FOOD AND BIOPRODUCTS PROCESSING 95 (2015) 7–18

Contents lists available at ScienceDirect

Food and Bioproducts Processing

journal homepage: www.elsevier.com/locate/fbp

Valorization of Nixtamalization wastewaters (Nejayote) by integrated membrane process

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ARTICLE INFO

Article history:
Received 27 November 2014
Received in revised form 16 February 2015
Accepted 14 March 2015
Available online 22 March 2015

Keywords:
Nejayote
Sugars
Phenolic compounds
Microfiltration (MF)
Ultrafiltration (UF)

ABSTRACT

The purpose of this study was to analyze the potentiality of an integrated membrane process for the treatment of Nixtamalization wastewaters (well known as Nejayote). In particular, a sequence of one microfiltration (MF) pre-treatment step followed by two ultrafiltration (UF) processes was investigated on laboratory scale operating in selected process conditions. The performance of selected membranes in terms of productivity, fouling index, and water permeability recovery was evaluated and discussed.

The produced fractions were analyzed for their total soluble solids (TSS), total solids content (TSC), pH, electrical conductivity, turbidity, total polyphenols, total carbohydrates and total organic carbon (TOC). The rejection toward compounds of interest was evaluated. On the basis of experimental results, an integrated membrane process for the treatment of Nejayote was proposed. The conceptual process design permitted to achieve three streams as valuable products: a fraction enriched in carbohydrates, a fraction with high content of calcium components and clear fraction enriched in phenolic compounds. The obtained solution enriched in carbohydrates is of interest for preparing formulations to be used in food industry. Besides, the solution enriched in polyphenols can be used in cosmetic or pharmaceutical applications. Finally, the integrated membrane process used in this study can be used to fractionate Nixtamalization wastewaters as well as avoid the water and environmental pollution by the effluent.

1. Introduction

The maize (*Zea mays*) processing industry generates large amounts of wastewaters due for Nixtamalization process as pretreatment, which is the basis of the commercial methods to produce products such as instant corn flours, tortilla and other corn-based foods (Rojas-Molina et al., 2008, 2009; Gutiérrez et al., 2007). In common process, the maize grains are cooked in a saturated calcium hydroxide solution; the grains are separated from the alkaline solution in order to obtain the Nixtamal product (Valderrama-Bravo et al., 2012). The alkaline solution is well known as Nejayote; within this extract several parts of the grain are lost such as pericarp, germ, dietary fiber and endosperm (Acosta-Estrada et al., 2014). Nixtamalization process is carried out in Mexico, Central America, United States and parts of Europe, a traditional process requires a large volume approximately, 75 L of water is used to process 50 kg of corn kernels, meaning that a similar amount of alkaline wastewater is produced (Niño-Medina et al., 2009). Salmeron-Alcocer et al. (2003) reported that a production of 600 Ton corn/day generates between 1500 and 2000 m^3 of Nejayote, the estimated monthly volume generated in Mexico is about 1.2 millon m^3.

This wastewater contains significant amounts of soluble and insoluble solids consisting of organic and inorganic compounds, the physicochemical properties of Nejayote

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http://dx.doi.org/10.1016/j.fbp.2015.03.006

reported by Valderrama-Bravo et al. (2012) are pH (11.39), total solids (2.28%), ash (0.767%), carbohydrates (0.862%), electrical conductivity (4510.12 μS cm^{-1}), total suspended solids (8342.5 mg L^{-1}), turbidity (963.3 NTU) and chemical oxygen demand (40058.14 $mgO_2\,L^{-1}$). However, it has been reported the presence of bioactive compounds in this wastewater as carbohydrates such as arabinoxylans (Niño-Medina et al., 2009; Ayala-Soto et al., 2014), dietary fiber (Acosta-Estrada et al., 2014) and polyphenols (Gutiérrez-Uribe et al., 2010; Lopez-Martinez et al., 2009) such as hydroxycinnamic, p-cumaric, feluric, dehydrodiferulic and dehydrotriferulic acids (Ayala-Soto et al., 2014), but these authors do not describe the method used for separating these chemical compounds from the flowing liquid. Taking into account the characteristics and its chemical composition of this alkaline wastewater, there is strong evidence that the effluent contribute to the water and environmental pollution due to its high biological oxygen demand (BOD) and chemical oxygen demand (COD) (Gutiérrez-Uribe et al., 2010). Nejayote is directly discarded with additional water treatment cost with environmental laws but there are few studies about agro-industrial applications. The extract has been treated to obtain raw material employed as an ingredient to improve boiler foods (Velasco-Martínez et al., 1997), to enrich bread (Acosta-Estrada et al., 2014) or tortillas (Gutiérrez-Uribe et al., 2010).

The water pollution by this wastewater can be avoided trying to recover components from the water stream. Until now, the treatment of Nejayote by membrane operations has been no reported and less has no used as via of recovery of components derived from the wastewater. Microfiltration (MF) and ultrafiltration (UF) processes have been successfully employed for the recovery, purification and concentration of bioactive compounds from different wastewaters such as artichoke wastewaters (Conidi et al., 2014), orange press liquor (Conidi et al., 2012), olive mill wastewaters (Cassano et al., 2013) and winery sludge (Galanakis et al., 2013).

In this work, membrane operations are proposed as an alternative for the treatment of Nejayote. An integrated membrane process based on the use of MF and UF processes was investigated in order to fractionate the Nejayote extract and exploit their high molecular weight components such as suspended solids, calcium components, polyphenols and carbohydrates. In particular, the Nejayote extract was submitted to a preliminary cross-flow MF process in order to recover parts of the grain (fiber, endosperm, pericarp) and suspended solids of high molecular weight. The MF permeate was then processed by two different UF membranes; a concentrated solution in carbohydrates was obtained by 100 kDa membrane, the permeate was then submitted to a 1 kDa membrane, a concentrated solution rich in calcium components was produced and finally, a clear solution enriched in polyphenols was obtained. The rejection toward compounds of interest was evaluated.

The performance of the selected membranes was also determined in terms of productivity, fouling index and recovery of hydraulic membrane permeability.

2. Materials and methods

2.1. Solutions and reactants

2.1.1. Preparing the extracts

To prepare Nejayote, Nixtamalization was carried out as follows: total of 5 kg of corn was cooked during 38 min at 92 °C in a solution prepared with 50 g of calcium hydroxide (Nixtacal-Mexico) dissolved in 10 L of water. Then corn kernels were rested during 12 h; the Nejayote (cooking liquor) was drained by decantation. The extract was then stored at −17 °C until used.

2.2. Experimental set-up and procedures for the treatment of Nejayote by membranes

2.2.1. Selection of the feed flow rate condition in MF and UF membranes

In order to find the optimal feed flow rate of Nejayote in the membranes. The influence of the feed flow rate on permeate flux at 25 °C at optimal transmembrane pressure was analyzed. Same experiments were carried out for all membranes according to the procedure described by Cassano et al. (2007), the maximum permeate fluxes were found at 58 L h^{-1} for all membranes.

2.2.2. Microfiltration treatment

Nejayote was submitted to a preliminary MF process in order to eliminate suspended solids and high molecular weight compounds to reduce fouling phenomenon in the next UF processes. MF experiments were performed using laboratory unit, the MF unit was equipped with a polysulfone hollow fiber membrane module (Amersham Biosciences Corp., Model CFP-1-E-4A, USA) with nominal pore size of 0.2 μm.

The MF system was operated at a TMP of 1.3 ± 0.03 bar, at an axial flow rate (Q_f) of 58 L h^{-1} and a temperature of 25 ± 1 °C according to the batch concentration mode (recycling the retentate stream and collecting separately the permeate). Permeate fluxes were measured up to volume reduction factor (VRF) of 7. The VRF was defined as the ratio between the initial feed volume and the volume of resulting retentate according to the following Eq. (1) (Garcia-Castello et al., 2011):

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

where V_F, V_p and V_r are the volumes of feed, permeate and retentate, respectively.

2.2.3. Ultrafiltration treatment

Permeate obtained from MF was submitted to a UF step. The UF step was performed using a laboratory unit with feed tank, and peristaltic pump, a water bath at constant temperature was used to avoid significant increasing in the fluid temperature, a manometer and pressure-regulating valve. The UF unit was equipped with two different polysulfone hollow fiber membranes (Amersham Biosciences Corp., Model UFP-100-E-4A & UFP-1-E-4A, USA) of 100 and 1 kDa. The specifications of the membranes are reported in Table 1. UF experiments were carried out according to the recirculation configuration at an operating temperature of 25 ± 1 °C at different transmembrane pressures (0.3, 0.6, 1.0, 1.3 and 1.7 bar) in order to find the optimal pressure that provides the limiting flux for carrying out batch concentration configuration.

The experimental runs were performed according to the batch concentration mode up to a final VRF of 6, at TMP of 1.3 bar and VRF of 5, at TMP of 1.7 bar for 100 and 1 kDa membranes, respectively. The system was operated at a temperature of 25 ± 1 °C. The feed flow rate was fixed at 58 L h^{-1}.

For MF and UF steps, the permeate flux was measured gravimetrically as the change of permeate weight with time