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Solvent resistant nanofiltration based-process for production of steryl esters enriched extracts

A.R.S. Teixeira*, J.L.C. Santos, J.G. Crespo
Universidade Nova de Lisboa, Portugal

Deodorizer distillates are a residual stream produced in the last step of the refining of vegetable oils. Their high content in bioactive compounds (may vary between 2-20%w/w), such as sterols, steryl esters tocopherols and squalene, could make them a valuable source. However, deodorizer distillates have been discarded mainly due to their high content in pesticides.

The present work proposes a membrane-based process to valorise vegetable oil deodorizer distillates, producing a pesticide-free extract rich in bioactive compounds.

Based on the molecular weight of vegetable oil deodorizer distillates constituents (Figure 1), it is clear that the only bioactive compounds that are possible to be efficiently separated from pesticides are the steryl esters.

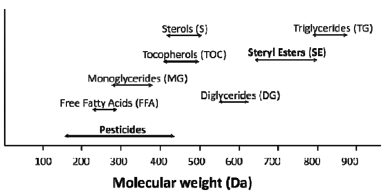

Figure 1. Molecular weight of vegetable oil deodorizer distillates constituents

The membrane-based process which is studied in this work involves three steps:

- Enrichment of deodorizer distillates in steryl esters, producing them by enzymatic esterification of FFAs and sterols (one of the advantages of producing steryl esters is related with their higher bioactivity comparatively to sterols, as well as their higher lipophilicity, making easier their incorporation in fat-based products);
- Recovery of the enzyme by ultrafiltration for its reuse in consecutive batch reactions;
- Production of a pesticides-free stream rich in steryl esters by dia-nanofiltration.

Concerning the first step, the enzymatic reaction using *Candida Rugosa* lipase was optimized in terms of concentration, initial FFAs:Sterol molar ratio and water activity, enabling a yield of steryl esters production in the range 80-90% in 24 hours [1].

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Subsequently, the enzyme was totally recovered by ultrafiltration using a ceramic membrane with a nominal pore size of 30nm (Inopor® Ultra). However, enzyme activity decreased in consecutive batch reactions. Results suggest that oxidation products are responsible for such decrease which indicates that a reduction in the enzyme activity in consecutive batches is inevitable, requiring an additional purification step or a make-up with fresh enzyme. This make-up strategy was optimized in order to assure high conversion yields and productivity with the lowest amount of enzyme added [2].

The last step comprises the production of a stream rich in steryl esters free of pesticides by dia-nanofiltration. This method was applied by continuously feeding oleic acid at the same rate as permeate is recovered. Steryl esters are mostly retained by the membrane while pesticides are washed-out in the permeate.

Several commercial solvent resistant nanofiltration membranes were tested at laboratory scale, using a dead-end system filtration cell. Their compatibility with oleic acid and efficiency in retaining steryl esters were compared under the same conditions of pressure and temperature (identical to the set-point used during enzymatic reaction at 40ºC). The influence of temperature and transmembrane pressure on the rejection and permeability was determined for the membranes with the best performance (030306 and 030306F from Solsep, NP1 from Borsig and S380 from Evonik). Figure 2 shows the results obtained for the membrane 030306F from Solsep in terms of steryl ester rejection at different temperatures and transmembrane pressures.

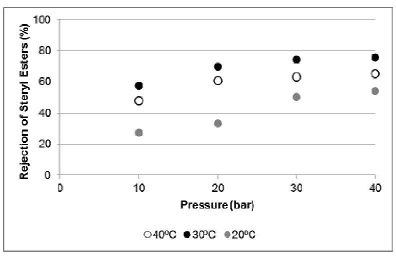

Figure 2. Rejection of steryl esters at different temperatures and transmembrane pressures for the membrane 030306F from Solsep.

The dia-nanofiltration process showed to be suitable for an efficient removal of pesticides, with a steryl esters rejection of 80% with Solsep membranes, while the rejection of pesticides was significantly lower, (between -37 and 15%).

The proposed dia-nanofiltration process was scaled-up to a pilot scale unit comprising a commercial spiral wound module. The mass transfer behaviour of the spiral wound module was assessed in a Sepa CF cross flow system (GE Osmonics, USA) both by the velocity variation method and by computational fluid dynamics (CFD). For that purpose, the same feed spacer from the spiral wound module was used in the Sepa CF system. The results were then be compiled in the form of a mass transfer correlation to be used for process optimization.

References:

[1] Teixeira, A. R. S.; Santos, J. L. C.; Crespo, J. G. Production of Steryl Esters from Vegetable Oil Deodorizer Distillates by Enzymatic Esterification. Industrial & Engineering Chemistry Research. 2011, 50, 2865–2875.

[2] Teixeira, A. R. S., Santos, J. L. C., Crespo, J. G., Lipase-Catalyzed Consecutive Batch Reaction for Production of Steryl Esters from Vegetable Oil Deodorizer Distillates, Industrial & Engineering Chemistry Research - (Accepted 18 Mars 2012)

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