17th International Conference on Inorganic Membranes
New Frontiers for Inorganic Membranes
July 1 - 5, 2024
Oceania Convention Center - Florianópolis - Brasil

Performance of asymmetric spinel hollow fiber for clove basil aqueous extract clarification

K. R. Dorneles[a], G. G. Ascendino[a], W. A. Dias[a] and M. H. M. Reis[a*]
[a] Department of Chemical Engineering, Federal University of Uberlândia, Uberlândia, Brazil
[*]miria@ufu.br

Introduction

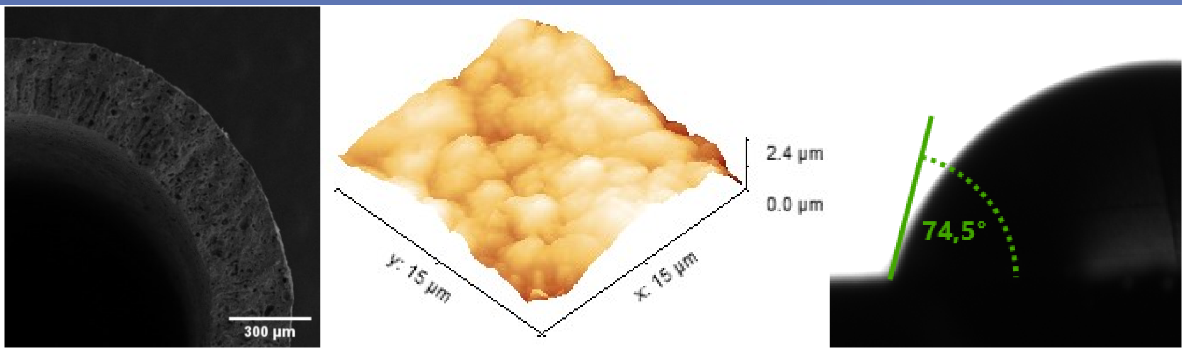
The increasing demand for functional food calls attention to new sources of natural ingredients, like underutilized plants. These species show significant sources of proteins, micronutrients and health-promoting compounds [3, 5]. Clarification of aqueous extracts containing phenolic compounds is a key stage in food processing. It impacts the stability of the extract, reduces suspended solids content and turbidity and ensures polyphenol availability [2, 5]. The use of ceramic membranes for extract purification is suggested due to their thermal, mechanical and chemical stability as well as a high packing density and a low energy demand [2]. In this work, we explored the application of a spinel hollow fiber membrane to clarify a crude clove basil aqueous extract.

Material and methods

Spinel hollow fibers were prepared according to the phase-inversion method, using alumina (42.8 wt.%), dolomite (14.3 wt.%), dimethyl sulfoxide (36.8 wt.%), polyethersulfone (5.7 wt.%) and polyvinylpyrrolidone (0.4 wt.%) to form the ceramic suspension. After extrusion, the hollow fibers were sintered in a tubular furnace at 1350 °C. The detailed procedure to fabricate the spinel hollow fibers is presented in [1]. The morphology of the hollow fibers was investigated using scanning electron microscopy (SEM, Tescan, model VEGA3) and atomic force microscopy (AFM, Shimadzu, model SPM-9600). The water contact angle of the membrane was verified using a drop-shape analyzer (Krüss, model DSA25E). The clove basil aqueous extract was prepared at a solid: liquid ratio of 10:1 (g of dried clove basil leaves: L of distilled water) and 75 °C under magnetic stirring for 120 min. The extract filtration was carried out using a single hollow fiber with a filtration area of 3.83×10^{-4} m^2 assembled in a permeation module. The fiber was compacted, and its hydraulic permeability was measured from 0.4 to 3.0 bar. Crossflow filtrations were performed at room temperature and 1.0 bar of transmembrane pressure. Permeate flow was measured during filtration. The characterization of the feed and permeate streams was done by their total phenolic content (TPC), expressed in mg of gallic acid equivalent per g of extract, and antioxidant potential (DPPH method), expressed in micromolar of Trolox equivalent per g of extract, using a UV-Vis spectrophotometer (Shimadzu, model UV 1280).

Results and discussion

SEM, AFM and water contact angle images of the spinel hollow fiber are grouped in Fig. 1. As presented in Fig. 1(a), subtle micro-channels and irregular micro-voids were formed through the fiber cross-section due to the contact of the ceramic mixture with the coagulant fluid in the phase-inversion stage [1, 4]. A dense sponge-like structure was formed on the fiber outer surface due to a slower phase-inversion process and the air-gap applied during extrusion [1, 4]. Some holes are observed in the fiber due to the reaction between alumina and dolomite that forms the spinel phase and other gas products [1]. The formed porous structures favored applications as liquid filtration membranes [1, 4]. According to the AFM analyses in Fig. 1(b), the roughness of the spinel hollow fiber was 125.26 ± 9.77 nm, which is in agreement with roughness values reported for ceramic hollow fibers in literature [1]. As shown in Fig. 1(c), the spinel hollow fiber is hydrophilic, with a water contact angle of 74.5°.



(a) (b) (c)

Fig. 1. (a) SEM, (b) AFM and (c) water contact angle of the spinel hollow fiber

Adjusting the water flux data to Darcy's law, we could determine the hydraulic permeability of the spinel hollow fiber as 90 $L \cdot h^{-1} \cdot m^{-2} \cdot bar^{-1}$, which is a little lower than values reported for ceramic hollow fibers [1, 2, 6]. The higher densification of the sponge-like layer seen in Fig. 1(a) could have decreased the membrane permeability but should be favorable for the membrane selectivity. During filtration, a significant flux decay up to 25 min was observed, and the estimated steady-state flux was 21.22 ± 0.89 $L \cdot h^{-1} \cdot m^{-2}$, which is similar to the report of [2] for filtrations of green tea extract though alumina hollow fibers.

Values for TPC and antioxidant potential (by DPPH method) of feed and permeate streams are shown in Table 1. Regarding the presence of phenolic compounds in the extract, values are comparable to those reported by [3, 6] for underutilized plants. After filtration, the permeate sample retained almost 60% of its phenolic content and more than 65% of its antioxidant potential. Retention of high-mass molecules allows both permeate and retentate to present antioxidant properties and other benefits.

Table 1. Phenolic compounds and antioxidant potential of clove basil aqueous extract before and after filtration

Sample	**TPC ($mg_{EAG} \cdot g^{-1}$)**	**DPPH ($\mu M_{ET} \cdot g^{-1}$)**
Feed	188.41 ± 6.99	899.88 ± 5.03
Permeate	110.10 ± 0.71	589.33 ± 11.06

Conclusion

The spinel hollow fiber is suitable for clarifying aqueous plant extracts, maintaining the antioxidant capacity and health-promoting compounds. Moreover, the fiber is porous, wettable and resistant, making it a valid alternative for industrial purposes.

References

[1] Bessa, L. P., Ferreira, E. de P., Magalhães, F. de S., Ferreira, F. B., Cardoso, V. L., & Reis, M. H. M., “Micro-structured and reinforced spinel hollow fiber membranes: Influence of sintering temperature and ceramic powder composition”, Ceram. Int., *45*(17), 23632–23642 (2019).

[2] Bindes, M. M. M., Terra, N. M., Patience, G. S., Boffito, D. C., Cardoso, V. L., & Reis, M. H. M., “Asymmetric Al_2O_3 and PES/Al_2O_3 hollow fiber membranes for green tea extract clarification”, J. Food Eng., 277 (2020).

[3] De Souza, T. C. L., Da Silveira, T. F. F., Rodrigues, M. I., Ruiz, A. L. T. G., Neves, D. A., Duarte, M. C. T., Cunha-Santos, E. C. E., Kuhnle, G., Ribeiro, A. B., & Godoy, H. T., “A study of the bioactive potential of seven neglected and underutilized leaves consumed in Brazil”, Food Chem., 364, 130350 (2021).

[4] Fung, Y. L. E., & Wang, H., “Nickel aluminate spinel reinforced ceramic hollow fibre membrane”, J. Membr. Sci., 450, 418–424 (2014).

[5] Gerke, I. B. B., Hamerski, F., Scheer, A. P., & Silva, V. R., “Clarification of crude extract of yerba mate (*Ilex paraguariensis*) by membrane processes: Analysis of fouling and loss of bioactive compounds”, Food Bioprod. Process., 102, 204–212 (2017).

[6] Paraíso, C. M., dos Santos, S. S., Pereira Bessa, L., Lopes, A. P., Ogawa, C. Y. L., da Costa, S. C., Reis, M. H. M., Filho, U. C., Sato, F., Visentainer, J. V., & Madrona, G. S., “Performance of asymmetric spinel hollow fiber membranes for hibiscus (*Hibiscus sabdariffa* L.) extract clarification: Flux modeling and extract stability”, J. Food Process. Preserv., 44(12) (2020).