

OBTAINING FRUCTOOLIGOSACCHARIDES FROM YACON (*Smallanthus sonchifolius*) BY AN ULTRAFILTRATION PROCESS

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Abstract - The objective of this study was to evaluate the separation of fructooligosaccharides (FOS) from yacon extract by an ultrafiltration process using membranes of 10 and 30 kDa. The total resistance (R_t), membrane resistance (R_m), fouling resistance (R_f), and concentration polarization (R_c) during the separation process were also assessed. The operating pressures were 1.2 and 0.75 bar for UF-10 and UF-30, respectively. The permeate flux increased upon increasing the pressure from 0.5 to 2 bar and the resistance values showed a slight increase with increasing pressure. The fouling percentages were 61.24% and 57.33% for the membranes UF-10 and UF-30, being reversible after the cleaning procedure with acidic and basic solution, resulting in high percentages of flux recovery of 76.46% and 83.56% for U-10 and UF-30, respectively. The FOS retention values were 24.48% and 6.49% for both membranes UF-10 and UF-30, corresponding to 24% and 18.4% purity.

Keywords: Yacon; Fructooligosaccharides; Ultrafiltration.

INTRODUCTION

Nowadays, the demand for foods that promote health and well-being is increasingly evident. Thus, food that was considered for many years as a source of nutrients needed to sustain life has become the object of studies related to disease prevention and improved function of organs and tissues.

In this context, yacon roots, which originated from Andean regions, contain from 60 to 70% inulin (Vilhena *et al.*, 2000). The native inulin is a mixture of oligomers and polymers containing long chain molecules of different degrees of polymerization (DP) from 2 to 60 units, with an average DP of about 12; the inulin present in yacon has low DP, between 3 and 10, therefore being considered fructooligosaccharides (FOS) (Goto *et al.*, 1995; Villegas and Costell, 2007).

According to Lewis (1993), FOS are defined as a combination of three sugar molecules: 1-kestose (GF2), nystose (GF3) and fructofuranosyl nystose (GF4) where the units fructosyltransferase (F) are combined with sucrose (GF) at the β position (2 \rightarrow 1). Compounds such as FOS may be used as sugar substitutes, as they are more soluble and sweeter than native and long-chain inulin (Meyer *et al.*, 2011).

FOS have been designated as prebiotics and soluble fiber, since they pass through the stomach and small intestine without being digested, so they provide no extra calories. However, they are fermented by bacteria, such as bifidobacteria, helping to increase the microflora that is related to improving the health of the colon, affecting the general welfare (Gibson and Roberfroid, 1995). For this reason, it is important to isolate these compounds in order to

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incorporate them for human consumption. An alternative is the membrane separation process (MSP) considered to be a clean technology. Ultrafiltration (UF) is a type of MSP considered to be a very attractive alternative method since it does not use heat and involves no phase change, which makes the concentration process more economical, with the advantage of not using chemicals, since only a driving force is required to separate the compounds of interest (Kamada *et al.*, 2002b).

UF membranes have pore diameters varying between 0.01 μm and 0.001 μm , and are capable of retaining and fractionating macromolecules such as lipids, proteins and colloids present in solution whose molecular weight varies from 300 to 500,000 Daltons (Da) (Mulder, 1996). These membranes can retain solutes of different molecular weights, usually being specified by a nominal molecular weight cut-off or MWCO, which establishes the lowest molecular weight that is retained with an efficiency of at least 95% (Modler, 2000).

The use of UF to separate inulin and FOS from yacon has been reported in several studies. Gibertoni *et al.* (2006) evaluated the production of rich fructans syrup from clarified yacon juice using a ceramic microfiltration membrane with nominal pore size of 0.14 μm , and UF membrane with molecular weight cut off of 50 kDa. Kamada *et al.* (2002a) evaluated the combination of ultrafiltration and nanofiltration on the purification and concentration of FOS from yacon roots, removing most of the mono- and disaccharides and obtaining a concentrated product with DP of three or more, and 98% purity.

The MSP have some limitations, highlighting the phenomena of concentration polarization and fouling at the membrane surface. The polarization occurs in the first minutes of the process when the solute is retained at the membrane surface, making difficult the solvent permeation and resulting in a permeation flux decline, which is a reversible phenomenon. In contrast, the fouling is characterized by a permeation flux decline resulting from clogging of the membrane pores, which can be an irreversible phenomenon depending on the situation (Wu *et al.*, 1990).

The resistance-in-series model has been used to evaluate fouling in the membrane separation process (Brião and Tavares, 2012). These resistances include membrane resistance, an external or reversible fouling, which consists of cake layer deposition and concentration polarization, and irreversible resistance (Bagci, 2014; Rezaei *et al.*, 2014). The latter is due to particle and macromolecule deposition and adsorption of smaller-sized solutes onto the membrane pore walls (Ng *et al.*, 2014; Rezaei *et al.*, 2014).

The aim of this study was to evaluate the separation of FOS from yacon extract using ultrafiltration as a membrane separation process. The parameters of total resistance, membrane resistance, fouling resistance, and concentration polarization during the separation process were also investigated.

MATERIAL AND METHODS

Raw Material

The yacon roots were cleaned and selected considering the absence of visual damage and infections, and peeled and sliced with a mean diameter of 4.55 ± 0.25 mm and a thickness of 1.75 ± 0.35 mm (Scher *et al.*, 2009) and subjected to a blanching treatment for 4 minutes at 100 °C, followed by cooling in an ice bath for 3 minutes (Fante and Noreña, 2012). The juice was extracted using a food processor. The remaining sugars were extracted from the pulp obtained after separation of the juice by addition of water at 80 °C in a 2:1 ratio (weight water/weight pulp), maintaining the mixture at $80 \text{ °C} \pm 2 \text{ °C}$ for one hour, according to the methodology proposed by Toneli *et al.* (2007). The yacon juice and the liquid solution obtained from the pulp were separately filtered under vacuum. The filtrates were mixed, which constituted the yacon extract, to which 1% (w/v) citric acid was added and filtered on Whatman filter paper N°. 01 to remove suspended solids.

Membrane Separation Process

Polyethersulfone UF membranes (Synder Filtration Headquarters, Vacaville, USA) with nominal molecular weight cut off (NMWCO) of 10.000 and 30.000 Daltons were used. UF experiments were performed under the conditions of the configuration shown in Figure 1, which comprises: (1) a feed jacketed glass vessel with a total volume of 2.0 Liters connected to an ultrasonic thermostatic bath (model CE-110/10, CienlaB, Brazil); (2) pneumatic diaphragm pump (model Ingersoll-Rand PD05P-ARS-PAA-B); (3) pre-filter; (4) AISI 316 stainless steel flat membrane module with area of 0.008118 m^2 ; (5) and (6) AISI 316 stainless steel pressure gauges, scale 0-12 bar; (7) pressure valves; (8) bypass valve. Prior to the UF process, the extract passed through a pre-filter with a nominal pore size of 5 μm .

Initially, the new membranes were compacted following the methodology described by Rai *et al.* (2007), which consists of the densification of the microstructure of the membranes using TMP of 2.5

bar higher than the working pressure. Then the water flux (J_p) was calculated.

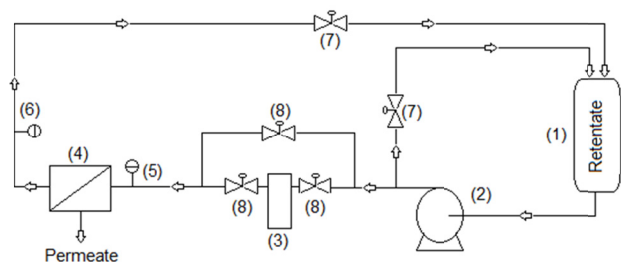


Figure 1: Schematic representation of the UF process. Legend: (1) feed tank, (2) pump, (3) pre-filter, (4) membrane module, (5) input manometer, (6) manometer output, (7) pressure control valve, (8) bypass valve.

The hydraulic permeability for the new membrane (L_{pi}) ($L \cdot m^{-2} \cdot h^{-1} \cdot bar^{-1}$) was calculated from the slope of the permeate flux as a function of transmembrane pressure. The process was conducted until the water permeation flux became constant with time, with volume measurements at intervals of 15 minutes, with a total process time of 150 minutes for both membranes.

The separation processes were performed in batch mode with complete recirculation of the retentate to the feed tank and permeate removal, as described by Kamada *et al.* (2002a). The permeate flux was measured every 15 minutes during the first hour and every 30 minutes in the following hours. The UF process lasted 6 hours for UF-10, and 5 hours and 30 minutes for UF-30. Finally, a cleaning procedure was realized using NaOH solution, $0.35 \text{ g} \cdot L^{-1}$, pH 10-10.5; citric acid solution, $5 \text{ g} \cdot L^{-1}$, pH 2.0.

The same procedure used to find (L_{pi}) was used to calculate the hydraulic permeability after the separation process of the yacon extract (L_{pf}) and after the cleaning procedures (L_{pc}).

Resistance Analysis

The decline of the permeate flux was analyzed by the resistance-in-series model (de Bruijn *et al.* 2002; Jiratananon and Chanachai, 1996). is the total resistance (m^{-1}), which can be expressed in terms of the other resistances according to Equation (1):

$$R_t = R_m + R_c + R_f \quad (1)$$

where R_m is the resistance of the new membrane with water (m^{-1}); R_c is the resistance of the concen-

tration polarization on the membrane surface (m^{-1}); and R_f is the fouling resistance (m^{-1}). These resistances were calculated by using the values of hydraulic permeability, in accordance with the equations described by Cassano *et al.* (2007):

$$R_m = \frac{1}{\mu_w L_{pi}} \quad (2)$$

$$R_t = \frac{1}{\mu_w L_{pf}} \quad (3)$$

$$R_m + R_f = \frac{1}{\mu_w L_{pc}} \quad (4)$$

where μ_w is the water viscosity (Pa.s).

The resistance R_c was calculated from the difference between the other resistances, using Equation (1). R_c and R_f are also denominated reversible and irreversible fouling resistances, respectively (Rezaei *et al.*, 2014).

Membrane Fouling, Cleaning Efficiency Protocol and Membrane Retention

Both membrane fouling and flux recovery were calculated by Equations (5) and (6) according to Saha *et al.* (2007) and Liikanen *et al.* (2002), respectively:

$$\text{Fouling} = \left(\frac{L_{pi} - L_{pf}}{L_{pi}} \right) \times 100 \quad (5)$$

$$\text{Flux recovery} = \left(\frac{J_{pc}}{J_{pi}} \right) \times 100 \quad (6)$$

where J_{pc} is the water permeation flux after the cleaning procedure ($L \cdot m^{-2} \cdot h^{-1}$) and J_{pi} is the water permeation flux of the new membrane ($L \cdot m^{-2} \cdot h^{-1}$). Both flows were measured at the same pressure.

The observed retention (%) was calculated according to Equation (7) as described by Cissé *et al.* (2011):

$$R_{obs} = \left[1 - \frac{C_p}{C_r} \right] \times 100 \quad (7)$$

where C_p is the permeate concentration of a given sugar accumulated in the tank (g.L^{-1}) and C_r is the retentate concentration of the same sugar (g.L^{-1}) at the end of the process. The R_{obs} value varies from 100% (complete retention of solute) to 0% (solute and solvent pass freely through the membrane).

Sugar Assays

For determination of inulin, glucose and fructose contents, the samples were defrosted, filtered through $0.22 \mu\text{m}$ membrane and placed in a sonicator for 3 minutes prior to HPLC analysis. Analyses were performed by the method described by Zuleta and Sambucetti (2001) with adaptations, which consisted of direct determination by High Performance Liquid Chromatography (HPLC-RI), using a Perkin Elmer series 200 chromatograph with refractive index detector, Milli-Q water as mobile phase at a flow rate of $0.6 \text{ mL} / \text{min}$, temperature of $80 \text{ }^\circ\text{C}$, column Phenomenex Rezex RHM Monosaccharide, $300 \times 7.8 \text{ mm}$, and total run time of 14 minutes. The procedure was performed in duplicate for each sample.

Statistical Analyses

Data were assessed by ANOVA and Tukey's test for multiple comparisons between means, using SAS 9.3 (SAS Institute Inc.).

RESULTS AND DISCUSSION

Sugar Concentration in the Juice, Pulp and Yacon Extract

The sugar content of yacon juice was $5.42 \pm 0.01 \text{ g}/100 \text{ g (dm)}$ for inulin, $4.74 \pm 0.01 \text{ g}/100 \text{ g (dm)}$ for glucose, and $11.00 \pm 0.03 \text{ g}/100 \text{ g (dm)}$ for fructose. Lago *et al.* (2012) found sugar concentration values of 1.07 ± 0.18 , 3.30 ± 0.28 and $2.99 \pm 0.18 \text{ g}/100 \text{ g (dm)}$ for inulin, glucose and fructose in yacon juice, and 3.15 ± 0.16 , 10.98 ± 0.32 and $4.30 \pm 0.57 \text{ g}/100 \text{ g (dm)}$ for inulin, glucose and fructose in the pulp. Nieto (1991) studied 10 yacon species and obtained values of 2.47, 1.63, and $2.51 \text{ g}/100 \text{ g}$ for fructose, glucose and sucrose.

The solution extracted from the pulp presented 1.41 ± 0.00 , 2.57 ± 0.09 , $3.17 \pm 0.02 \text{ g}/100 \text{ g (dm)}$ inulin, glucose and fructose, respectively. The final yacon extract containing yacon juice and pulp leach

solution had a final composition of 8.21 ± 0.01 , 11.36 ± 0.08 , $16.44 \pm 0.07 \text{ g}/100 \text{ g (dm)}$ of inulin, glucose and fructose, respectively.

Ultrafiltration Process

Initially, the experiments were performed by compaction tests and, after that, measurements of water permeation flux as a function of transmembrane pressure were realized. The permeation flux increased linearly with increasing pressure for both membranes, as shown in Figure 2. The values of hydraulic permeability with water were $12.05 \text{ L.m}^{-2}.\text{h.bar}^{-1}$ and $22.45 \text{ L.m}^{-2}.\text{h.bar}^{-1}$ for the membranes UF-10 and UF-30. This difference in permeability is due to the increased water flux of the membrane UF-30 as compared to membrane UF-10, whose performance is explained by the larger pore size of the first membrane, since the larger the pore size the higher the flux under the same processing conditions. Recirculation flow was 26.5 L.h^{-1} and it was the same for all pressure conditions. Saha *et al.* (2007) found that the hydraulic permeability values for a new membrane using a pressure of 1 bar increased from $38 \text{ L.m}^{-2}.\text{h.bar}^{-1}$ to $134 \text{ L.m}^{-2}.\text{h.bar}^{-1}$ by changing the membrane UF-30 for UF-50. The same effect was observed by Cissé *et al.* (2011) using membranes from 5 to 150 kDa and a pressure of 5 bar.

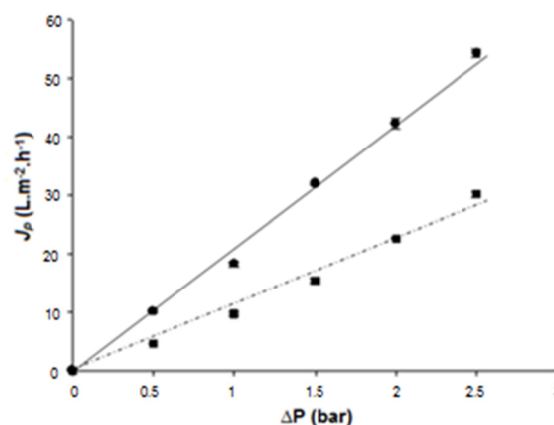


Figure 2: Permeate flow of water as a function of transmembrane pressure at $25 \text{ }^\circ\text{C}$. UF-10 (■); UF-30 (●).

Effect of Pressure on Permeate Flux of Yacon Extract

Figure 3 shows the graphs of J_p versus operation time for the membranes UF-10 and UF-30 at pressures from 0.5 to 2 bar. According to Rai *et al.* (2007), each curve can be divided into two stages, the first corresponding to a rapid decline of the permeate

flux, which can be attributed to the effect of the concentration polarization that results in the reduction of the effective driving force (Cheryan, 1998; Reis and Zydnei, 1999). The thickness of the layer also increases with time, thus increasing the resistance to mass transfer, as shown by the decrease in permeate flux (Benhabiles *et al.*, 2013). In addition, the flux decrease was higher with increasing pressure, and varied from 26.6 to 39.6% and 10.2 to 61% as compared to the initial flux of the membranes UF-10 and UF-30, respectively. The second stage is characterized by a less pronounced decrease of the flux until reaching a steady-state. In this condition, in accordance with Arthanareeswaran *et al.* (2010), the steady-state permeate flux increased with increasing pressure values. Mulder (1996) reported that the concentration polarization influences the retention of the solute during the separation of mixtures containing macromolecular solutes, where the polarization has decisive influence on the selectivity of the process. The particles with higher molecular weight retained by the membrane eventually form a layer on the surface thereof, which retains a larger number of solutes having smaller molecular weights. According to Jiratananon and Chanachai (1996) and Mondal *et al.* (2011), an increase in recirculation flow reduced concentration polarization, enhanced the mass transfer coefficient and increased the permeation flux. This way, an increase in tangential velocity means a decline of the relative participation of cake formation in membrane fouling, because the higher tangential velocity removes a considerable part of the deposited cake due to the increased shear (de Bruijn and Bórquez, 2006). On the other hand, when the transmembrane pressure increases and the tangential velocity is maintained constant, as in our case, de Bruijn and Bórquez (2006) mention that both blocking of the pore entrance with or without superposition of solute and blocking by a cake layer are almost constant during UF, with internal pore blocking dominating over cake formation.

Figure 4 shows the effect of TMP on the steady-state permeate flux for membranes UF-10 and UF-30. The permeate flux showed a linear increase at lower TMP pressures, while at higher pressures, the permeate flux values were close to a threshold value (J_{lim}), regardless of further increases in pressure values. The point at which the pressure is clearly independent is considered to be the optimum ΔP , which is the limiting flux that can minimize both fouling and the tendency to concentration polarization (Baker, 2004). For membrane UF-10 under pressure values above 1.5 bar, the permeate flux of yacon extract was independent of pressure, indicating that the limiting flux

was reached, whereas for the membrane UF-30, the limiting flux was reached at a pressure of 1 bar. Similar observations were realized by Rektor *et al.* (2004) and Cassano *et al.* (2008).

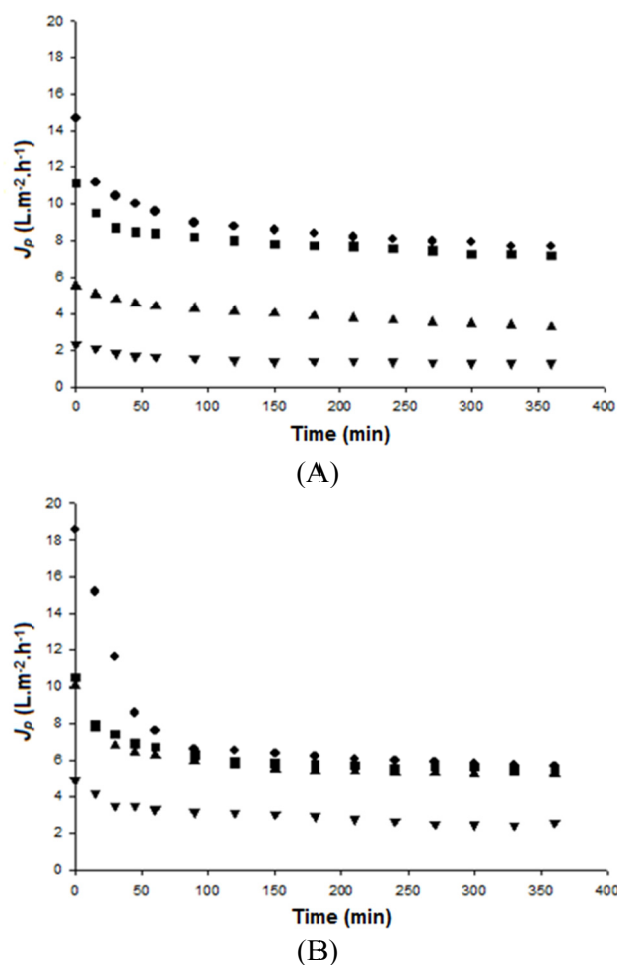


Figure 3: Permeate flow of yacon extract a function of time at 25 °C at different pressures using the UF-10 membrane (A), UF-30 membrane (B), (∇) 0.5 bar, (\blacktriangle) 1 bar, (\blacksquare) 1.5 bar, (\bullet) 2 bar.

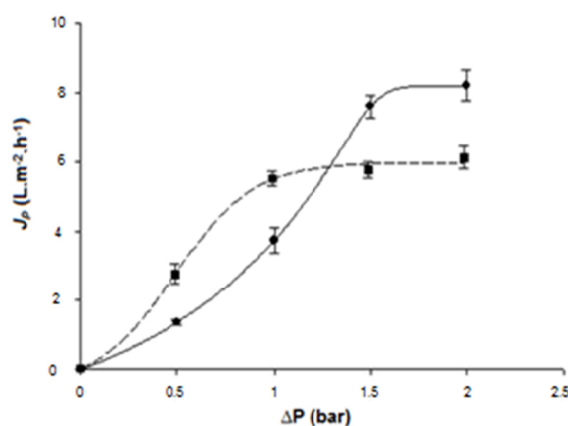


Figure 4: Effect of pressure on permeate flow of yacon extract in the steady-state at 25 °C. UF-10 (\bullet), UF-30 (\blacksquare).

According to Habert *et al.* (2006), from a practical point of view, any membrane system should be operated at pressures lower than those values that may lead to a limiting flux, in order to minimize the effects of fouling and concentration polarization (Cassano *et al.*, 2008). Following this line, the transmembrane pressure chosen for the UF process was 1.2 bar and 0.75 bar for the membranes UF-10 and UF-30, respectively, since these pressure values provided the most acceptable permeate fluxes. In these conditions, the permeate flux also decreased with time, from 5.9 to 3.4 L.m⁻².h⁻¹ and from 6.9 to 4.1 L.m⁻².h⁻¹ for UF-10 and UF-30, respectively, due to the boundary-layer phenomenon, where an increased concentration of solute near the membrane surface (concentration polarization) took place. Moreover, the accumulation of macromolecules in the membrane pores (membrane fouling) could also have contributed to decreasing flow.

Effect of Fouling on Permeate Flux of Yacon Extract

A major limitation of the membrane separation process is fouling. It may decrease permeate flux, increase energy consumption, and lead to frequent cleaning procedures or replacement of membranes (Liao *et al.*, 2008).

The membrane fouling and concentration polarization are limiting factors for MSP, since there is a decrease in permeate flux with time caused by accumulation of components from the feed solution in the membrane pores and on the surface (Czekaj *et al.*, 2001). Figure 5 shows the water permeate flux before (J_{pi}) and after (J_{pf}) the ultrafiltration process, and after the cleaning procedures (J_{pc}) for the membranes UF-10 and UF-30. From this figure the hydraulic permeabilities with water were calculated. For the membrane UF-10, the values were 12.05, 4.67 and 9.12 L.m⁻².h.bar⁻¹, respectively, for the water permeation flow of the new membrane, and after the experiments and cleaning procedures. For the membrane UF-30, these values were 22.45, 9.58 and 18.76 L.m⁻².h.bar⁻¹, respectively.

Habert *et al.* (2006) reported that the hydraulic permeability of the membranes with pure water must be monitored before and after the process to check if there was adequate cleaning and ensure the membrane integrity. A sharp decline in permeability was observed, indicating that the membrane suffered severe changes during the process.

The membrane fouling estimated from the values of hydraulic permeability of the permeate fluxes

were 61.24% (UF-10) and 57.33% (UF-30). The main phenomena contributing to fouling include pore blocking, particle adsorption on the membrane surface (cake) and / or inside its pores due to the interaction between the solute in the feed solution and the membrane material, and formation of a gel layer, resulting in high concentrations of solute at the membrane surface (Liao *et al.*, 2004). Hasan *et al.* (2013) indicated that the phenomenon of pore blocking is assumed to occur during the first moments of filtration and its effects are included in the membrane resistance. Therefore, the consequences of fouling are an increased resistance to membrane separation, decreased efficiency and / or changes in membrane selectivity. This affects the separation performance of the target species, with a consequent low product recovery (Li *et al.*, 2010).

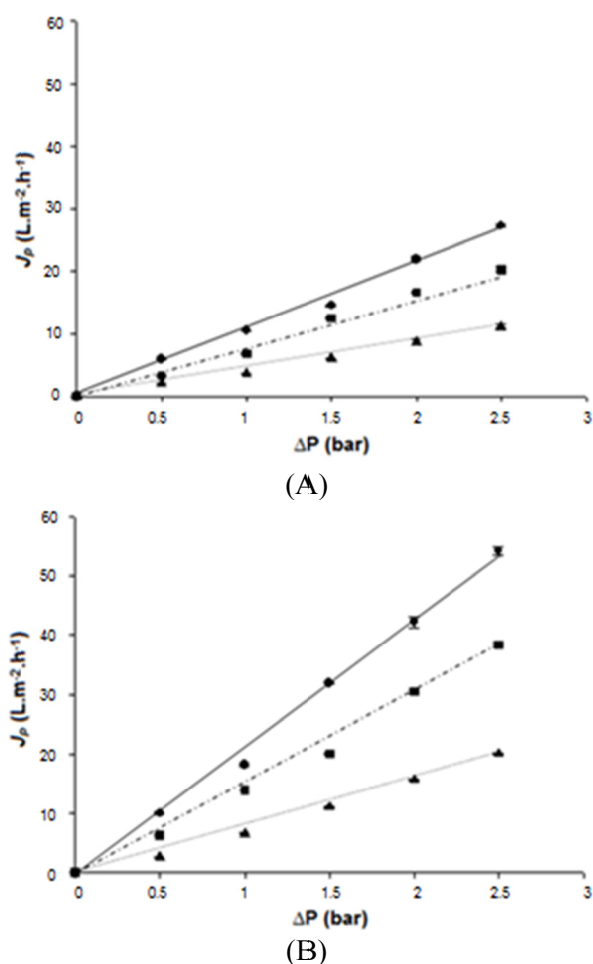


Figure 5: Permeate flow of water as a function of transmembrane pressure at 25 °C for membranes UF-10, 1.2 bar (A); UF-30, 0.75 bar (B) (●) Permeate flow of water in the new membrane after compaction (J_{pi}), (■) Permeate flow of water after the cleaning procedure (J_{pc}), (▲) Permeate flow of water after ultrafiltration of the extract of yacon (J_{pf}).

Saha *et al.* (2007) studied sugarcane juice in a UF system, and obtained fouling values of 52.63% for a 30 kDa membrane, and 42.60% for a 50 kDa membrane. The authors concluded that sugarcane juice contained various macromolecules that caused rapid clogging of pores and increased fouling, with significant flow reduction.

After the cleaning procedure, the flux recoveries were 76.46% and 83.56% for the membranes UF-10 and UF-30, respectively. From these results, it was possible to restore the permeate flux and reuse both membranes in a new process with the yacon extract. Rodrigues *et al.* (2003) studied banana juice clarification using membranes of 10 and 30 kDa, and reported that, after the cleaning procedure with NaOH solution (pH 10) and 0.8% NaClO solution for 2 hours, a flux recovery of about 95% was observed for the membrane UF-10 and 75% for the UF-30. Souza and Quadri (2013) mentioned that the efficiency of polymeric membranes decreases with time due to chemical degradation, fouling, thermal instability, low fluxes and compaction, as well as the occurrence of swelling phenomena.

Effect of Operating Conditions on Resistances

Figure 6 shows the effect of TMP on the total resistance, membrane resistance, fouling resistance, and polarization resistance for the membranes UF-10 and UF-30.

The membrane resistances increased slightly with increasing pressure, with values of $3.25 \times 10^{13} \text{ m}^{-1}$ for the lowest pressure and $3.41 \times 10^{13} \text{ m}^{-1}$ for a pressure of 2 bar using the membrane UF-10, whereas this value increased from 1.69×10^{13} to $1.89 \times 10^{13} \text{ m}^{-1}$ for the membrane UF-30. This effect was also observed by Gökmen and Cetinkaya (2007) using a membrane with molecular weight cut off of 10 kDa for ultrafiltration of apple juice. Although R_m values showed little change when the pressure increased from 1 to 4 bar, changing from 0.83×10^{13} to $1.24 \times 10^{13} \text{ m}^{-1}$, they decreased with increasing membrane molecular weight cut off. Wan *et al.* (2012) found that R_m decreased from $8.40 \times 10^{13} \text{ m}^{-1}$ using a 1 kDa membrane to $2.47 \times 10^{13} \text{ m}^{-1}$ using a 10 kDa membrane.

The increase in resistance values with increasing pressure can be attributed to the increased convective flow of solute into the membrane with increasing pressure. The increase in R_t leads to a more pronounced concentration polarization, causing an increase in R_c (Li and Chen, 2010). Labbe *et al.* (1990) reported that, at higher pressures, more solutes such

as sugars and acids passed through the membrane pores, increasing fouling resistance R_f . Thus, the reversible resistance was the main resistance during all filtration. Rezaei *et al.* (2014) demonstrated that the concentration polarization and cake resistances are much more important for the total resistance than other effects. On the other hand, irreversible resistance is caused by strong adsorption onto the membrane surface and the intensity of this phenomenon depends on the interactions between solute and membrane (Brião and Tavares, 2012).

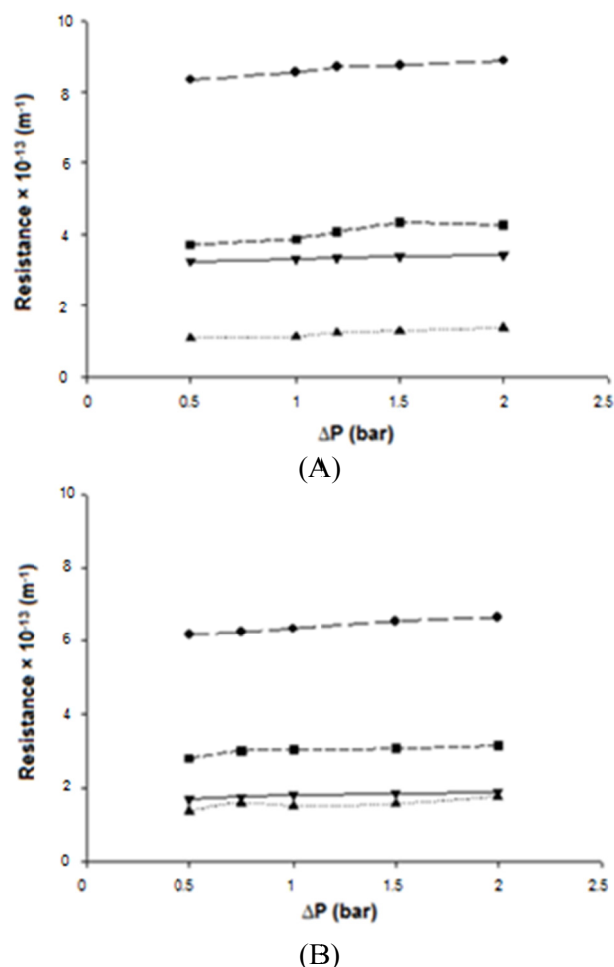


Figure 6: Effect of the pressure on the transmembrane pressure resistance at 25 °C for membranes UF-10, 1.2 bar (A); UF-30, 0.75 bar (B), (\bullet) R_t (\blacksquare) R_c (\blacktriangledown) R_m (\blacktriangle) R_f .

Saccharides Retention and Purity

The UF membranes used in this study were able to retain the FOS according to their pore size. The highest R_{obs} for FOS was reached with the membrane UF-10 (24.48%) as compared to the membrane UF-30 (6.49%). This difference was expected due to differences in the molecular weight cut off. Similar

behavior was observed for glucose (12.95 and 9.11%) and fructose (22.18 and 11.31%). Kuhn *et al.* (2010) emphasized that the simple sugars and FOS have a molar mass in Daltons smaller than the pore size of the membranes UF-10 and UF-30 (glucose and fructose: 180; sucrose: 342; 1-kestose (GF2) 504, nystose (GF3): 666 and fructofuranosilnystose (GF4): 828, FG5: 1080; GF6: 1260; GF7 1440; GF8: 1620; GF9: 1800), which allowed them to pass through the pores and be collected in the permeate.

The degree of the purity of the FOS in the permeate was 24.08% and 18.43% for UF-10 and UF-30, respectively. This degree was obtained from the relationship between FOS and sugar total concentration into the permeate. The degree of purity in the juice and yacon extract was 25.61% and 22.79%, respectively. It can be noticed that the enrichment in FOS versus simple sugars was not as high as desirable. A principal reason is that the membranes have a pore size distribution which does not allow effective separation of FOS from glucose and fructose molecules. Zhu *et al.* (2012) reported that the major FOS in yacon are 1-kestose (GF2), nystose (GF3) and 1-fructofuranosyl nystose (GF4) whose content in oligosaccharides were 12.29%, 12.17%, 6.20%, respectively. The molecular weights of these FOS are less than 1 kDa (Kuhn *et al.*, 2010). For this reason it is necessary to include in this process the separation by nanofiltration. Kamada *et al.* (2002a) used the combination of NF-UF to purify FOS present in a mixture of sugars derived from yacon and observed retention of 54.8% and the retention values of 14.0% for monosaccharides, 46.2% for disaccharides, 80.9% for trisaccharides and from 91.5 to 99.9% for sugars with DP \geq 4, using a 1 kDa membrane and pressure of 5 bar.

CONCLUSIONS

The effects of pressure on the total resistance, membrane resistance, fouling resistance and polarization resistance were evaluated using polyethersulfone membrane with nominal molecular weight cut offs of 10 and 30 kDa. It was observed that the increase in pressure resulted in a slight increase in all resistance values.

It was found that, for both membranes, the reversible resistance was the dominant resistance which reduced the permeate flux, while irreversible fouling resistance gave less of a contribution.

A good restoration of the hydraulic permeability was obtained after the cleaning procedure with acidic and basic solution.

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REFERENCES

- Arthanareeswaran, G., Mohan, D. and Raajenthiren, M., Preparation, characterization and performance studies of ultrafiltration membranes with polymeric additive. *Journal of Membrane Science*, 350, 130-138 (2010).
- Bagci, P. O., Effective clarification of pomegranate juice: A comparative study of pretreatment methods and their influence on ultrafiltration flux. *Journal of Food Engineering*, 141, 58-64 (2014).
- Baker, R. W., *Membrane Technology and Applications*. 2nd Ed., John Wiley and Sons, England (2004).
- Benhabiles, M. S., Abdi, N., Drouiche, N., Lounici, H., Pauss, A., Goosen, M. F. A. and Mameri, N., Protein recovery by ultrafiltration during isolation of chitin from shrimp shells *Parapanaeus longirostris*. *Food Hydrocolloids*, 32, 28-34 (2013).
- Brião, V. B. and Tavares, C. R. G., Pore blocking mechanism for the recovery of milk solids from dairy wastewater by ultrafiltration. *Brazilian Journal of Chemical Engineering*, 29, 393-407 (2012).
- Cassano, A., Conidi, C. and Drioli, E., Clarification and concentration of pomegranate juice (*Punica granatum* L.) using membrane processes. *Journal of Food Engineering*, 107, 366-373 (2011).
- Cassano, A., Mecchia, A. and Drioli, E., Analyses of hydrodynamic resistances and operating parameters in the ultrafiltration of grape must. *Journal of Food Engineering*, 89, 171-177 (2008).
- Cassano, A., Donato, L. and Drioli, E., Ultrafiltration of kiwifruit juice: Operating parameters, juice quality and membrane fouling. *Journal of Food Engineering*, 79, 613-621 (2007).
- Cheryan, M., *Ultrafiltration and Microfiltration Handbook*. Lancaster, Technomic Publishing Company (1998).
- Cissé, M., Vaillant, F., Pallet, D. and Dornier, M., Selecting ultrafiltration and nanofiltration membranes to concentrate anthocyanins from roselle extract (*Hibiscus sabdariffa* L.). *Food Research International*, 44, 2607-2614 (2011).
- Czekaj, P., López, F. and Güell, C., Membrane fouling by turbidity constituents of beer and wine: Characterization and prevention by means of infrasonic pulsing. *Journal of Food Engineering*, 49, 26-36 (2001).

- de Bruijn, J. and Bórquez, R., Analysis of the fouling mechanisms during cross-flow ultrafiltration of apple juice. *LWT - Food Science and Technology*, 39, 861-871 (2006).
- de Bruijn, J., Venegas, A. and Borquez, R., Influence of crossflow ultrafiltration on membrane fouling and apple juice quality. *Desalination*, 148, 131-136 (2002).
- Fante, L. and Noreña, C. P. Z., Enzyme inactivation kinetics and colour changes in Garlic (*Allium sativum* L.) blanched under different conditions. *Journal of Food Engineering*, 108, 436-443 (2012).
- Gibertoni, C. F., Nogueira, A. M. P. and Venturini, W. G., Ultra e micro filtração de suco de yacon (*Polymnia sonchifolia*) na obtenção de xarope rico em frutanos. *Revista Raízes e Amidos Tropicais*, 2, 68-81 (2006). (In Portuguese).
- Gibson, R. and Roberfroid, M., Dietary modulation of the human colonic microbiota: Updating the concept of prebiotics. *Journal of Nutrition*, 125, 1401-1412 (1995).
- Gökmen, V. and Cetinkaya, O., Effect of pretreatment with gelatin and bentonite on permeate flux and fouling layer resistance during apple juice ultrafiltration. *Journal of Food Engineering*, 80, 300-305 (2007).
- Goto, K., Fukai, K., Hikida, J., Nanjo, F. and Hara, Y., Isolation and structural analysis of oligosaccharides from yacon (*Polymnia sonchifolia*). *Bio-science Biotechnology Biochemistry*, 59, 2346-2347 (1995).
- Habert, A. C., Borges, C. P. and Nobrega, R., Processo de Separação por Membranas. Editora E-papers, Rio de Janeiro (2006). (In Portuguese).
- Hasan, A., Peluso, C. R., Hull, T. S., Fieschko, J. and Chatterjee, S. G., A surface-renewal model of cross-flow microfiltration. *Brazilian Journal of Chemical Engineering*, 30, 167-186 (2013).
- Jiratananon, R. and Chanachai, A., A study of fouling in the ultrafiltration of passion fruit juice. *Journal of Membrane Science*, 111, 39-48 (1996).
- Kamada, T., Nakajima, M., Nabetani, H. and Iwamoto, S., Pilot-scale study of the purification and concentration of non-digestible saccharides from yacon rootstock using membrane technology. *Food Science and Technology Research*, 8, 172-177 (2002a).
- Kamada, T., Nakajima, M., Nabetani, H., Saglam, N. and Iwamoto, S., Availability of membrane technology for purifying and concentrating oligosaccharides. *European Food Research and Technology*, 214, 435-440 (2002b).
- Kuhn, R. C., Filho, F. M., Silva, V., Palacio, L., Hernandez, A. and Pradanos, P., Mass transfer and transport during purification of fructooligosaccharides by nanofiltration. *Journal of Membrane Science*, 365, 356-365 (2010).
- Labbe, J. P., Quemeiras, A., Michel, F. and Daufin, G., Fouling of inorganic membranes during whey ultrafiltration: Analytical methodology. *Journal of Membrane Science*, 51, 293-307 (1990).
- Lago, C. C., Bernstein, A., Brandelli, A. and Noreña, C. Z., Characterization of powdered yacon (*Smallanthus sonchifolius*) juice and pulp. *Food and Bioprocess Technology*, 5, 2183-2191 (2012).
- Lewis, D. H., Nomenclature and diagrammatic representation of oligomeric fructans. A paper for discussion. *New Phytologist*, 124, 583-594 (1993).
- Li, H. and Chen, V., Membrane Fouling and Cleaning in Food and Bioprocessing. In: Cui, Z. F. and Muralidhara, H. S. *Membrane Technology. A Practical Guide to Membrane Technology and Applications in Food and Bioprocessing*, Chapter 10, Elsevier (2010).
- Liao, B. Q., Bagley, D. M., Kraemer, H. E., Leppard, G. G. and Liss, S. N., A review of biofouling and its control in membrane separation bioreactors. *Water Environmental Research*, 76, 425-436 (2004).
- Liikanen, R., Yli-Kuivila, J. and Laukkanen, R., Efficiency of various chemical cleanings for nanofiltration membrane fouled by conventionally-treated surface water. *Journal of Membrane Science*, 195, 265-276 (2002).
- Meyer, D., Bayarri, S., Tárrega, A. and Costell, E., Inulin as texture modifier in dairy products. *Food Hydrocolloids*, 25, 1881-1890 (2011).
- Modler, W. H., Milk Processing. In: Shuryo, N. and Modler, W. H. *Food Protein Processing Applications*. 1st Ed., Wiley, New York (2000).
- Mondal, S., Cassano, A., Tasselli, F. and De, S., A generalized model for clarification of fruit juice during ultrafiltration under total recycle and batch mode. *Journal of Membrane Science*, 366, 295-303 (2011).
- Mulder, M., *Basic Principles of Membrane Technology*. 2nd Ed., Kluwer Academic Publishers Group, Netherlands (1996).
- Ng, C. Y., Mohammad, A. W., Ng, L. Y. and Jahim, J. M., Membrane fouling mechanisms during ultrafiltration of skimmed coconut milk. *Journal of Food Engineering*, 142, 190-200 (2014).
- Nieto, C. C., Agronomical and bromatological studies in jicama (*Polymnia sonchifolia* Poep et Endl.). *Archivos Latinoamericanos de Nutrición*, 41, 213-221 (1991).
- Rai, P., Majumdar, G. C., Das Gupta, S. and De, S., Effect of various pretreatment methods on permeate

- flux and quality during ultrafiltration of mosambi juice. *Journal of Food Engineering*, 78, 561-568 (2007).
- Reis, R. and Zydnei, A., Protein Ultrafiltration. In: Flickinger, M. C. and Drew, S. W. (Eds.). *Encyclopedia of Bioprocess Technology: Fermentation, Biocatalysis, and Bioseparation*, New York, John Wiley and Sons (1999).
- Rektor, A., Pap, N., Kókai, Z., Szabó, R., Vatai, G. and Békássy-Molnár, E., Application of membrane filtration methods for must processing and preservation. *Desalination*, 162, 271-277 (2004).
- Rezaei, H., Ashtiani, F. Z. and Fouladitajar, A., Fouling behavior and performance of microfiltration membranes for whey treatment in steady and unsteady-state conditions. *Brazilian Journal of Chemical Engineering*, 31, 503-518 (2014).
- Rodrigues, S. L. C., Moreira, R. L. S., Cardoso, M. H. and Merçon, F., Avaliação de parâmetros de ultrafiltração de suco de banana. *Ciência e Tecnologia de Alimentos*, 2, 98-101(2003). (In Portuguese).
- Saha, N. K., Balakrishnan, M. and Ulbricht, M., Sugarcane juice ultrafiltration: FTIR and SEM analysis of polysaccharide fouling. *Journal of Membrane Science*, 306, 287-297 (2007).
- Scher, C. F., Rios, A. O. and Noreña, C. P. Z., Hot air drying of yacon (*Smallanthus sonchifolius*) and its effect on sugar concentrations. *International Food Science and Technology*, 44, 2169-2175 (2009).
- Souza, V. C. and Quadri, M. G. N., Organic-inorganic hybrid membranes in separation processes: A 10-year review. *Brazilian Journal of Chemical Engineering*, 30, 683-700 (2013).
- Toneli, J. T. C. L., Mürr, F. E. X., Martinelli, P., DalFabbro, I. M. and Park, K. J., Optimization of a physical concentration process for inulin. *Journal of Food Engineering*, 80, 832-838(2007).
- Vilhena, S. M. C., Câmara, F. L. A. and Kakihara, S. T., O cultivo de yacon no Brasil. *Horticultura Brasileira*, 18, 5-8 (2000). (In Portuguese).
- Villegas, B. and Costell, E., Flow behaviour of inulin-milk beverages. Influence of inulin average chain length and of milk fat content. *International Dairy Journal*, 17, 776-781 (2007).
- Wan, Y., Prudente, A. and Sathivel, S., Purification of soluble rice bran fiber using ultrafiltration technology. *LWT - Food Science and Technology*, 46, 574-579 (2012).
- Wu, M. L., Zall, R. R. and Tzeng, W. C., Microfiltration and ultrafiltration comparison of apple juices clarification. *Journal of Food Science*, 55, 1162-1163 (1990).
- Zhu, Z. Y., Lian, H. Y., Si, C. L., Liu, Y., Liu, N., Chen, J., Ding, L. N., Yao, Q. and Zhang, Y., The chromatographic analysis of oligosaccharides and preparation of 1-kestose and nystose in yacon. *International Journal of Food Science and Nutrition*, 63, 338-42 (2012).
- Zuleta, A. and Sambucetti, M., Inulin determination for food labeling. *Journal of Agricultural and Food Chemistry*, 49, 4570-4572 (2001).