Synthesis and characterization of alginate/inulin aerogel macrospheres using supercritical fluid technology

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ABSTRACT

Aerogel is defined as an open porous and light material, which is derived from a gel when the liquid is substituted by a gas. The interest in this class of materials has increased due to potential food, biomedical and pharmaceutical applications. Previous studies have shown that beads of alginate/inulin dried by freeze-drying have a high adsorption capacity towards phenolic compounds. Delayed release of the extract loaded into the beads and their disintegration during digestion was attributed to the key role of inulin. Based on that, the purpose of this work was to produce and characterize alginate/inulin aerogel macrospheres using supercritical CO_2 drying. Polysaccharide solutions were prepared with alginate (1, 2 and 3%, w/w) and inulin (0, 5, 10, and 15%, w/w). Then, the polymeric solution was dripped through a nozzle (0.56 mm) into a crosslinking solution containing calcium chloride (3.3 g/L). Once gelled the macrospheres (3.24 ± 0.06 mm) were collected and subjected to a solvent exchange, in which they were progressively immersed into ethanol/water mixtures (10, 30, 50, 70, 90 and 100%, v/v) for one day, except the last step that was repeated three times. Finally, the macrospheres were dried with a continuous supercritical CO_2 flow at 40 ± 1 °C and 12 ± 0.5 MPa. The surface and shape of the gel, alcogel and aerogel particles were examined by electron microscopy and the data were processed by ImageJ image software. The reduction of the particle size between the processes was calculated and expressed as relative shrinkage. The specific surface area of the macrospheres was determined by low-temperature nitrogen adsorption–desorption analysis using Brunauer–Emmett–Teller (BET) model. Moderate shrinkage was obtained by increasing the alginate and inulin concentration, in which 15% inulin halved the shrinkage when compared to pure alginate. Circularity was found to be larger than 0.75. The specific surface area raised with increasing alginate concentration, however, demonstrated a decrease with increasing inulin concentration. As this study is in progress, results for the adsorption of phenolic compounds will discriminate most promising formulations.

Keywords: porous material; supercritical fluid; polysaccharide; surface area.