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Kaolin hollow fiber membranes for efficient bixin concentration from annatto seeds

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Introduction

The restriction on the use of synthetic dyes in the food industry leads to an interest in replacing these dyes with natural ones [1]. The use of carotenoids stands out as an alternative to chemical additives in food, which includes annatto-based dyes [1-2]. Bixin is the primary natural pigment of the annatto seed (*Bixa orellana L.*) and solid: liquid extraction is usually applied to obtain bixin extracts for further applications. The membrane separation process is a promising green technology and is effective in purifying bioactives present in fruit and plant extracts, and have been applied in several areas, including chemical, food and biotechnology industries [3]. The application of ceramic membranes is advantageous mainly in terms of chemical and thermal stabilities. Also, the use of hollow fiber membranes is suggested to obtain large permeation volumes in small filtration units. Ceramic hollow fibers with asymmetric pore size distribution are recommended to obtain high permeation fluxes. The use of low-cost ceramic materials, such as kaolin, is suggested in the literature to produce ceramic membranes [3]. Here we innovatively applied kaolin hollow fiber membranes with asymmetric pore size distribution to concentrate the bioactive compounds from the ethanolic extract of annatto seeds.

Material and methods

Annatto seeds were obtained in the city of Uberlândia - MG and stored in dark-colored polyethylene bags in a refrigerator (at -5 °C) for further analysis and processing. The extraction process was carried out using water and ethanol (50:50, volumetric basis) as solvent at a seed:solvent ratio of 1:30 (mass of seeds (g): solvent volume (mL)) at 60°C for 60 min in a Soxhlet apparatus, as indicated in the literature [4].

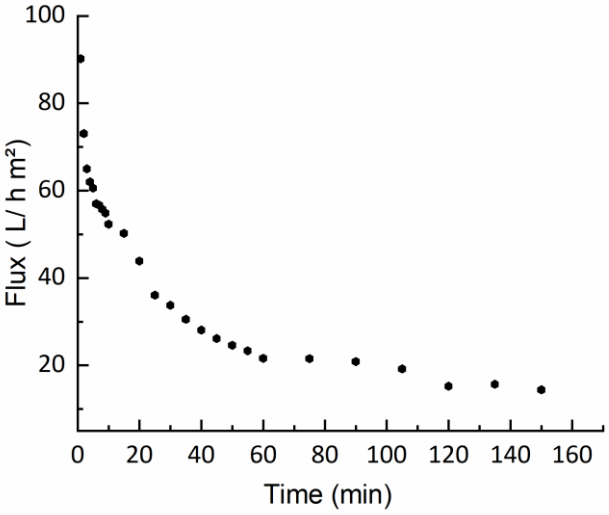
Kaolin hollow fibers with asymmetric pore size distribution were produced according to the phase inversion process followed by single sintering step. The detailed procedure to produce the kaolin hollow fibers is presented by [3] . Briefly, the ceramic powder (kaolin, 34.58 wt%) was mixed with the polymer (polyether sulphone, 9.88 wt%), the solvent (dimethyl sulfoxide, 55.17 wt%) and the additive (Arlacel®, 0.37 wt%). The ceramic suspension was extruded in a tube-in-spinneret with water as bore fluid and discharged in a water coagulation bath. After extrusion, the hollow fiber precursors were sintered at 1250°C in a tubular furnace (Carbolite, model TZF 15).

For the extract filtration, the hollow fiber membrane with a filtration area of 1.7 $x10^{-5}$ m^2 was assembled in a filtration module. The feed stream was displaced by a diaphragm pump to the membrane shell side and the permeate was collected from the membrane lumen side. The retentate stream was continuously returned to the feed tank. The cross-flow filtration was carried out at room temperature (approximately 25°C) and transmembrane pressure of 1.5 bar up to a concentration factor of 1.5.

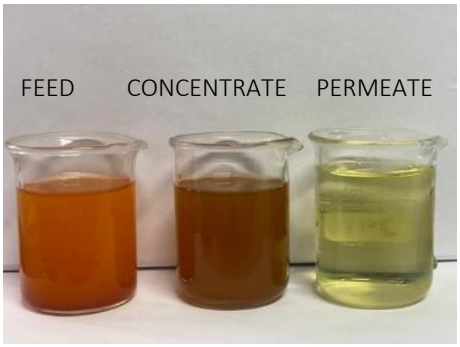
Feed, permeated and concentrated extracts were dried under vacuum in a rotatory evaporator (IKA HB100) for further characterizations. For bixin concentration measurements, the dried extract was diluted in acetone and the absorbance was verified in a spectrophotometer (Shimadzu UV-1280). The bixin concentration was calculated according to Lambert-Beer law with extinction coefficients ($E^{1\%}_{1cm}$) of 3090 $(g/100\ mL)^{-1}\ cm^{-1}$ for bixin in acetone, as reported by [5]. The total phenolic content in the extracts was determined by the Folin-Ciocalteu method [6].

Results and discussion

Figure 1(a) presents the flux decay data during the filtration of the annatto seed extract through the kaolin hollow fiber membrane. A pronounced flux decay was observed in the first 40 min of filtration and the steady stated flux was recorded at approximately 20 L h^{-1} m^{-2}. Magalhaes et al. [3] reported initial and steady state permeate fluxes of approximately 90 and 20 L h^{-1} m^{-2}, respectively, during the filtration of an aqueous bacteria solution though kaolin hollow fibers. Miguel et al. [7] applied a commercial polymeric ultrafiltration membrane for the concentration of annatto seed extracts and obtained a steady state flux of 24.02 ± 0.16 kg h^{-1} m^{-2}. Thus, the application of kaolin hollow fiber membranes is suggested as alternative to use a low-cost membrane with suitable permeate fluxes.



(a)



(b)

Figure 1. (a) Permeate flux for the filtration of annatto seed extract through the kaolin hollow fiber. (b) Visual appearance of feed, permeate and concentrate streams.

Figure 1(b) shows the visual appearance of the feed, concentrate and permeate samples. The permeate sample is clearer than the feed and concentrate samples, which indicated that most of the carotenoid compounds were retained by the ceramic membrane. Also, some specific carotenoid compounds were permeated though the membrane so that the concentrate sample is brownish, while the feed and permeate samples present a dark- and light- yellow color.
The bixin content values of the feed, concentrate and permeate annatto extract samples were 2.22 ± 0.004, 3.01 ± 0.003 and 0.03 ± 0.001%, respectively. Similar results were observed by [4-8] for annatto seed extracts. The total phenolic content values of the feed, concentrate and permeate annatto extract samples were 2.07 ± 0.003, 2.53 ± 0.007 and 0.74 ± 0.003 mg_{GAE}/(g of extract), respectively. Quiroz et al. [5] reported similar values of total phenolic contents of annatto seed extracts. The kaolin hollow fiber membrane was able to concentrate 26% of the bixin molecules and 18% the phenolic compounds from the feed extract.

Conclusion

In this study, the efficiency of the filtration process was tested using a kaolin membrane to concentrate bioactive compounds present in the ethanolic extract of annatto seeds. The extract showed significant contents of bixin, an important carotenoid, phenolic compounds. Bixin and phenolic compounds were concentrated in 26 and 18%, respectively, after the filtration of the annatto seed extract through the kaolin hollow fiber membrane. Thus, this low-cost ceramic membrane is suggested to be used for the concentration of bioactive compounds from annatto seed extract.

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