Effect of starch-packing on alginate hydrogel carrier quality: Particle characterization

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Introduction

Hydrogel material is one of the most commonly used carriers to encapsulate sensitive bioactive compounds for preservation purpose. This is because the encapsulation process could be performed in a simple, mild and safe condition. In order to achieve better preservation and handling of the encapsulated bioactive products, freeze-drying is often used to produce dried particulates. During the drying process, sublimation of frozen water from the hydrogel carrier could leave void areas within the dried structure. As a result, the carriers may possess several undesirable qualities such as distorted shape, poor mechanical strength and high porosity. These inferior characteristics may not only cause difficulty in handling and processing, it may also affect the function and performance of encapsulation.

The objective of this work is to improve the quality of dried alginate hydrogel carrier by packing high concentration of starch granules inside alginate beads. The dried Starch-Packed Alginate Beads (SPAB) were produced by using simple extrusion technique and subsequently freeze-dried. The effect of starch-packing on particle characteristics of SPAB were investigated and discussed.

Material and Methods

Materials: Sodium alginate Manugel GHB (ISP Technologies Inc, UK) in medium range (mannuronic acid 37%, guluronic acid 63%), Corn starch (SIGMA-ALDRICH Inc, USA), calcium chloride (MERCK, Germany).

Preparation of alginate-starch beads: Alginate-starch solutions of 10 – 60% (w/v) starch concentration and 2% (w/v) alginate concentration were prepared. The homogeneously mixed solution was extruded through a needle and cross-linked with calcium chloride solution 1.5% (w/v). Needles of different sizes were used to obtain freeze-dried SPAB of uniform diameter. These beads were hardened for 30 minutes, rinsed with distilled water and then dried in a freeze dryer for 24 hours.

Characterization of beads: The size and shape of beads were characterized by diameter and shape factor (SF) respectively. The images of the beads were captured by using a digital camera and analyzed by Sigma Scan Pro5 image analyzing software. SF was used to indicate the sphericity of the beads, with a value of 1.0 indicating perfect sphere. Tapped density was determined by tapping the samples into a measuring cylinder until constant reading was obtained. Flowability of the beads was assessed by Hausner ratio by determination of tapped over bulk density value. Mechanical strength of the beads was determined by analyzing the force-displacement curves obtained from uniaxial compression tests. Distribution patterns of starch particles within the bead were observed from x-ray micro-computed tomography images. The x-ray images were further analyzed by using the TView program, where relative porosity of the beads was determined.

Results and Discussion

Figure 1 shows the size and shape of dried beads containing different starch concentration. The size of beads was successfully controlled at about 2.0mm by using the appropriate needle size. This is to eliminate the effect of particle size when characterizing other particle properties. As for the bead shape, it was found that the sphericity of the beads could be improved by incorporating starch granules. In contrast, the shape of ca-alginate beads without starch granules was distorted as there was no solid particle to support the hydrogel structure during drying (Figure 2).

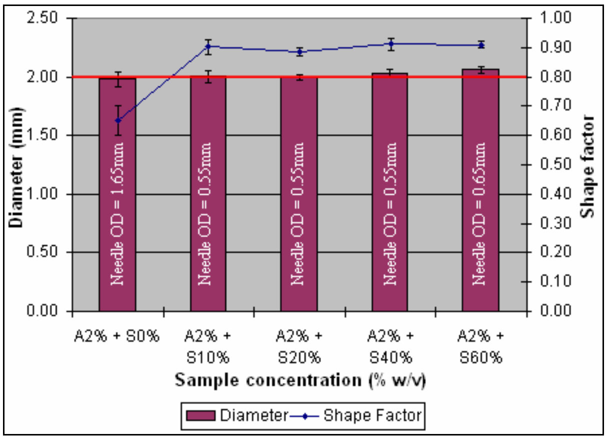

Figure 1: Size and shape of freeze-dried SPAB of varying starch concentrations

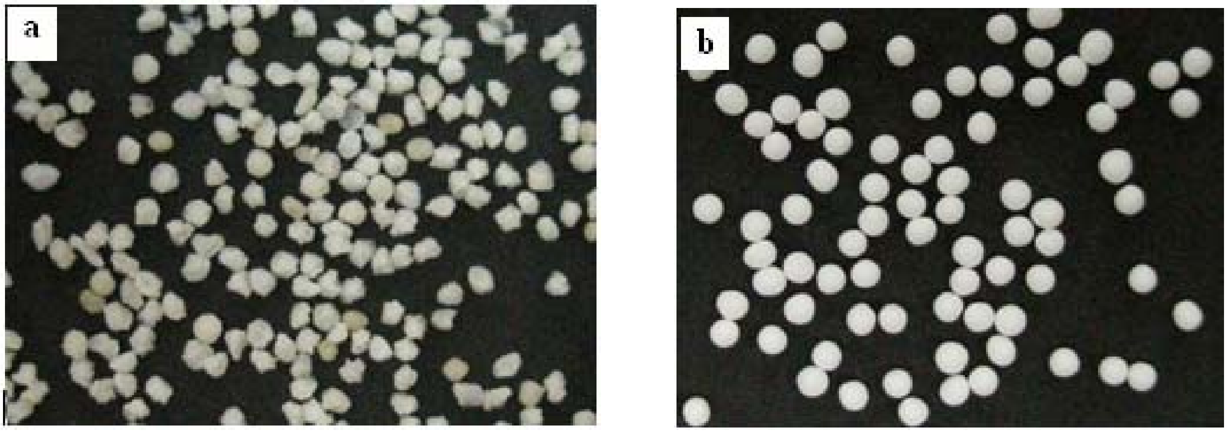

Figure 2: Freeze-dried SPAB a) Alginate beads without starch granules b) Starch-packed alginate beads

Figure 3 shows the bulk and tapped densities of beads with different starch concentrations. As expected, increasing starch concentration resulted in higher bulk and tapped density. Also, the bead tapped density was found to be slightly higher than the bulk density as tapping reduced the interparticle void spaces. On the other hand, it was found that all the beads were having Hausner value of less than 1.20 (as shown in Table 1), which is an indication of good flowability (Kumar *et al.*, 2001).

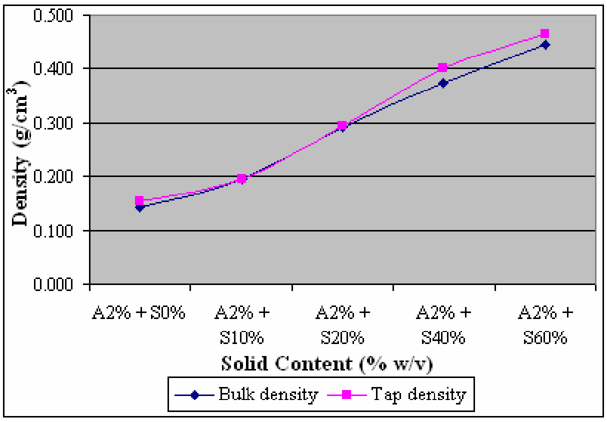

Figure 3: Bulk and tapped density at different starch concentration.

Concentration (% w/v)		Hausner ratio
Alginate	Starch	
2.0	0	1.024
2.0	10.0	1.000
2.0	20.0	1.017
2.0	40.0	1.070
2.0	60.0	1.049

Table 1: Hausner ratio of alginate-starch beads at different starch concentration.

Figure 4 shows the mechanical strength of beads at 10% deformation. It was found that the force required to cause 10% deformation of the beads increased proportionally with increasing starch concentrations of up to 40% w/v. No significant increase in mechanical strength was found beyond this concentration. The data suggests that the mechanical strength of dried alginate beads could be improved by approximately 10-fold by packing with starch granules. The results are in good agreement with previous works (Tal *et al.*, 1997). The increase in the mechanical strength could be explained by the role of starch granules that functioned as structure providers that filled the interstitial spaces between hydrogel networks as well as to reinforce the network structure. This speculation was verified by the x-ray micro-computed tomography images that show the distribution pattern of starch granules within the dried beads (Figure 5). It clearly shows that the beads without starch granules were distorted in shape and contained relatively higher void spaces. In comparison, starch-packed alginate beads were spherical in shape and with significantly less void spaces. As shown in Figure 6, the relative porosity of the alginate beads without starch could be three times more porous than the starch-packed alginate beads.

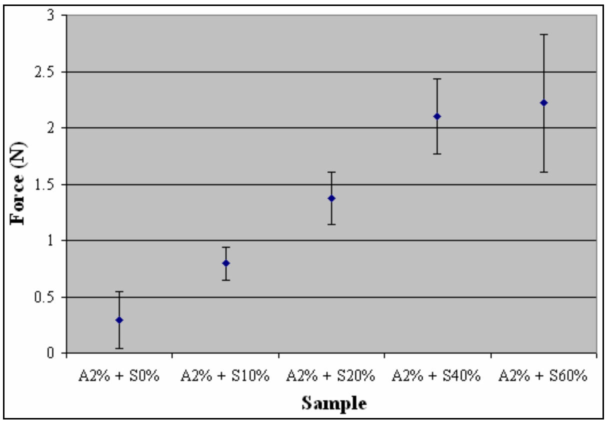

Figure 4: Mechanical strength of alginate-starch beads at 10% deformation

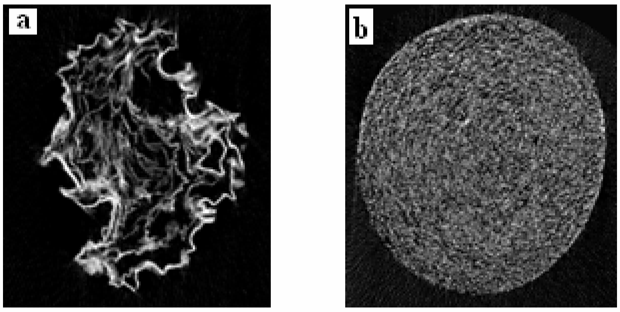

Figure 5: X-ray micro-computed tomography images
a) Alginate beads without starch granules b) Starch-packed alginate beads

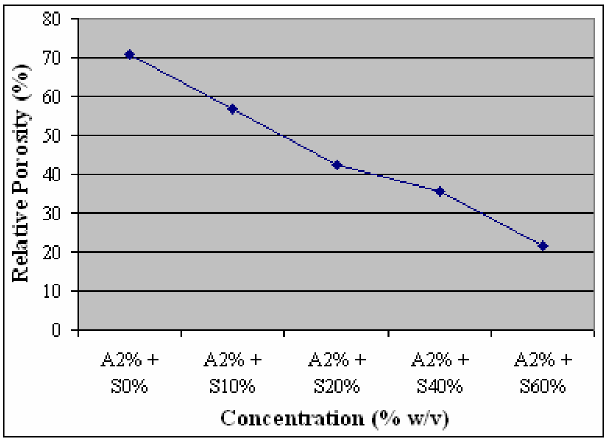

Figure 6: Relative porosity of beads produced via SPT at different concentrations

Conclusions

In conclusion, it was found that the hydrogel carrier quality could be improved by packing with high concentration of starch granules. The starch-packed beads were found to have better visual quality, good flowability, higher mechanical strength and lower porosity. These are the desirable particle qualities for encapsulation of bioactive compounds for preservation purpose.

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