Pectin + lentil protein gels dried by supercritical CO_2

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1. Introduction

Recently, legume proteins like as lentil protein have gained much interest as natural surfactants/wall materials in encapsulation systems due to their widespread abundance, low allergenicity, low cost, nutritional benefits, and functional attributes. Pectin, a plant-based polysaccharide, has also been extensively used as an alternative functional material for delivery of bioactives. Emulsions, emulsion-filled gels/emulsion gels, and aerogels are some examples of encapsulating and delivery systems. Processes like high-intensity ultrasound (HIUS) has been used to produce kinetically stable emulsions and emulsion-filled gels[1,2]. Since the moisture content of the emulsion-filled gels is high, disintegration of the gel network might occur. Therefore, emulsion-filled hydrogels in dry form can provide an alternative delivery system to improve the shelf stability and bioavailability due to their pore structure, large surface area, and low density[3]. Drying techniques such as supercritical carbon dioxide (SC-CO_2) drying and freeze-drying have been used to dry hydrogels to form a porous structure[4]. SC-CO_2 drying is a green alternative approach known to form particles and aerogels with uniform porosity and improved textural properties. CO_2 is the most used fluid for the supercritical drying process due to its high diffusivity, low viscosity, inertness, and operation at low temperatures, which is critical for heat-sensitive components[3]. Pectin based aerogels have been formed by the researchers for its applications in packaging and control-release of bioactives. Most of the protein-based aerogels formed by the researchers are of animal sources. There is a growing interest towards plant-based foods and to cater those needs, we used lentil protein and pectin to form the aerogel matrix. The main objective of this research was to study the influence of pectin concentration on the physical characteristics such as microstructure, pore diameter, absorption capacity, density, and color of the aerogels.

2. Materials and Methods

2.1. Materials

Lentil protein concentrate was provided by AGT foods (Saskatoon, SK, Canada). Pectin (DE>78%) was purchased from Sigma Aldrich (Oakville, ON, Canada). Sunflower oil (100% purity) was purchased from a local store, No Frills (Edmonton, AB, Canada). CO_2 (99.9% purity) was purchased from Praxair Canada Inc. (Mississauga, ON, Canada). Ethanol and HPLC grade water were obtained from Fischer Scientific (Ottawa, ON, Canada).

2.2. Aerogel production by SC-CO_2

A laboratory-scale SC-CO_2 system was used to form the aerogels[5]. First, the emulsion-filled hydrogel was prepared by high-intensity ultrasound (HIUS) treatment following the method described by Mekala et al.[2]. The biopolymer solution was prepared using pectin (1,3 and 5% w/v) and lentil protein suspension in citrate buffer (1.5 g/100g). The emulsion-filled hydrogels were formed at varied concentrations of pectin:lentil protein (1:1 and 2:1 w/w respectively). Then, the water in the hydrogel system was replaced by ethanol to form an alcogel as described by Ciftci et al.[5]. These alcogels were then placed in a polypropylene container before loading it into the stainless-steel reactor. The operating conditions followed for drying process were temperature (40-60 °C), pressure (100-120 bar), CO_2 flow rate (0.5-1.0 mL/min) and time (45, 60, 120, 180, 240, and 360 min). After the drying process, the SC-CO_2 system was depressurized, and the samples were stored at 4 °C. The moisture content of the aerogels was calculated to establish the drying curve. The formed aerogels were characterized for their microstructure, pore diameter, absorption capacity, density, and color.

3. Results and discussion

Pectin can be grouped depending on the degree of esterification as low-methoxy pectin (DE<50%) and high-methoxy pectin (DE>50%) with different gelation mechanisms. In this study, high-methoxy pectin was used to evaluate its effect on aerogel physical stability, pore diameter and specific surface area. The pH of the pectin + lentil protein gels formed in this study was around 2.5 which was sufficient for form stable hydrogel structures. The emulsion-gelation method is an interesting alternative approach to traditional acidic and ionic gelation to form

protein + polysaccharide gels with enhanced applications in the form of sauces, yoghurts, salad dressings among many others[2]. The aerogels with pectin concentration of 3g/100 g and lentil protein concentration of 1.5g/100g in the ratio 1:1 w/w were named low pectin aerogels. The concentration of high pectin aerogels was pectin (5g/100g), lentil protein (1.5g/100g) in the ratio 2:1 w/w.

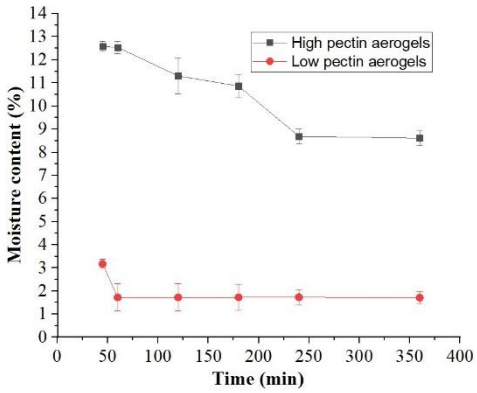

Figure 1. Moisture content of pectin + lentil protein aerogels

Figure. 2. Microstructure of pectin + lentil protein gels

Figure 1 shows the moisture content of the aerogels as function of drying time at 100 bar, 40° C, using a CO_2 flow rate of 1 mL/min up to 360 min. As the drying time increased, the moisture content of the gels decreased, being constant after 240 min. Comin et al.[3] observed the moisture content was constant after 4h for gels made with barley β-glucan, which was longer than pectin + lentil protein aerogels in our study. The drying time for low pectin concentration gels was 60 min while for high pectin gels was 240 min. The initial moisture content of low pectin aerogels was 3.17% at 45 min, decreased to 1.72% at 60 min and was constant after this time. Here, it can be observed that the concentration of pectin played a significant role in the drying of pectin + lentil protein gels. However, the hydrogel formed with low pectin concentration was not stable and the gel network deteriorated after 24 h of storage i.e., the syneresis observed was about 71-79%. Higher pectin concentration was responsible for formation of stronger gel network and the syneresis observed was in the range of 92-99%. Since the hydrogels formed with higher pectin percentages were compact, the solvent exchange of ethanol could not completely replace the water molecules present in the hydrogel, resulting in secondary gelation in ethanolic solution. Tkalec et al.[6] has formed high-methoxy pectin aerogels using ethanol-induced gelation mechanism followed by SC-CO_2 drying for 7 h. They observed the highest surface area (386 m^2/g) for aerogels formed at 4% pectin concentration compared to 1 and 2% pectin concentration. Hence, the high pectin concentration (5g/100g) in our study might have contributed to the high surface area and low shrinkage.

Figure 2 shows the SEM images of the gels (freeze-dried and SC-CO_2 dried). The images showed that the freeze-dried gels have sheet like structure while SC-CO_2 dried gels have aerated structure, indicating higher porosity for delivery systems of bioactive compounds and oils.

4. Conclusions

This study evaluated the potential of lentil protein and pectin as suitable plant-based matrices for the formation of aerogels. Aerogels and hydrogels produced with high pectin concentration were stable. Due to the ability of pectin to gel in ethanolic solutions, it exhibited a plasticizing nature and was responsible for increased drying time. The emulsion gelation method followed in this study to produce hydrogels can be tailored for delivery systems of both hydrophilic and hydrophobic bioactive compounds. The HIUS treatment was responsible for the formation of homogeneous gel structures. The aerogels produced in this study can be used in various applications such as fat replacers, yoghurts, and sauces.

References

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