Journal of Food Engineering 180 (2016) 120–128

Contents lists available at ScienceDirect

Journal of Food Engineering

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

Clarification and concentration of oligosaccharides from artichoke extract by a sequential process with microfiltration and nanofiltration membranes

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ARTICLE INFO

Article history:
Received 28 September 2015
Received in revised form
20 February 2016
Accepted 27 February 2016
Available online 3 March 2016

Keywords:
Cynara scolymus
Industrial waste
Prebiotic sugars
Fouling mechanism
Membrane process

ABSTRACT

Artichoke solid waste is considered a cheap fructooligosaccharides source. The extract from this material contains suspended solids, macromolecules and prebiotic sugars. The prebiotic sugars need to be clarified, purified and concentrated to increase its value. In this study, we evaluated the technical feasibility of sequential microfiltration (to clarify the artichoke extract) and nanofiltration (to purify and concentrate the prebiotic sugars). The investigation was carried out in laboratory scale, using different microfiltration and nanofiltration membranes subjected to different operation pressures. Microfiltration was able to clarify the extract with almost 100% of prebiotic sugars recovered in permeate. Microfiltration processes presented flux decline (the final flux was between 60 and 80% of the initial one) caused by cake layer formation, as observed by surface analysis. The membrane that presented the lowest flux decline and less solids deposition on its surface (polyethersulfone with a pore size of 0.05 μm), had its permeate subjected to nanofiltration. In this last step, 100% of retention of prebiotic sugar was obtained, mainly using polyamide membrane with molecular weight cut off of 150–300 Da. The sequential process allowed to produce a concentrate fraction can be used as an ingredient on foodstuff applications.
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1. Introduction

Artichoke (*Cynara scolymus* L.) is cultivated for its immature inflorescence, in which the head flower composes the edible portion. The global production in 2012 was of 1,634,219 tonnes (FAOSTAT, 2012) and the main producers are Egypt, Italy and Spain. It is widely consumed as fresh, frozen or conserved vegetable. As only small part of this vegetable (around 30%) is used in the food industry, artichoke processing generates a huge amount of solid waste (mainly bracts of the raw material) that is used as animal feedstuff or manure (López-Molina et al., 2005). Nowadays, attempts have been made to reuse waste solid material as a source of health-promoting compounds, generating better management of industrial residue and economic benefits for the agricultural and food sector (Goula and Lazarides, 2015; Castro-Muñoz and Yánez-Fernandez, 2015).

Among the bioactive compounds found in artichoke, the fructooligosaccharides (FOS) are functional sugar with confirmed health benefits, mainly on the digestive system. It is known as prebiotic oligosaccharides (Gibson and Wang, 1994). These sugars are one of the prebiotic oligosaccharides widely commercialized in the world market and are used as ingredient in several products, such as biscuits, drinks, yogurts, breakfast cereals, and sweeteners.

FOS can be obtained by an extraction method. In our previous work (Machado et al., 2015), we recovered prebiotic sugar from artichoke bracts using ultrasound-assisted extraction in water medium. The sugars identified in the selected extraction condition (60 °C of temperature, 360 W of ultrasound power and 10 min of process) were fructose, glucose, sucrose (non prebiotic sugars), kestose, nystose, fructofuranosyl-nystose (FOS) and raffinose (prebiotic oligosaccharide).

This extract, as many others and products obtained by FOS synthesis (from transfructosylation reaction with sucrose), was not composed only of prebiotic sugars. However, the product added value increases with prebiotic sugar purification. Because of this,

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http://dx.doi.org/10.1016/j.jfoodeng.2016.02.018
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researchers have sought methods for purification and concentration of these compounds (Goulas et al., 2002; Montañés et al., 2012; Zhao et al., 2013; Kuhn et al., 2014; Moreno-Vilet et al., 2014).

Membrane filtration technology has been used to clarify, fractionate and concentrate several raw materials. These processes offer several advantages, such as consuming less space, requiring less energy, absence of phase transition and high quality of product, preserving the natural flavor and nutritional components of raw material. Microfiltration processes have been applied for the clarification of different products, such as Nixtamalization wastewaters (Nejayote) (Castro-Muñoz and Yáñez-Fernández, 2015), passion fruit juice (Domingues et al., 2014), raw rice wine (Li et al., 2010) and açai pulp (Machado et al., 2012). This step can eliminate the suspended solids and stabilizes the functional and microbiological characteristics of the product. However, the disadvantage is that suspended solids, as well as macromolecules in the raw material, can cause concentration polarization and fouling, reducing the permeate flux during filtration and, consequently, losing process productivity (Cheryan, 1998). Therefore, it is necessary to study the fouling during the microfiltration in order to get higher permeate flux and greater extract clarification.

As the next step in the valorization process, nanofiltration of clarified extract can be employed. Nanofiltration process has been proposed by many authors as a method for oligosaccharides purification and concentration (Feng et al., 2009; Goulas et al., 2002; Michelon et al., 2014; Moreno-Vilet et al., 2014; Zhao et al., 2013). However, these authors used model sugar solutions (made of water, non-prebiotic and prebiotic sugar) to study the filtration, because the nanofiltration process on the food solution (such as juice and extract) is complex. As carbohydrates are neutral molecules, their separation is based on differences in molecular size (from 180 to 828 g/mol) and convective and diffusion phenomena (Goulas et al., 2002). Furthermore, when natural solutions are used, the permeate flux and rejection parameter in the membrane filtration can also be strongly affected by feed composition, membrane type, pressure, etc. Depending on feed composition, interactions between solutes can occur (protein and saccharides, for example). They can also occur between these solutes and the membranes employed, causing differences in rejection (Hwang and Chiang, 2014; Susanto et al., 2008; Yao et al., 2010). Thus, it is important to evaluate different nanofiltration membranes to study the separation and concentration of oligosaccharides from clarified extract.

A sequential membrane process using microfiltration and nanofiltration was evaluated to clarify the artichoke extract and to separate and concentrate the oligosaccharide from it.

2. Material and methods

2.1. Preparation of raw artichoke industrial waste extract

Artichoke waste (bracts) originated from canning industry was collected from the Bonsucesso factory (Sao Roque, Sao Paulo, Brazil). The extract was prepared according to Machado et al. (2015). Frozen artichoke bracts were homogenized with distilled water at 100 °C, at a ratio of bracts to water of 1:40 (g/mL), using an industrial blender during 15 s. The suspensions obtained were immediately placed in a jacketed reactor and a sonication probe (DES500 model, Unique, Indaiatuba, Sao Paulo, Brazil) was fitted into the reactors with its tip dipped into the suspension. The process was carried out at 60 °C of temperature, 360 W of ultrasound power, for 10 min. After, samples were filtered through a sieve (80 mesh) to eliminate large particles. The extract volume obtained was homogenized, divided into small fractions and stored at −18 °C until the process were performed. Table 1 shows the composition of raw artichoke extract.

Table 1
Composition (g/100 g) of raw artichoke waste extract.

Analysis	Mean ± SD
Moisture (f.w.)	99.69 ± 0.05
Ash (d.w.)	7.14 ± 0.97
Protein (d.w.)	28.94 ± 3.05
Lipids (d.w.)	7.97 ± 0.45
Dietary fiber (d.w.)	5.81 ± 1.13
Total carbohydrates (d.w.)	55.85 ± 1.50

f.w.: fresh weight; d.w: dry weight.
Data presented correspond to an average of five determinations, with standard deviations.

2.2. Sequential membrane process

In the present work, a sequential membrane process using microfiltration and nanofiltration was evaluated in order to purify and concentrate the prebiotic sugars from artichoke extract. The first one, microfiltration, was carried out for suspended solids reduction, with the purpose of extract clarification, oligosaccharides recovery in permeate fraction, and fouling reduction during the next filtration step. The second one, nanofiltration, was developed in order to study the separation of oligosaccharides from their mixture with non prebiotic sugars, and also the concentration of the first compounds, as is shown in Fig. 1.

For both processes, the membranes evaluation was performed in a dead-end filtration cell. The filtration cell consists of a cylinder jacket of stainless steel. Its useful volume is 800 mL, the effective membrane area is 50.24 cm^2 and the maximum operational pressure for this cell was 40 bar. The cell was pressurized using nitrogen.

2.2.1. Microfiltration procedure of raw extract

For extract clarification, the raw extract was subjected to microfiltration process using two flat sheet membranes, which are described in Table 2. Before each trial, the virgin membrane was conditioned for a period of 12 h at room temperature (25 °C).

Experimental runs were performed at 1, 2 and 3 bar, with fixed temperature and stirring rate at 25 ± 2 °C and 600 rpm, respectively. During the process, permeate from the bottom of the cell was continuously collected and its volume measured. Permeate was removed until obtaining a concentration factor (CF) of around 4.0. The CF is calculated according to Eq. (1):

$$CF = \frac{m_f}{m_c} \tag{1}$$

where m_f is the total volume used in the feed (g) and m_c is the volume collected in the concentrate fraction (g).

A curve J versus t was obtained for each run; the permeate flux to build the curve was calculated in time intervals according to Eq. (2).

$$J = \frac{Vp}{t \times A} \tag{2}$$

where J is the permeate flux (L/m^2 h), A is the effective membrane area (m^2) and V_p is the permeate volume collected (L) during the filtration time t (h).

After these processes, the membrane fouling was investigated by fouling resistance, blocking mechanisms models during filtration and analysis of membrane surface by atomic force microscopy (AFM). The feed and permeates were analyzed for color characteristics, clarity, total and soluble solids and saccharides content. For these last compounds, the results were showed as the retention

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