Modifying the properties and the morphology of starch aerogels using cellulose fibers

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Aerogels are nanostructured lightweight mesoporous/microporous materials with high specific surface areas. Starch-based aerogels showed great potential as carriers for active compounds and controlled release, templates or making novel materials, packaging materials, thermal insulators, and adsorbents. The properties of starch-based aerogels are mainly tuned by the processing parameters (source of starch, biopolymer concentration, amylose/amylopectin ratio, and gelatinization degree). By combining other biopolymers with starch, an immense influence on the properties of the final materials can be made. [1,2]

This study aimed to investigate the effect of bacterial nanocellulose (BNC) and cellulose nanocrystals (CNC) addition on the properties and morphology of starch aerogels. The preparation of starch and composite starch-cellulose aerogels included hydrogel preparation with different BNC and CNC content, followed by solvent exchange and supercritical drying. A comprehensive characterization was conducted through N2 absorption-desorption analysis, thermogravimetry (TGA) and differential scanning calorimetry (DSC), scanning electron and ion microscopy (SEM-FIB), Fourier transform infrared spectroscopy (FTIR), and swelling behavior. Obtained aerogels were impregnated with xanthohumol (XH) from the ethanol solution. The polymeric structures loaded with XH were afterward exposed to supercritical carbon dioxide in the presence of usnic acid (UA) to perform one-step supercritical drying and impregnation.

All prepared aerogels encompassed high specific surface areas, ranging from 180-220 m²/g, depending on the BNC/CNC content. The higher the BNC/CNC content, the higher the surface area. Cellulose-starch aerogels were shown to be more thermally stable than neat starch aerogels. SEM imaging of surfaces and cross-sections revealed highly porous structures with interconnected networks of pores. Fig. 1 shows the cross-section of starch/CNC aerogel. The possible applicability of prepared aerogels was investigated through the swelling behavior over seven days. It was shown that all aerogels reached 250-550 % of the swelling ratio, depending on the BNC/CNC ratio. The higher the BNC/CNC content, the greater the swelling was.

The impregnation process was successful. It was shown that the addition of BNC/CNC improved the aerogel sorption capacity and, therefore, the XH intake (12.9 – 16.6 µg/g). The higher the BNC/CNC concentration, the higher the XH intake. The UA loading acquired in the next step was less affected by cellulose. Most importantly, post-impregnation characterization studies confirmed the preserved structure of starch and starch-cellulose aerogels loaded with active substances.

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