International Journal of Engineering Research & Technology (IJERT)
ISSN: 2278-0181
Vol. 3 Issue 9, September- 2014

Analysis of Clarification Process of Purple Cactus Pear (Opuntia Ficus Indica) Juice by Ultrafiltration

Roberto Castro-Muñoz, Carlos Orozco-Álvarez and Jorge Yañez-Fernández*.
Departamento de Biotecnologia Alimentaria.
Instituto Politécnico Nacional, Unidad Profesional Interdisciplinaria de Biotecnología.
Av. Acueducto s/n, Col. Barrio La Laguna,
Ticomán, Mexico City, Mexico.

Abstract— **Juice rich in betalains (with 62.69 mg /L) and with a high antioxidant activity (14.35 TEAC) from purple cactus pear was submitted to clarification process by membrane technology. It was analyzed the viability of the process in terms of productivity (flux permeate of 22.94 L/ m^2 h^{-1}), fouling index (53.40 %), cleaning efficiency (100%) by batch concentration configuration. The juice quality was conserved due to there were no changes on betalains content (66.05 ± 0.87 mg/L) and antioxidant capacity (14.53±0.05 TEAC). On the other hand, there were significant changes on color properties due to was decreased the L, a*, b* and C values by removal of turbidity on the juice and finally, the hº value was decreased (from 40.76 to 30.37), it, means the redness grade was increased.**

Keywords— Transmembranal pressure (TMP); cleaning efficiency (CE); fouling index (FI); volume reduction factor (VRF); betalains content; antioxidant capacity.

I. INTRODUCTION

Purple cactus pear (*Opuntia ficus indica*) fruit can be considered a food with nutraceutical and functional properties due to its high content of bioactive compounds [4]. A marked antioxidant activity was recognized in several aqueous extracts as red, yellow, purple and white fruits [1]. This biological activity its due to the presence of various pigments, such as betalains [7]; [10]. Fruits of the *Opuntia* genus have little industrial utilization but are widely consumed as fresh fruits. Due to their high water content (a_w > 0.8), neutral pH, and soluble solid content, the pulp has high susceptibility to microbial attack, which makes the postharvest handling of these fruits difficult. Cactus pear processing represents a very interesting challenge for taking advantage of this resource [16]. Some cactus pears are attractive alternatives to the synthetic colorants that are commonly are used in foods.

Last decade, food industry has had several problems in terms of processing, obtaining new products with high added value. Membrane processes as microfiltration (MF), ultrafiltration (UF) and nanofiltration (NF) have been widely applied to the diary, food and beverage industry in order to reduce the storage and shipping costs, and to achieve longer storage in food products, by the way the membrane processes have been widely used due to their characteristics that represents great advantages during the processing [4]; [14]. Specifically, UF processes offers particular advantages in terms of absence of phase transition, mild operating conditions, low energy requirement, separation efficiency and easy scaling up when compared with conventional methodologies [11]; [12]; [5]; [6].

UF technologies has been used in order to clarified many fruit juices and large variety of new products, based on clarified fruit juices, have appeared in the markets this products can be achieved only by completely removing all suspended solids [22], that is related to high turbidity in the juices [23]. These processes have been used for the recovery of several compounds, including phenols and acids, i.e., citric, ascorbic, folic, and glutamic acids from fruit juices, such as yellow cactus pear [4], orange [2], bergamot [9] and kiwi [3].

The aim of this study is to analyze the ultrafiltration process in order to clarify purple cactus pear juice, analyzing the viability process in terms of productivity (flux permeate), fouling index, cleaning efficiency, and influence of the membrane on juice quality.

II. MATERIALS AND METHODS.

A. Materials.

The purple cactus pear fruits used in this study were harvested in July 2012 from San Martin de las Piramides, México (geographical coordinates: 19° 37' 05" and 19° 46' 20" north latitude and between 98° 45' 40" and 98° 53' 27" west longitude).

B. Methods.

1) Pre-treatment of purple cactus pear juice.

The fruits were washed with water, disinfected with 1 mg/L sodium hypochlorite solution, and then were chopped and pulped using a ®TURMIX juice extractor. The juice obtained was centrifuged at 10,332 xg for 20 min in a Beckman Coulter centrifuge (model J2-MC, USA).

2) Clarification of purple cactus pear juice.

a) Experimental set-up and procedures.

The supernatant obtained was submitted to a UF process in order to eliminate suspended solids and high molecular weight compounds. UF experiments were performed using laboratory unit, the UF unit was equipped with a feed tank, and peristaltic pump, a cooling coil working with tap water, a manometer and pressure-regulating valve.

The UF unit was equipped with different polysulfone hollow fiber membrane module (Amersham Biosciences Corp. Modelo CFP-1-E-4A, USA) of 30 kDa. UF experiments were carried out according to the recirculation configuration at an operating temperature of 25 ±1 °C, at an axial flow rate (Q_f) of 58 L/h, at different transmembrane pressures (35, 69, 103, 137 and 171 kPa) in order to find the optimal pressure that provides the limiting flux for carried out batch concentration configuration.

b) Batch concentration configuration.

The UF system was operated at a TMP of 137 kPa ± 1 kPa, at an axial flow rate (Q_f) of 58 L/h and a temperature of 25 ±1 °C according to the batch concentration mode (recycling the retentate stream and collecting separately the permeate). Volume permeation fluxes were measured up to volume reduction factor (VRF) of 6. The VRF was defined as the ratio between the initial feed volume and the volume of resulting retentate according to the following (1) [13]:

$$\mathrm{VRF} = \frac{V_F}{V_R} = 1 + \frac{V_P}{V_R} \qquad (1)$$

where Vf, Vp and Vr are the volumes of feed, permeate and retentate, respectively. Samples of feed, permeate and retentate collected during experimental runs were kept in the freezer (-17°C) until analysis.

3) Membrane parameters.

a) Water permeability (WP).

According to the Darcy´s law [21]:

$$Jv = \frac{L_P}{\Delta P} \qquad (2)$$

the water permeability (Lp) of the membrane was determined by the slope of the straight line obtained plotting the water flux (Jv) values, measured in fixed conditions of temperature (25°C) against the applied transmembrane pressure (Δp).

b) Fouling index (FI).

The fouling index (I_f), expressed as a percentage drop in water permeability, was estimated by measuring the water permeability before and after treatment of the juice, according the following (3):

$$I_f = \left(1 - \frac{Lp_1}{Lp_0}\right) \bullet 100 \qquad (3)$$

where Lp_0 and Lp_1 are the water permeabilities measured before and after clarification, respectively.

After the clarification, the UF membrane was raised with tap water for 20 min and their water permeabilities were measured again. UF membrane was submitted to an enzymatic cleaning with Ultrasil 67 (Ecolab, Minnesota; USA) at 0.5% (v/v) (60 min, 55°C). At the end of each cleaning procedures membrane was rinsed with tap water for 30 min and water permeability was measured again.

c) Cleaning efficiency (CE).

Cleaning efficiency was evaluated by using the flux recovery (FR,%) method described in [8], according to the following equation:

$$F_R = \left(\frac{Lp_2}{Lp_0}\right) \bullet 100 \qquad (4)$$

where Lp_2 is the water permeability measured after enzymatic cleaning.

4) Physicochemical parameters on clarified juice.

a) Antioxidant activity.

The antioxidant activity was determined using the DPPH method (stability of 1,1-diphenyl-2-picrylhydrazyl radical) according to [15]. Briefly, 0.05 mL of the clarified juice was added to 2.95 mL of methanol (dilution factor = 60) and 2 mL of the DPPH 0.1 μmol/L methanolic solution. The absorbances of the solution samples were measured at 517 nm at 30-min intervals, and the antioxidant activity was calculated by interpolation using a standard curve constructed with Trolox and DPPH 0.1 μmol/L methanolic solution. The results are expressed as μmol of Trolox equivalents per milliliter of sample (TEAC) [15].

b) Quantification of betalains.

The betalains was quantified through the spectrophotometric method described by [4], with some modifications. First, 10 g of juice or the spray-dried was mixed with 80% ethanol-aqueous solution to a final volume of 100 mL. The mixture was filtered and stored in an amber bottle, and 1 mL of the mixture was diluted to a final volume of 5 mL using 80% ethanol-aqueous solution. The resulting diluted mixture was magnetically stirred for 1 min. The absorbance of the solution was measured at 538 nm (for betacyanins) and 476 nm (for betaxanthins). The pigment content was determined using the following (5):

$$\mathrm{BC} = \left(\frac{\mathrm{A} \cdot \mathrm{DF} \cdot \mathrm{V} \cdot 1000}{\mathrm{E}_{1\mathrm{cm}}^{1\%} \cdot \mathrm{L} \cdot \mathrm{P}}\right) \qquad (5)$$

where BC is the betalain content (mg betalains/L in juice), A is the absorbance for each pigment, DF is the dilution factor, P is the weight or volume of the sample, V is the aliquot volume, $E^{1\%}_{1cm}$ is the extinction coefficient in ethanol (betacyanins = 1120 and betaxanthins = 750 [19]., and L is the path length (1 cm).

c) Color measurement.

First, 30 mL of the juice was placed in a Gerber container, and the L*, a*, and b* color parameters of the CIELAB scale were measured using a colorimeter (®Konica Minolta Colorimeter CR-10, Japan). The hue angle (h°) and chroma (C) values were calculated using (6) and (7), respectively [16]:

$$h^{o}= \tan^{-1}\left(\frac{b^*}{a^*}\right) \quad (6)$$

$$C= \left(a^{*2}+b^{*2}\right)^{1/2} \quad (7)$$

III. RESULTS AND DISCUSSION.

A. *Effect of operating parameters on the permeate flux.*

UF experiments, carried out according to the total recycle mode, were performed in order to study the effect of TMP on the permeate fluxes. Fig.1 shows permeate flux values at steady state versus the applied TMP.

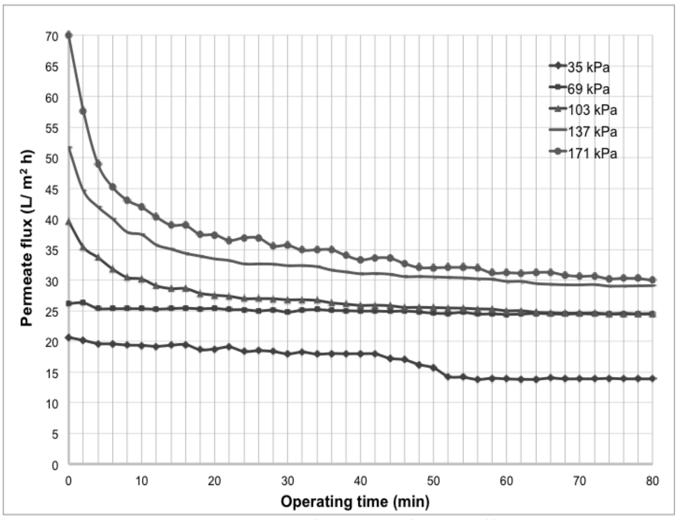

Fig.1.Operating time of permeate flux at different TMP (Conditions:T=25ºC, Q_f=58 L/h).

For small pressures the flux is proportional to the applied pressures, as the pressure is increased flux shows a deviation from a linear flux-pressure behavior and it becomes independent of pressure. In these conditions a limiting flux is reached at a TMP value of 137 psi (See Fig.2) and any further pressure increase determines no significant increase of the permeate flux.

The existence of a limiting flux can be related to the concentration polarization phenomenon that arises as the feed solution is convected towards the membrane where the separation of suspended and soluble solids from bulk solution takes place [2].

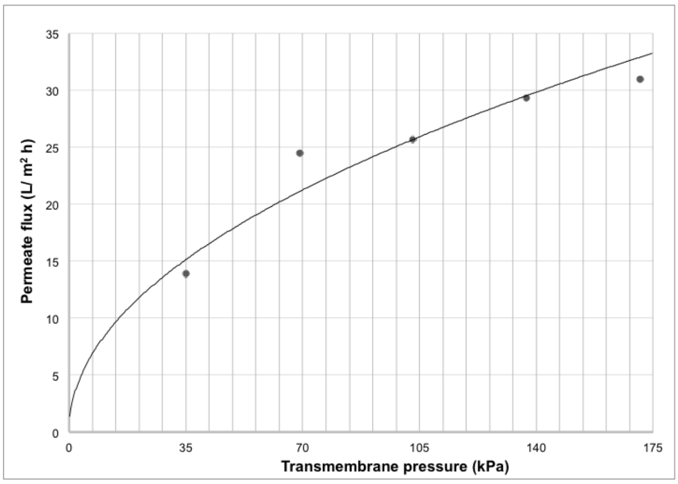

Fig.2. Effect of the TPM on the permeate flux (T=25ºC, Q_f=58 L/h).

A concentration profile from bulk solution to membrane surface is generated by the rejected material accumulated on the membrane, the formation of a viscous and gelatinous-type layer is responsible for an additional resistance to the permeate flux in addition to that of the membrane [2].

Basically, the TMP limiting values depends on physical properties of the suspension and feed flow rate. The cross-flow velocity affects the shear stress at the membrane surface and, consequently, the rate of removal of deposited particles responsible of flux decreased.

B. *Batch concentration configuration.*

An increasing of temperature produces higher permeate fluxes, an operating temperature of 25ºC was chosen in experimental runs performed according to a batch concentration mode to preserve the organoleptic and nutritional properties of the fresh juice.

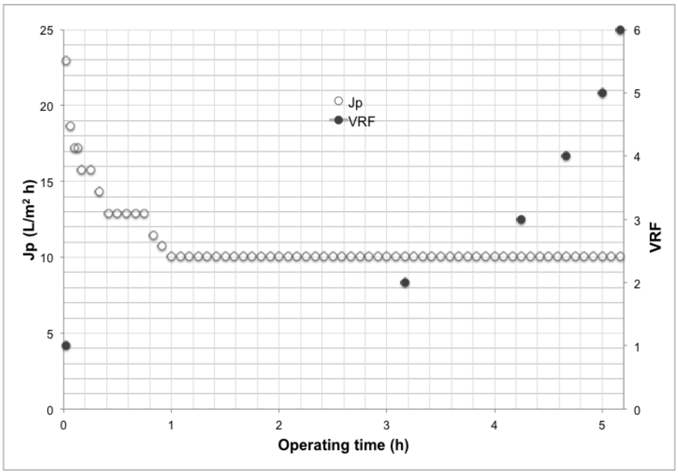

Fig.3. Operating time of permeate flux and VRF (TMP=137 kPa, T=25ºC, Q_f=58 L/h)

Results showed that the permeate flux decreased gradually with the operating time by increasing the volume reduction factor (VRF) (see Fig. 3) due to concentration polarization phenomenon and gel formation. The initial permeate flux of 22.94 L/ m^2 h decreased to about 10.04 L/ m^2 h when the VRF value reached 6.

C. Viability of the UF process:Fouling and cleaning efficiency.

In recirculation configuration, Table I shows that the lower fouling index was at 35 kPa of TMP which was recovered the initial permeability of the membrane (cleaning efficiency 100%), however also was recovered totally this property at 69 and 137 kPa. In terms of production at 137 of TMP, it´s better to perform the clarification process due to the higher permeate flux.

TABLE I. EVALUATED PROPERTIES ON THE MEMBRANE DURING RECIRCULATION AND BATCH CONCENTRATION CONFIGURATIONS

Membrane properties	**Transmembrane pressures (kPa)**				
	35	***69***	***103***	***137***	***171***
Lp_0 (L/m^2 h kPa)	0.891	1.039	1.205	0.896	0.964
Lp_1 (L/m^2 h kPa)	0.686	0.569	0.733	0.417	0.340
Lp_2 (L/m^2 h kPa)	0.893	1.039	0.896	0.896	0.935
Fouling index (%)	23.09	45.90	39.11	53.40	64.66
Cleaning efficiency (%)	100.0	100.0	74.37	100.0	96.99

During batch concentration configuration, the initial permeability was 0.896 L/ m^2 h kPa, after clarification process the permeability was decreased up to 0.417 L/ m^2 h kPa and finally, the membrane permeability was recovered totally after enzymatic cleaning (0.896 L/ m^2 h kPa) that means a cleaning efficiency of 100 %.

It can see in the experiments that it can be recovered the initial permeability from 74.37 up to 100 % with enzymatic cleaning. Basically, it were recovered the permeabilities due to ULTRASIL 67 is conformed by several enzymes that hydrolyze all polysaccharides rejected from the juice on the membrane surface.

D. Influence of clarification on physicochemical properties of the juice.

As shown in Table II, the membrane process favorably influences the properties of normal juice by decreasing the L values from 19.3 to 11.43 and the C values 3.8 to 3.3. The high values obtained on normal juice are due to the high turbidity of the juice due to the presence of polysaccharides, which were separated in the membrane step. All data obtained from the measurements of the color parameters in the juice before and after clarification are shown in the first quadrant of the CIELab color chart [17].

TABLE II. PHYSICOCHEMICAL PROPERTIES OF THE JUICE BEFORE AND AFTER THE CLARIFICATION PROCESS.

Property	**Before clarification**	**After clarification**
Betalain content (mg/ L)	62.69±0.89	66.05±0.87
Antioxidant activity (TEAC)	14.35±1.19	14.54±0.85
Colorimetry		
L	19.3±0.00	11.43±0.00
a*	2.9±0.00	2.9±0.00
b*	2.5±0.00	1.7±0.00
C	3.8±0.00	3.36±0.00
h°	40.76±0.00	30.37±0.00

The h° value decreased from 40.76 to 30.37 in juice before and after clarification process, respectively, which indicated an increase in the degree of redness in the permeate. In addition, the a* values did not present changes (2.9 both cases), and the b* values decreased from 2.5 to 1.7. Compared with the results reported by [18], who found L, C a* and b* colorimetry values for purple cactus pear juice of 13.99, 8.00, 7.98 and 0.57, respectively, the respective values for permeate obtained in this study are lower, i.e., 11.43, 3.36, 2.9 and 1.7, respectively. The h° value of permeate was 30.37, which is higher than the value of 0.07 reported in [18]. All these changes can be attributed to the membrane process use in our study due to another study that only used a simple centrifugation.

Additionally, the concentration of betalains increase on clarified juice from 62.69 to 66.05 mg /L caused an increase in the antioxidant activity. The maximal antioxidant activity obtained in the clarified juice was 14.53±0.05 TEAC, whereas that found in juice before clarification was 14.35±1.19 TEAC (or 15.06 µmol Trolox/ g). This value is higher that the values of 4.20± 0.51 µmol Trolox/ g and 3.64 ± 0.27 µmol Trolox/ g reported by [1] and [20], respectively.

IV. CONCLUSIONS.

The best TMP in order to clarify purple cactus pear juice was 137 kPa using UF membrane of 30 kDa however at this TMP was the higher fouling index. The permeability of the membrane can be recovered totally (cleaning efficiency 100%) by enzymatic cleaning which allows viewing ultrafiltration as a viable technology for clarification of juices with high productivities (permeate fluxes).

Basically there were no changes on physicochemical properties (as betalains content and antioxidant capacity) on the juice after clarification, it means was conserved the initial properties of the juice. However, there were major changes on color properties due to increased the redness grade and decreased L, a*, b* and C of the juice by the removal of the high molecular weight polysaccharides on the natural juice, the removal of this components decreasing directly the initial turbidity on the juice. The amazing changes on colorimetry are beneficials to the sensorial properties of the fruit during a possible commercial production.

ACKNOWLEDGMENT

The authors acknowledge to CONACyT (Fellowship 439602/267705) and IPN (SIP 20140554) for financial support.

REFERENCES

[1] Butera, D., Tesoriere, L., DiGaudio, F., Buongiorno, A., Allegra M., Pintauri, A.M., Kohen, R. & Livrea, M.A. (2002). Antioxidant activities of sicilian prickly pear (Opuntia ficus indica) fruit extracts and reducing properties of its betalains: batanin and indicaxanthin, *Journal of Agricultural and Food Chemistry*, 50, 6895–6901.

[2] Cassano A., Marchio M. & Drioli E. (2007). Clarification of blood orange juice by ultrafiltration: analyses of operating parameters, membrane fouling and juice quality. *Desalination,* 212, 15–27.

[3] Cassano A., Donato L., Conidi C. & Drioli E. (2008). Recovery of bioactive compounds in kiwifruit juice by ultrafiltration. *Innovative Food Science and Emerging Technologies,* 9, 556–562.

[4] Cassano A., Conidi C. & Drioli E. (2010). Physico-chemical parameters of cactus pear (Opuntia ficus-indica) juice clarified by microfiltration and ultrafiltration processes. *Desalination*, 250, 1101–1104.

[5] Cassano A., Conidi C. & Drioli E. (2011a). Comparison of the performance of UF membranes in olive mill wastewaters treatment. *Water research*, 45, 3197-3204.

[6] Cassano A., Conidi C. & Drioli E. (2011b). Clarification and concentration of pomegranate juice (*Punica granatum* L.) using membrane processes. *Journal of Food Engineering*, 107, 366–373.

[7] Castellar, M.R., Obón, J. M., & Fernández-López, J. A. (2006). The isolation and properties of a concentrated red-purple betacyanin food colourant from *Opuntia stricta* fruits. *Journal of the Science of Food and Agriculture*, 86, 122–128.

[8] Chen V., Li H., Tan D.S. & Petrus H.B. (2006). Cleaning strategies for membrane fouled with proteins mixtures. *Desalination*, 200, 198-200.

[9] Conidi C., Cassano A. & Drioli E. (2011). A membrane-based study for the recovery of polyphenols from bergamot juice. *Journal of Membrane Science*, 375, 182–190.

[10] Díaz Sánchez, F., Santos López, E. M., Filardo Kerstupp, S., Villagómez Ibarra, R., & Scheinvar, L. (2006). Colorant extraction from red prickly pear (*Opuntia lasiacantha*) for food application. *Electronic Journal of Environmental, Agricultural and Food Chemistry*, 5(2), 1330-1337.

[11] Dova M.I., Petrotos K.B. & Lazarides H.N. (2007a). On the direct osmotic concentration of liquid foods. Part I: Impact of process parameters on process performance. *Journal of Food Engineering,* 78, 422–430.

[12] Dova M.I., Petrotos K.B. & Lazarides H.N. (2007b). On the direct osmotic concentration of liquid foods: Part II. Development of a generalized model. *Journal of Food Engineering*, 78, 431–437.

[13] Garcia-Castello E.M., Mayor L., Chorques S., Arguelles A., Vidal-Brotons D. & Gras M.L. (2011). Reverse osmosis concentration of press liquor from orange juice solid wastes: flux decline mechanisms. Journal of Food Engineering, 106, 199-205

[14] Jiao B., Cassano A. & Drioli E. (2004). Recent advances on membrane processes for the concentration of fruit juices: a review. *Journal of Food Engineering,* 63, 303–324.

[15] Kuskoski, E.M., Asuero, A.G.,Troncoso, A.M., Mancini-Filho, J. & Fett, R. (2005). Application of several chemical methods to determine antioxidant activity in fruit pulps. *Ciencia y Tecnología de Alimentos, Campinas*, 25(4), 726-732.

[16] León-Martínez, F.M., Méndez-Lagunas, L.L. & Rodríguez-Ramírez, J. (2010). Spray Drying of nopal mucilage (*Opuntia ficus-indica*): Effects on powder properties and characterization. *Carbohydrate Polymers*, 81, 864–870.

[17] Mapari, S. A., Meyer, A.S., & Thrane, U. (2006). Colorimetric characterization for comparative analysis of fungal pigments and natural food colorants. *Journal of Agricultural Food Chemistry.* 54, 7027-7035.

[18] Obón, J.M., Castellar, M.R., Alacid, M. & Fernández-López, J.A. (2009). Production of a red-purple food colorant from *Opuntia stricta* fruits by spray drying and its application in food model systems. *Journal of Food Engineering*, 90, 471-479.

[19] Soriano-Santos, J., Franco-Zavaleta, M.E., Pelayo-Zaldivar, C., Armella- Villalpando, M.A., Yáñez- López, M.L. & Guerrero-Legarreta, I. (2007). A partial characterization of the red pigment from the mexican fruit cactus "Jiotilla" (*Escontria chiotilla* [Weber] Britton & Rose). *Revista Mexicana de Ingeniería Química*, 6, 19-25.

[20] Stintzing, F.C., Herbach, K.M., Mosshammer, M.R., Carle, R., Yi, W.,Sellappan, S., Akoh, C.C., Bunch, R. & Felker, P. (2005). Color, Betalain Pattern, and Antioxidant properties of Cactus pear (*Opuntia* spp.) clones. *Journal of Agricultural and Food Chemistry*, 53, 442-451.

[21] Strathmann H., Giorno L. & Drioli E. (2006). An introduction to Membrane Science and Technology. Consiglio Nazionale delle Richerche. Rome, Italy.

[22] Vaillant F., Millan A., Dornier M., Decloux M. & Reynes M. (2001). Strategy for economical optimisationof the clarification of pulpy fruit juices using crossflow microfiltration. *Journal of Food Engineering*, 48, 83-90.

[23] Vaillant F., Pérez A.M., Acosta O. & Dornier M. (2008). Turbidity of pulpy fruit juice: A key factor for predicting cross-flow microfiltration performance. *Journal of Membrane Science*, 325, 404–412.