



Stabilization of açai (*Euterpe oleracea* Mart.) juice by the microfiltration process

Flávio Caldeira Silva^{1*}, Daise Aparecida Rossi², Vicelma Luiz Cardoso³ and Miria Hespanhol Miranda Reis³

¹Setor de Agroindústria, Instituto Federal do Triângulo Mineiro, Av. Belarmino Junqueira, s/n., 38305-000, Ituiutaba, Minas Gerais, Brazil. ²Escola de Medicina Veterinária, Universidade Federal de Uberlândia, Uberlândia, Minas Gerais, Brazil. ³Faculdade de Engenharia Química, Universidade Federal de Uberlândia, Uberlândia, Minas Gerais, Brazil. *Author for correspondence. E-mail: flaviocaldeira@iftm.edu.br

ABSTRACT. Açai berry, a Brazilian palm fruit widely distributed in northern South America, is acknowledged for its functional properties such as high antioxidant capacity and anti-inflammatory activities. Although the açai juice is highly appreciated in Brazil and even worldwide, its commercialization is still limited. Microfiltration process is largely applied in juice processing, eliminating many of the traditional processing steps and reducing time, energy and addition of clarifying agents. Furthermore, microfiltration process may eliminate microorganisms and compounds responsible for turbidity in the juice. Current assay applies a microfiltration process to obtain a stabilized açai permeate pulp. Microfiltrations of açai pulp were carried out in a dead end configuration with a flat membrane of 0.22 µm pore size. Permeate pulp was characterized according to its turbidity, lipid concentration and microbiological analysis. Initial permeate flux was 103 kg m⁻² h⁻¹. After an initial flux decline during 30 min., due to membrane compaction and fouling occurrences, flux was stabilized at 20 kg m⁻² h⁻¹. The microfiltration process reduced the initial açai pulp turbidity by 99.98% and lipids were not identified in the permeate. Microbiological analysis showed that the contamination by microorganism decreased in the permeate pulp when compared to that in raw açai pulp.

Keywords: açai, stabilization, membrane process, microfiltration.

Suco de açai estabilizado utilizando processo de microfiltração

RESUMO. O açai é um fruto da Amazônia reconhecido por suas propriedades funcionais, devido à sua elevada capacidade antioxidante e anti-inflamatória. Apesar da apreciação do suco de açai no Brasil e no mundo, sua comercialização ainda é limitada. O processo de microfiltração é largamente aplicado no processamento de sucos, eliminando etapas de processamento tradicionais, reduzindo tempo, energia e adição de agentes de clarificação. Ademais, a microfiltração pode eliminar micro-organismos e compostos responsáveis pela turbidez do suco. Este estudo visou à aplicação do processo de microfiltração para obtenção de permeado estabilizado de polpa de açai. Microfiltrações da polpa foram realizadas em um módulo de filtração com membrana plana com tamanho de poro de 0,22 µm. O permeado foi caracterizado quanto à turbidez e concentração de lipídeos, além de análises microbiológicas. O fluxo de permeado inicial foi de 103 kg m⁻² h⁻¹. Após um declínio fluxo inicial durante 30 minutos, devido à compactação e ao entupimento da membrana, o fluxo foi estabilizado em 20 kg m⁻² h⁻¹. A microfiltração reduziu a turbidez inicial da polpa de açai em 99,98%, e lipídeos não foram identificados no permeado. As análises microbiológicas mostraram que houve diminuição da contaminação por micro-organismos no permeado em relação à polpa de açai.

Palavras-chave: açai, estabilização, processos de separação por membranas, microfiltração.

Introduction

Açai is a Brazilian dark purple berry fruit averaging 2 cm in diameter, with a nutty flavor, lingering metallic undertones and a creamy, oily texture (Schreckinger, Lotton, Lila, & de Mejia, 2010). Its palm, *Euterpe oleracea*, is widely distributed in northern South America, mainly in the Amazonian delta. The fruit is generally consumed as juice and it is acknowledged for its functional

properties in food and nutraceutical products, due to its extremely high antioxidant capacity and potential anti-inflammatory activities. Açai fruit has received international interest mainly due to its potential health benefits associated to phenolic composition and antioxidant capacity (Del Pozo-Insfran, Brenes, & Talcott, 2004; Lichtenthaler et al., 2005; Schauss et al., 2006; Rufino et al., 2011). Muller, Gnoyke, Popken, and Bohm (2010) showed that the oxygen

radical absorbance capacity rate of the açai juice is nearly twice as high when compared to its ferric-reducing antioxidant power and Trolox equivalent antioxidant capacity, revealing the excellent capacity of açai anthocyanins to scavenge peroxy radicals.

Although highly appreciated in Brazil and even worldwide, the commercialization of the açai fruit is still limited since it is a highly perishable fruit and processing is essential to preserve its bioactive compounds (Del Pozo-Insfran et al., 2004; Gallori, Bilia, Bergonzi, Barbosa, & Vincieri, 2004; Coisson, Travaglia, Piana, Capasso, & Arlorio, 2005). Pavan, Schmidt, and Feng (2012) proposed three drying methods (freeze-drying, refractance window-drying (RW), and hot-air drying) to dehydrate açai juice, and concluded that the three methods produced samples with low water activity and relatively good product stability during storage.

Microfiltration is characterized as a non-thermal treatment. It is actually a potential process for fruit juice industries due to a better conservation of sensorial characteristics and phytochemical properties (Laorko, Tongchitpakdee, & Youravong, 2013). Microfiltration process may provide high quality, natural fresh taste and additive free food products. It is also simple, easy to scale up and characterized as a low energy consumption process (Cassano, Conidi, Timpone, D'Avella, & Drioli, 2007; Coutinho et al., 2009).

Juice microfiltration reduces many of the traditional steps in processing, such as time, energy and addition of clarifying agents (Echavarria et al., 2012). Further, the microfiltration process eliminates microorganisms and compounds which cause juice turbidity (Coutinho et al., 2009; Girard & Fukumoto, 2000). Membrane technology has been largely applied for the processing of some juices, such as apple, tomato, pineapple and passion fruit juices (Domingues, Ramos, Cardoso, & Reis, 2014; Girard & Fukumoto, 2000). Machado, Haneda, Trevisan, and Fontes (2012) analyzed the influence of the microfiltration process on the antioxidant capacity of the obtained permeate of açai juice and observed that this parameter was reduced by 36 and 27% for açai juices with and without enzymatic pretreatment, respectively. Similarly, Cruz et al. (2011) verified that the microfiltration process of açai pulp did not significantly influence the contents of bioactive components and the antioxidant activity of permeate and concentrated fractions. Couto et al. (2011) showed that the nanofiltration process was able to retain over 99% of the anthocyanins in the

açai pulp. Although membrane filtration processing is suitable for açai juice treatment, its application still needs further studies.

Current study proposes a microfiltration process to obtain a stabilized açai permeate pulp. Açai permeate pulp stabilization has been evaluated mainly in terms of turbidity and microbial count reductions which will ensure an easier commercialization of the juice. Moreover, decrease in the lipid contents in açai permeate pulp was also evaluated to verify the influence of the microfiltration process on this nutritional parameter. Since açai juice is a high nutritional fat product, reduction in lipid contents may increase its consumption.

Material and methods

Commercial frozen açai pulp (medium type, 11-14% total solids) was purchased on the local market in Uberlândia, Minas Gerais State, Brazil, and stored at -10°C until processing. Before processing, the açai pulp was defrosted at room temperature and microfiltered without any dilution and/or pretreatment.

Microfiltration processes were carried out using a cellulose ester microfiltration membrane (Milipore) with 0.22 µm pore size in a dead-end filtration cell (Figure 1) with total capacity of 1.5 L and 6.36 x 10⁻³ m² of permeation area. Operation conditions were 1.0 bar of transmembrane pressure and temperature of 25°C.

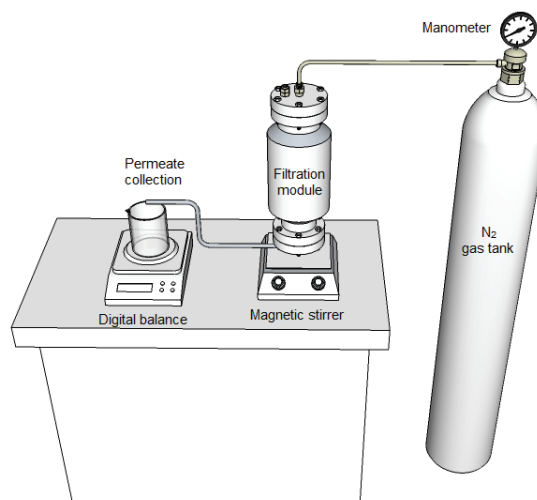


Figure 1. Dead-end filtration cell.

Permeate flux due to filtration time was calculated by Equation (1).

$$J = \frac{m}{At} \quad (1)$$

where J is the permeate flux ($\text{kg m}^{-2} \text{h}^{-1}$); A is the filtration area (m^2); m (kg) is the measured permeate mass at filtration time t (h).

Raw and permeate açai pulps were characterized according to their moisture, total solids, ashes, pH, lipids (AOAC, 2000) and turbidity. Turbidity was measured with a Nova Organica HD 114 turbidity meter (Brazil).

Microbiological analysis (mold and yeast, total counts and coliforms) were carried out in raw and permeate açai pulps according to the Bacteriological Analytical Manual (2001). These analyses were also carried out in permeate samples stored for 24h and 30 days, at room temperature ($25 \pm 5^\circ\text{C}$), with and without conservative additions. Sodium benzoate or benzoic acid was used as conservative agent at a concentration of 0.01% (m m^{-1}). Further, permeate samples were stored at 5°C without the addition of conservatives. These samples were packed in 50 mL glass vials, which were sealed with rubber stoppers and aluminum seals. All microbiological analyses were conducted in triplicate for each sample.

Results and discussion

Figure 2 presents permeate flux according to filtration time during açai pulp microfiltration and shows the typical behavior of a microfiltration process curve at a dead-end operation. Figure 2 reveals that the permeate flux decreases very rapidly during the first minutes of the procedure; the decrease rate diminishes with time and becomes very small for periods longer than 2 hours. The flow remains almost constant for the remainder of the microfiltration operation.

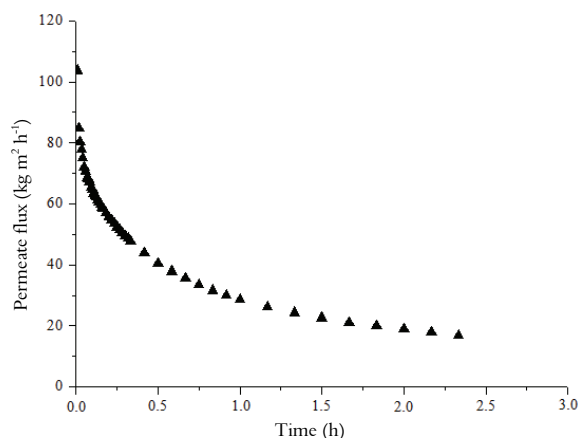


Figure 2. Permeate flux over time in the membrane during the microfiltration.

The initial permeate flux reached $103.7 \text{ kg m}^{-2} \text{h}^{-1}$ and abruptly decreased in the first 30 minutes of filtration, probably due to the formation of the cake

layer on the membrane surface. After approximately 90 min. of operation, the permeate flux remained constant and the final flow equaled $16.8 \text{ kg m}^{-2} \text{h}^{-1}$. The concentration factor was equal to 2. Açai pulp is rich in lipids (5-7% on a wet basis), protein and insoluble dietary fibers (Rufino et al., 2011; Schauss et al., 2006) and this abrupt flux decline is probably due to the deposition of these and other compounds on the membrane surface. Machado et al. (2012) showed that the enzymatic pretreatment increased the observed permeate flux of açai juice (up to $83 \text{ L h}^{-1} \text{m}^{-2}$ for a $0.2 \mu\text{m}$ pore size membrane) due to the pectinase action on pectin-protein complexes. Cruz et al. (2011) observed a similar steady state permeate flux ($18 \text{ L h}^{-1} \text{m}^{-2}$) during the filtration of açai pulp using a polymeric membrane. However, higher flux rates were registered by Cruz et al. (2011) and Couto et al. (2011) when they used ceramic membranes (about $100 \text{ L h}^{-1} \text{m}^{-2}$) in a cross-flow filtration. The cross-flow filtration probably reduced fouling occurrences and increased the permeate flux.

Table 1 shows the properties of açai pulp before and after the microfiltration process. Further, pH rate was not significantly affected by the processing, a fact already reported by Campos et al. (2002) and Carneiro, Sa, Gomes, Matta, and Cabral (2002) for microfiltrations of cashew apple and pineapple juices, respectively. There was a great reduction in the turbidity rate after the microfiltration process due to the removal of macromolecules from the raw pulp. Domingues et al. (2014) also observed a decrease in turbidity after microfiltration of passion fruit juice. Decrease in turbidity enhances juice commercialization due to lower decantation of suspended solids. The applied analytical method did not detect lipids in the permeate juice and showed that the microfiltration process was able to reduce significantly the lipid content in the raw pulp. Reduction may contribute towards the nutritional characteristics of açai juice, featured as a high fat food. Cesar et al. (2014) showed that the use of pectinolytic enzymes associated to chitosan as a clarifying agent for açai resulted in a clear lipid-free juice.

Table 1. Physical and chemical characteristics of açai pulp before (feed) and after (retentate and permeate) microfiltration process.

	Feed	Retentate	Permeate
Moisture ($\text{g } 100 \text{ g}^{-1}$)	91.83	98.68	82.57
Total solids ($\text{g } 100 \text{ g}^{-1}$)	8.17	1.32	17.43
Ashes ($\text{g } 100 \text{ g}^{-1}$)	0.32	0.33	0.24
pH	4.01	4.04	4.03
Lipids ($\text{g } 100 \text{ g}^{-1}$)	4.29	8.08	0.0
Turbidity (NTU)	87200	74970	13.9

Table 2 presents the results of microbiological analysis for feed and permeate açai pulp samples.

Table 2. Microbiological analysis for feed and permeate açai pulp samples.

Samples	24 hours		30 days	
	Total coliforms (MPN mL ⁻¹)	Mold and yeast (CFU mL ⁻¹)	Total coliforms (MPN mL ⁻¹)	Mold and yeast (CFU mL ⁻¹)
Feed	0.36	1.0 x 10 ¹	n.m.	n.m.
Permeate (room temperature, 25°C)	< 0.3	2.0 x 10 ¹	< 0.3	2.0 x 10 ¹
Permeate (room temperature, 25°C) + 0.01% benzoic acid	< 0.3	< 1.0 x 10 ¹	< 0.3	< 1.0 x 10 ¹
Permeate (room temperature, 25°C) + 0.01% sodium benzoate	< 0.3	< 1.0 x 10 ¹	< 0.3	< 1.0 x 10 ¹
Permeate (refrigerated, 5°C)	< 0.3	< 1.0 x 10 ¹	< 0.3	< 1.0 x 10 ¹

n.m.: not measured.

According to Brazilian requirements for juices and drinks, mesophilic bacteria counts must be less than 10² MPN mL⁻¹, but there are no Brazilian specific requirements for molds and yeast. Molds and yeast and mesophilic bacteria counts were less than 10 CFU mL⁻¹ and total coliforms were less than 0.3 mL⁻¹ for all stored açai permeate samples. The microfiltration process reduced total coliform concentration of açai pulp after 1 or 30 days of storage for all permeate samples (at room temperature or refrigerated, with or without the addition of conservatives). Mold and yeast concentrations of permeate samples stored at room temperature during 1 and 30 days increased after the microfiltration process. Increase may be associated to the manipulation of the sample or the spores may have crossed through the membrane. Since this sample was stored at room temperature, it is possibly that the favorable conditions of substrate, temperature and humidity triggered mold and yeast development. However, increase did not occur when permeate samples were stored at 5°C (refrigerated) and when conservatives were added. There was no proliferation of bacteria, mold and yeast in any sample and, thus, the microfiltration process ensured the required product stabilization.

Considering the nutritional composition of the fruit pulps, it may be presumed that they are able to sustain the growth of bacteria, molds and yeasts. However, pH is lower than the range that favors bacterial growth, or has rates below that at which bacterial growth is possible. In the case of açai pulp, Brazilian law establishes minimum and maximum pH rates respectively at 4.0 and 6.2.

Campos et al. (2002) verified shelf-life stability of cashew apple juice clarified by microfiltration using a 0.3 µm pore size polyethersulfone membrane at 200 kPa of transmembrane pressure and temperature of 30°C. According to their results, the microfiltration process stabilized cashew apple juice. Carneiro et al. (2002) showed that the microfiltration process produced a clarified pineapple juice that fulfills the requirements of microbiological safety, whilst Oliveira, Doce, and Barros (2012) showed that the microfiltration process was as efficient as pasteurization to treat passion fruit juice.

Conclusion

The proposed microfiltration process was able to produce a stabilized açai pulp, free from microorganisms and suspend solids. The addition of conservative agents (sodium benzoate or benzoic acid at a concentration of 0.01 wt%) or even storage at refrigerate conditions (5°C) improved reductions in mold and yeast concentrations of permeate açai samples. Reduction in turbidity was greater than 99%. This clarified pulp may be mainly used in carbonated beverages and sports drink liqueurs, in which the presence of suspended solids is not desired. Lipid reduction was equal to 100% and this reduction may contribute towards the nutritional characteristics of açai juice, considered a high fat food.

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