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Evaluation of nanofiltration membranes for retention of anthocyanins present in açai (*Euterpe oleracea Mart.*) juice

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INTRODUCTION: Açai is an Amazonian palm. Its berries are harvested as food and it can be described as one of the most important plant species as these fruits make up the major component of the Amazonian people diet so that it is very economically and social valuable in this region. The açai juice and pulp of açai are used in various juice blends, sodas, and other beverages. In Amazonian region, açai pulp is traditionally served with cassava flour or fish but, depending on the local preference, it can be consumed either salty or sweet. Açai exhibits significantly high antioxidant capacity and therefore, it may have possible health benefits. It is rich in anthocyanins that are natural pigments, water soluble, intensely colored and widely distributed in nature that is found mainly in fruits and leaves, belonging to the family of flavonoids (BRITO et al, 2007). They represent together with the carotenoids the largest class of colored substances in the plant kingdom (GONNET, 1998) and they are responsible for the color of açai. Membrane separation processes have the ability to separate and concentrate bioactive compounds that can therefore be used as ingredients in food, cosmetic and pharmaceutical industries (MULDER, 2006).

MATERIALS AND METHODS: The raw material for the nanofiltration process was clarified açai juice. The pulp was microfiltrated in a 0.2 µm pore size tubular ceramic membrane with total permeation area of 0.022 m² at 2 bar and 35°C ± 2°C. Nanofiltration was carried out in a plate and frame system with 150 cm² of total permeation area. Six membranes were evaluated: (i) NF270 (Dow/Filmtec, composite polyamide polysulphone membrane, 99% MgSO₄ nominal rejection), (ii) UTC 60 (Toray, aromatic polyamide, 55% NaCl rejection at 15 bar and 25°C), (iii) MPF 36 (Koch membrane systems, 1 kDa cut off), (iv and v) DK and DL (GE Osmonics, composite membranes, 98% MgSO₄ and 96% MgSO₄ rejection at 25°C, respectively) and (iv) NP010 (Microdyn Nadir, polietersulfone composite membrane, 1 kDa cut off). Before starting the juice nanofiltration, the membrane was conditioned with distilled water for one hour at 35°C and 20 bar. The experiments were carried out with no concentration of the product, that means that the permeate and retentate fractions were returned back to the feed tank, at 10, 15, 20 and 30 bar of transmembrane pressure and at 35°C. After the process, the juice inside the nanofiltration system was washed out and the equipment was rinsed to remove the remaining product. Before the evaluation of the water permeability the permeate flux was measured in order to determine how much the açai juice processing reduced the filtration efficiency of the membrane (F%).

$$F(\%) = \frac{PWF_b - PWF_a}{PWF_b} \times 100$$

PWF_b represents the water flux before processing and PWF_a represents the water flux just after processing without cleaning. For each process three liters of juice were used. At each 10-minute process, the permeate flux was determined in triplicate. It was made a repeatability test (three assays) with the membrane that shown the highest permeate flux and higher retention of anthocyanins. To check any possible permeation of anthocyanin through the membrane, samples of permeate were collected after the application of each transmembrane pressure to anthocyanins analysis by the pH differential method (FULEKI, FRANCIS, 1968). The result was expressed as cyanidin-3-glucoside. The rejection coefficient of the membrane to anthocyanin was calculated by the equation:

$$R(\%) = \left(1 - \frac{C_p}{C_f}\right) \times 100$$

C_p represents the concentration of anthocyanin at the permeate fraction and C_f the concentration of this compound at the feed.

RESULTS: The water permeability results showed that there was a linear increase of the permeate flux when the applied transmembrane was increased. NF270 membrane presented the higher permeate flux, 168 L/hm², 244 L/hm², 312 L/hm², 428 L/hm², at 10, 15, 20 and 30 bar respectively, with an average flux 30% higher than the flux of NP010, second higher permeate flux. MPF 36 showed the lowest permeate flux, 37 L/hm², 53 L/hm², 69 L/hm² and 106 L/hm² at 10, 15, 20 and 30 bar, respectively.

Regarding the juice permeation, NF270 membrane showed the highest flux in all pressure ranges, 67 L/hm² to 10 bar, 84 L/hm² 15 bar, 93 L/hm² 20 bar and 102 L/hm² to 30 bar. Samples of permeate showed 100 retention to anthocyanins. The membrane MPF 36 had the lowest permeability, 8 L/hm² at 10 bar, 12 L/hm² at 15 bar, 17 L/hm² at 20 bar, 27 L/hm² at 30 bar, with anthocyanin concentration in the permeate of 4.5 mg/L at 10 bar. NP010 membrane had a permeate flux higher than the MPF36, however there was a higher permeation of anthocyanins to the permeate stream, 27mg/L at 10 bar.

Regarding the retention coefficient it was verified that the membrane MPF36 had 99.6% and 99.8% for 10 bar and 15 bar, respectively and NP010 presented 97.7%, 98.5%, 99.1% and 99.7% for 10, 15, 20 and 30 bar, respectively. For all other membranes it was not observed anthocyanin presence in the permeate fractions. By increasing transmembrane pressure the retention coefficient also increased. This can be explained by the solute adsorption in the membrane pores and surface. When the adsorption occurs inside the pores more severe changes occur in both membrane permeate flux and selectivity.

After the fouling evaluation it was observed a reduction in the permeate flux with the NF270 membrane with higher permeate flux and a reduction in efficiency of 28% at all range of the evaluated transmembrane pressure. The membrane MPF36 also had the lowest permeate flux with reduced efficiency of 49%, 42%, 38% and 38% at 10, 15, 20 and 30 bar. The membrane NP010 had the second performance regarding the permeate flux but had an efficiency reduction of 70% at all applied transmembrane pressures.

CONCLUSION: This work suggests that the nanofiltration process is able to retain anthocyanins of açai so that it can be used for concentrate this substance. The NF270 membrane presented the highest permeate flux and the best relationship between permeate flux and anthocyanin retention coefficient.

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