature (22-24 °C), at which the time (seconds) was measured in relation to the water volume (mL). The water fluxes were measured by $J = V/a.t$, where: J = permeate flux; V = volume of permeate water (L); a = membrane area (0.05 m^2); and t = time (hours).

Five experiments of each trasmembrane pressures (1.5 and 3.0 bar) were conducted at ± 25 °C.

The color instrumental analysis was performed using the S&M Color Computer-SM-4-CH from Suga, USA, in the Hunter System. The color parameters in relation to Petri plate were L, a, b, ΔE, and turbidity (haze).

3 Results and discussion

From 1840 kg of pineapple, 611 kg of juice were obtained. The juice yield was about 33.22% over the raw material, as follows:

- "In natura" gross weight = 1840 kg;
- Post fruit peeling net weight = 1262 kg; and
- Pineapple juice weight = 611 kg.

The first trial (Table 1) was chosen due to its better juice reduced viscosity (27.6%) and also to reduce costs with the enzymes.

The comparison between pasteurized and the hydrolyzed juice particle size reduction showed that there was more than 50% particle size reduction in the hydrolyzed pineapple juice.

In the pasteurized pineapple juice the particles size ranged from 0.16 to 0.32 μ, and the ones with 0.22 and 0.32 presented high frequency when compared with the particle size of the hydrolyzed juice, in which the high frequency ranged from 0.16 to 0.22 μm. The statistical results showed 6.163% Coefficient of Variation (VC), 16.113% Standard Deviation (SD), 8.25% (VC), and 12.113% (SD) for the hydrolyzed pineapple juices, respectively.

The mean fluxes of 3109.70; 4255.32, and 5341.25 L/m^2/hours were obtained with the 0.3 μm membrane at 1.5, 2.0 and 2.5 bar, respectively. After each process, it was observed that there was a decrease in the membrane permeability, which was restored after the cleaning procedure (Figure 1).

From 18 L of hydrolyzed pineapple juice used as feeding, 11.25 L of permeate juice and 6.75 L of retentate were obtained after 3 hours of process. The permeate juice recovery was about 62.5% and the volumetric concentration was 2.6. The medium flux after the process was 57.55 L/m^2/hours (Figure 2).

From 14.5 L of hydrolyzed pineapple juice used as the feeding system, 9.35 L of permeated and 5.15 L of retentate juice were obtained, which corresponds to 64.48% of recovery. The volumetric concentration was 2.8. The medium flux after 4 hours was 46.85 L/m^2/hours (Figure 2). The medium flux obtained with 0.3 μm operated at 3.0 bar was lower than that obtained when the system was operated at 1.5 bar. However, an increase in the flux was observed after 2.5 hours. The juice recirculation into the membrane system might have promoted (a self-cleaning effect at membrane surface level increasing the permeate flux. Chamchong and Noomhorm (1991) verified an increase in the flux with the increase in the membrane cut-off.

On the other hand, Tarlerton and Walkeman (1993) observed that there is little membrane cut-off influence on the flux as well as on the macromolecules rejection when most of the feeding particles are higher than the membrane cut-off and the permeate quality. The flux is normally lower when most of the particles have molecular weight value close the membrane cut-off.

According to Riedl, Lencki and Girard (1996), the polysulfone and nylon membranes promote more resistance to concentration polarization than that promoted by polyethersulfone and polyvinylidene fluoride membranes. The drastic reduction of particles size caused by enzymatic hydrolysis can promote a flux decline on permeate juice because of pore obstruction (fouling).

Lenggenhager and Lyndon (1998) also observed juice concentration polarization during clarification with polysulfone, polyethersulfone, and polyvinylidene fluoride membranes. However, Gehlert, Luque and Belfort (1998) found that tubular polyethersulfone membrane promotes the highest fluxes.

According to Youn et al. (2004) polysacharides, proteins and colloidal materials are present as solid materials in juices, which form crystal with the increment of concentration polarization or become a gel layer which, in turn, accumulates on the surface to form a secondary membrane as the filtration goes on. The solute adsorption inside and/or on the membrane may change the MWCO membrane characteristics increasing the membrane resistance

A slight decrease in luminosity was observed for the pasteurized (12.9) and hydrolyzed (13.6) pineapple juices. The highest turbidity was observed in the hydrolyzed juice (97.5), probably due to the increased number of particles in suspension after hydrolysis. As expected, the luminosity of the permeate juice increased and the turbidity was almost completely removed (Table 3).

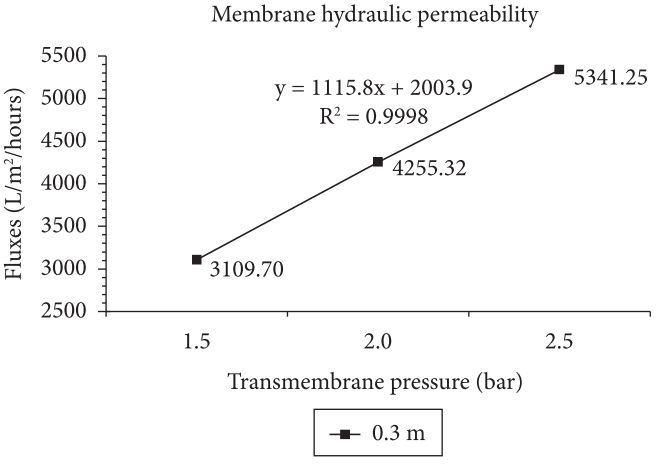

Figure 1. Hydraulic permeability of 0.3 μ polyethersulfone tubular membrane at various transmembrane pressures.

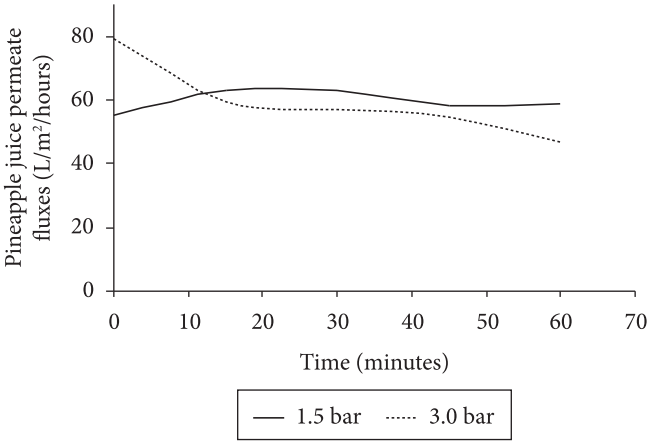

Figure 2. Pineapple juice fluxes at 1.5 and 3.0 bar.

Table 3. Pasteurized, hydrolyzed and permeated pineapple juices instrumental color, in relation to Petri plate.

Sample	TMP (bar)	Luminosity (L_{Hunter})	a_{Hunter}	b_{Hunter}	Haze
Pasteurized juice	-	12.9	0.2	6.7	97.3
Hydrolyzed juice (feed)	-	13.6	0.1	6.9	97.5
0.3 µ	1.5	97.9	–2.0	11.9	3.1
0.3 µ	3.0	98.7	–2.2	11.3	1.9

L_{Hunter} 0 = black and 100 = white; a_{Hunter} from –80 to zero = green; from zero to +100 = red; b_{Hunter} from –100 to zero = blue; from zero to +70 = yellow.

The luminosity was lower and, consequently, the turbidity of the permeated juice was higher, 1.5 bar (97.9 and 3.1, respectively), than when the system was operated at 3.0 bar (98.7 and 1.9, respectively) showing that the highest TMP (transmembrane pressures) promotes higher luminosity in the permeated juices.

Kim, Hur and Choi (1995) reported an increase of 90% in luminosity and a decrease in turbidity of sugar cane juice clarified by UF membranes.

4 Conclusions

The clarification of the pineapple juice by MF in a 0.3 µm polyethersulfone membrane was more effective when 1.5 bar TMP was applied, although concentration polarization and the fouling phenomenon occured during the process under both transmembrane pressures applied.

As expected, the luminosity of the permeate juice increased and the turbidity was almost completely removed.

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