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Concentration of grape juice (*Vitis labrusca*) by reverse osmosis process

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ABSTRACT

The objective of this work was to evaluate the concentration of grape juice by reverse osmosis (RO). The experiments were carried out in two steps. First, three transmembrane pressures (40, 50 and 60 bar) and four temperatures (20, 30, 40 and 50ºC) were evaluated without juice concentration. The following parameters were analyzed: acidity and pH, soluble solids content, concentration of phenolic compounds and concentration of monomeric and total anthocyanins, color index, color density, as well as the permeate flux. Under the evaluated conditions, the process conducted at 50ºC and 60 bar presented the higher permeate flux and the maintenance of all the physical and chemical parameters of the product. In the second step, the grape juice was concentrated at three temperatures, 20, 30 and 50ºC, always at 60 bar of transmembrane pressure. The temperature of 30ºC resulted in an adequate value for the permeate flux besides maintaining the physical-chemical and nutritional characteristics of the concentrated product when compared to the single strength juice. Moreover, in relation to the durability of the membranes, this condition corresponds to the Wagner Index equal to 1800, appropriate to maintain the integrity of the reverse osmosis membranes.

Keywords: Reverse osmosis; Grape juice, Membrane processes; Concentration of fruit juices; Physico-chemical characteristics; Wagner Index; Permeate flux

1. Introduction

Grapes are an important source of antioxidants as they contain a large amount of different phenolic compounds in skins, pulp and seeds, that are partially extracted during the processing of the juice and wine. In Brazil, grape producing region has just one annual vintage. So during the harvest the fruits have to be processed in order to provide sufficient raw material for the juice industries during the off-season [1].

Grape juice concentrated to 55, 65 or 68° Brix minimizes transportation and storage costs. This concentrate is diluted for use in single strength grape juice or multi-fruit and sparkling juice. Fruit concentrate is used full strength to sweeten jams, jellies, yogurt, frozen fruit desserts, cereals, cookies and other bakery products. Many consumers perceive fruit concentrates to be a healthy replacement to table sugar and corn sweetener. Traditionally, fruit juice concentration is carried out by vacuum evaporation that usually involves damage to the product such as loss of freshness and aroma [2]. The thermal processing can promote significant changes in the sensory and nutritional quality of the product, since these characteristics are conferred by volatile compounds and vitamins, most of them thermolabile.

Reverse osmosis is considered an adequate technology to concentrate fruit juices, and it presents as advantages the lower energy consumption, minimal

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heat damages of the quality properties, maintenance of sensory characteristics and nutritional value of the products, absence of caramelization reactions, compact and ease to operate facilities and higher purity of the permeate compared to other membrane processes [3,4].

The application of reverse osmosis aiming at the fruit juice concentration requires the use of high operation pressures due to the continuously increase of the osmotic pressure of the product along the concentration [5].

According to Mietton-Peuchot, Milisic and Noilet, RO allowed the concentration of grape must with excellent retention of the components and generated wines similar to those obtained by the chaptalization process [6].

Microfiltration and reverse osmosis were used to concentrate white and red grape juice, in a plate and frame module, at 35°C, 50 bar and flow rate of 300 l/h. The retention of total solids in the red grape must was very high (99%) as well as the retention of anthocyanins (99.5%), confirming that RO is effective to retain solutes of importance and it can improve the must quality for the wine industry [7]. Rektor et al. [8] evaluated the concentration by RO of red grape must from 16° Brix to 23° Brix. The process was conducted at 40°C and transmembrane pressures ranging from 40 to 50 bar. The permeate flux decreased from 8 kg/m²h to 1 kg/m²h after 1.5 h of concentration.

Kiss et al. evaluated the use of microfiltration associated with RO and nanofiltration to produce a grape juice with 45º Brix [9]. The authors concluded that the initial investment and operational costs of vacuum evaporation were greater than those associated with RO, due to the high cost of thermal energy.

Reverse osmosis allows the permeation of water from the juice but it is limited by the high osmotic pressures of the products that are being concentrated. So it is applied as a preconcentration technique that enable concentration values up to 35º Brix corresponding to osmotic pressures of about 50 bar [10]. Therefore, this membrane process should be coupled to others technologies such as osmotic evaporation in order to achieve the required concentration (60º Brix) [11–14]. The objective of this work was to evaluate the concentration process of grape juice by RO considering the parameters of the process and the quality of the concentrated juice.

2. Materials and methods

2.1. Raw material

Single strength or integral grape juice was used as raw material. This juice is obtained by hot pressing that is appropriate for deeply pigmented grapes where maximum colour extraction is desired. The production of the grape juice has a step of enzymatic treatment (celulases and pectinases) followed by filtration, so that this juice is clarified and do not contain suspended particles. So no pre-treatment process was necessary previous to the RO tests.

2.2. Reverse osmosis system

Reverse osmosis was carried out in a plate and frame module composed of HR98PP thin film composite membranes (DSS, Denmark), with total membrane area of 0.68 m² and rejection of 95% to a 0.25% NaCl solution at 25°C and 42 bar. A schematic representation is shown in Fig. 1. The cleanup of the module consisted in rinsing the system with tap water. Then, a 0.5% sodium hydroxide solution was recirculated for 30 min. Finally, the circuit was rinsed with distilled water at 40°C up to the measurement of the pH of the permeate stream indicated absence of sodium hydroxide.

Before each concentration assay, water permeability was determined at different temperatures and transmembrane pressures to assure the cleaning and integrity of the membranes.

2.3. Fruit juice concentration

The experiments were accomplished in two steps. First, the effect of the operational conditions on the quality of the product and permeate flux was evaluated. The temperature (20, 30, 40 and 50°C) and transmembrane pressure (20, 40 and 60 bar) were the chosen independent variables and the permeate flux and the physical and chemical characteristics of the retentate juice were the dependent ones. In these experiments the juice was not concentrated, i.e., the retentate and permeate

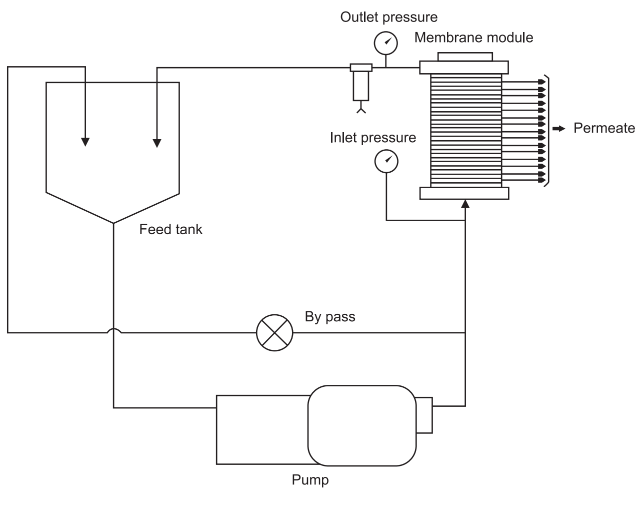

Fig. 1. Schematic representation of the reverse osmosis system.

streams were recirculated back to the feed tank under controlled temperature and transmembrane pressure over a two-hour period. All the experiments were conducted in duplicate.

After this step, the concentration of the grape juice by reverse osmosis was evaluated. Concentration tests were carried out at the transmembrane pressure and temperature defined in the first step, of 60 bar and 50°C. Lower temperatures, 20°C and 30°C were also evaluated in order to investigate the flux behavior in more kindly process conditions. In this step, the retentate stream was recirculated to the feed tank and the permeate was continuously collected. The processes were carried out in a fed batch mode. The concentration experiments were repeated three times for each temperature condition. At every 10 min the permeate flux and the soluble solids content of the retentate fraction (concentrated juice) were determined.

2.4. Process evaluation

All the processes were evaluated regarding the permeate flux, the volumetric concentration factor (VCF) and the quality characteristics of the juice. The permeate flux (measured in triplicate) and the VCF were calculated using the Eqs. (1) and (2):

$$J = \frac{V}{A * t} \quad (1)$$

$$\mathrm{VCF} = \frac{V_R}{V_F} \quad (2)$$

where V is the volume permeate during a determined time t, A is the membrane surface, V_R is the final retentate volume and V_F is the initial feed volume.

2.5. Analytical methods

Samples of the integral juice, the juice processed by reverse osmosis after 2 h without concentration (first step) and the concentrated juice were analyzed with respect to the following physical and chemical parameters: pH and total soluble solids [15]; total titrable acidity [16], colour index [17]; monomeric anthocyanins and colour density [18]; total anthocyanins [19]; degradation index [18] and total phenolic compounds [20,21]. The statistical Tukey test was performed using Excel software (Microsoft, Office Excel 2000).

3. Results and discussion

Regarding the reverse osmosis of the grape juice without concentration, the results showed that there was no significant difference in any of the quality parameters (total soluble solids, pH, total titrable acidity, monomeric anthocyanins, total anthocyanins, colour index, colour density, degradation index and total phenolic compounds) of the juice concentrated under any of the evaluated conditions ($p < 0.05$) as shown in Table 1. The temperature and the transmembrane pressure had a positive effect on the permeate flux. This effect is directly related to the juice viscosity, which is exponentially dependent on temperature (Arrhenius Law). The highest permeate flux was achieved when the process was carried out at 60 bar and 50ºC (Fig. 2).

Table 1
Permeate flux and physical and chemical evaluation of the grape juice processed for two hours by reverse osmosis at 40 and 60 bar and 20, 30, 40 and 50ºC (without concentration)

Temperature (ºC)	20	20	40	40	30	50
Pressure (bar)	40	60	40	60	50	60
Total acidity[1]	0.69[a]	0.71[a]	0.70[a]	0.68[a]	0.68[a]	0.66[a]
pH	2.98[a]	2.93[a]	2.93[a]	2.92[a]	2.96[a]	2.91[a]
Soluble solids	13.6[a]	14.4[a]	13.7[a]	13.7[a]	13.7[a]	13.9[a]
Color Index (ID)[2]	2.086[a]	2.124[a]	2.029[a]	2.049[a]	2.030[a]	2.061[a]
Color Density[2]	6.56[a]	7.05[a]	6.67[a]	6.73[a]	6.64[a]	6.62[a]
Total anthocyanins[3]	111.3[a]	112.3[a]	108.9[a]	106.6[a]	109.9[a]	105.3[a]
Monomeric anthocyanins[4]	85.1[a]	88.2[a]	84.1[a]	84.1[a]	82.9[a]	77.4[a]
Degradation index	2.38[a]	2.43[a]	2.49[a]	2.46[a]	2.44[a]	2.57[a]
Phenolic compounds[5]	1.87[a]	2.08[a]	2.04[a]	1.93[a]	1.98[a]	1.95[a]

[1] Expressed in g/100 g tartaric acid.
[2] Expressed as absorvance unit.
[3] Expressed in: mg of malvidin-3,5 diglucoside/100 g of sample.
[4] Expressed in: mg of malvidin-3,5 diglucoside/l.
[5] Expressed in g/l of galic acid.
[a, b, c, d, e, f] = average with equal letters in the same line denotes no statistical difference at a 95% significance level.

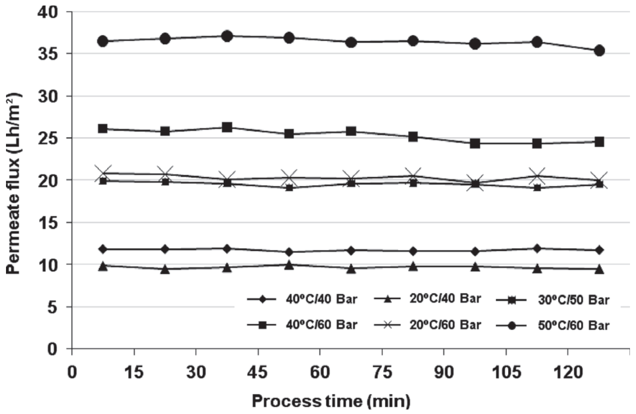

Fig. 2. Permeate flux during steady reverse osmosis processing.

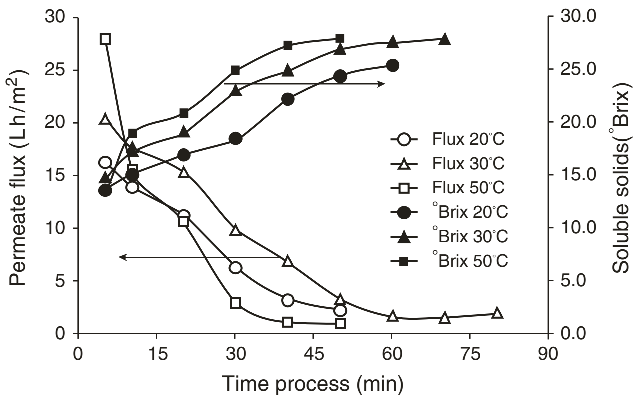

Fig. 3. Evolution of the permeate flux and soluble solids content during the concentration of grape juice by reverse osmosis at 50°C, 30°C and 20ºC and 60 bar.

The negligible influence of the RO temperature on the juice quality can be attributed to the extraction step that is used for the production of the grape juice. The extraction process of grape juice is accomplished by heating the fruits up to 90°C to maximize the extraction of the phenolic compounds [22,23], such as anthocyanins that are mostly present in the peel. However, the exposure of grape juice to high temperature for long periods can result in losses of phenolic compounds, as well as degradation of anthocyanins. Larrauri et al. evaluated the effect of temperature concentration (60ºC, 100ºC and 140ºC) on grape juice quality. It was not verified any significant changes in color characteristics, antioxidant activity or phenolic compounds content of the grape juice up to 60ºC. When the drying temperature was increased up to 100°C or 140°C, a significant reduction in the total polyphenols (18.6 and 32.6%), as well as a decrease in the antioxidant activity (28% and 50%) was observed [24].

Regarding the concentration processes, mean initial permeate fluxes were 27.91 h/m^2, 20.41 h/m^2 and 16.31 h/m^2, respectively, when the temperatures of the process were 50°C, 30°C and 20ºC (Fig. 3). The fed process minimizes the high fall of the permeate flux, but it does not hinder its decline. Regarding the mass balance, the permeate is being continuously removed (water), but the added feed has at least the solid concentration of the original juice, that contributes for the increase of the solid concentration in the tank, and consequently of viscosity and osmotic pressure.

It can be verified that there was a slight decline of the permeate flux along the processing. The decrease of permeate flux is usually attributed to concentration polarization and fouling phenomena. In this case, fouling is not favored since RO membranes have no pores to be blocked or adsorbed and the grape juice did not contain suspended solids. Specifically in the case of RO, the increased resistance is attributed to the membrane itself and to the physical changes that occur in the product during the concentration, mainly the increase in osmotic pressure and viscosity. The grape juice was being concentrated and consequently its osmotic pressure and viscosity increased over time. The concentration polarization is a phenomenon that occurs in the first moments of permeation, however, this was not evaluated in these experiments.

Recently, orange juice was concentrated by RO at 60 bar and 30ºC. The soluble solids content of the concentrated orange juice reached 36° Brix [4]. The maximum concentration value achieved in the RO process is limited by osmotic pressure and by the viscosity of the product. The operation pressure of the system must always be greater than the osmotic pressure of the feed [25].

The average VCF achieved in the processes performed at 50°C, 30°C and 20°C, was 1.89, 2.08 and 1.69, respectively. The processes conducted at 20ºC finished spontaneously probably due to the raise of the viscosity of the concentrated juice at this lower temperature. The higher initial flux was observed in the process performed at 50°C caused by the lower juice viscosity, but at this condition it can be verified a sharp drop of the permeate flux after five minutes of process.

It was already demonstrated that the composite membrane HR98PP (DSS) presents a high retention of volatile compounds [26]. This is another advantage of the use of RO in the concentration of grape juice, since, during the thermal evaporation process, losses of some substances that contribute to juice aroma can be higher than 90%. But the concentration of the grape juice at 50°C and 60 bar was accomplished by a high aroma compounds loss that was verified by gas chromatographic and sensory analysis (data not shown). This was another reason for the selection of lower temperatures, 30°C or 20°C for the juice concentration.

The Wagner Index (WI) is defined as the product of the temperature (ºC) by the transmembrane pressure (bar) of the process and it is used to establish the maximum operation condition to maintain the membrane integrity [27]. According to some membrane manufacturers, the correct WI to the RO process should be lower than 2000 in order to prevent damage to the membranes. The processes conducted at 30°C and 20ºC resulted in WI equal to 1800 and 1200, respectively, assuring better processing conditions and greater useful life of the membranes.

4. Conclusion

It was possible to concentrate grape juice by RO up to 30° Brix. For all the evaluated process conditions the concentration of grape juice by RO did not change the quality parameters of the concentrated juice when compared to the single strength one.

The selected condition for the concentration of grape juice by reverse osmosis was temperature of 30ºC and 60 bar transmembrane pressure due to the adequate value of the permeate flux, the maintenance of the product quality besides favoring the maintenance of the membranes integrity.

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