Production of Alginate Microbeads through Air Atomisation Technique

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Introduction

Many technologies to produce alginate beads have been developed. Among them are the electrostatic generator, jet-cutter, nozzle resonance technique, air-atomisation and emulsion technique. The choice of the encapsulation technology is mainly depending on the desired mean size, size distribution, productivity, capital cost and operating cost. For industrial scale, criteria such as low capital cost, low operating cost and small bead size (<1000 µm) are often the determining factors in selecting the choice of technique. In general, microbeads size less than 1000 µm required the use of equipment of higher capital cost or operating cost. This might be not suitable for penny products in large-scale production. Air atomisation technique could be the answer to these challenges. Air atomisation is ubiquitous in today's high technology world; and is used in numerous industries, such as medicine, food, chemical processing, and agriculture for production of fine powders. It is a well-established technique in spraying system. For example, spray drying has been used in food industries for decades in encapsulation of flavours, vitamins and fine powder for dairy products. Furthermore, air atomisation conditions are mild, there is no problem of plugging, the process is continuous and power cost is generally low [1]. The purpose of this study is to characterize the alginate microbeads via the effects air liquid mass ratio (ALMR), feed pressure, liquid flow rate, sodium alginate concentration and spraying distance on the spray-droplets by using an air-atomisation method.

Materials and Methods

Materials: Chemicals obtained from commercial sources were of analytical grade and were used without further purification. Sodium alginate Manugel DMB (mannuronic acid 37%, guluronic acid 63%), was purchased from ISP Technologies Inc. (U.K). Calcium chloride was purchased from Merck, (Germany), and Tween 80 was purchased from Fluka BioChemica (Switzerland).

Microbeads preparation: 0.2% (w/v) of Tween 80 (nonionic surfactant) was added to sodium alginate solution. The mixture was sprayed into a vessel containing 1000ml of 1.5% (w/v) calcium chloride, which had been mixed with Tween 80, 0.1% (w/v). The device used to form the droplets was Lab Plant SD-O5 spray drier with two-fluid nozzle. The size of the inner nozzle is 0.5mm. The calcium chloride solution was constantly stirred with a magnetic stirrer at the bottom of the vessel to keep the droplets from sticking together. Sodium alginate was gelled to form microgel droplets when in contact with divalent calcium ions. The microgel droplets were cured for 15 min and filtered with filter paper in a funnel. Then, the filtered alginate microbeads were washed with distilled water to remove excess calcium chloride and they were stored in distilled water until further measurements were carried out. All experiments were carried out at room temperature of 28°C.

Characterization of Alginate Microbeads: The microbeads were coloured with crystal violet solution for 30 minutes prior to microscopic views under 100X magnification with microscope. The size and image analysis of the microbeads was performed by using an image analyzer software, Able Image Analyzer version 3.1b and Sigma Scan Pro5. Sauter mean diameter (d_{32}) was determined. A total of 200 diameters of microbeads were measured for each sample.

Results and Discussion

Effect of Feed Pressure: The microbeads size (d_{32}) as a function of feed pressure is presented in Figure 1. The Sauter mean diameters of the microbeads show an inverse relationship with feed pressure where higher pressure has resulted in smaller bead size. This behaviour was consistent with previous study [2, 3, 11], in which alginate poly-l-lysine microparticles size decreased with an increase in feed pressure [3, 11].

Effect of Sodium Alginate Flow Rate: The effect of sodium alginate flow rate on the Sauter mean diameter (d_{32}) of the microbeads is shown in Figure 2, where an increase in sodium alginate flowrate had resulted in a smaller microbeads size. Flow rate has a direct relationship effect on drop size, whereby an increase in flow rate will increase the drop size and vice versa [2]. This result was in good agreement with the theoretical explanation where the nozzle's hydraulic energy can atomise more liquid when the liquid flow rate is increased [4]. Similar results were obtained in the previous studies where alginate poly-l-lysine and barium alginate microparticles were produced with an air atomisation technique [3,5]. However, no significant statistical difference on the Sauter mean diameter was observed when the sodium alginate flow rate was increased.

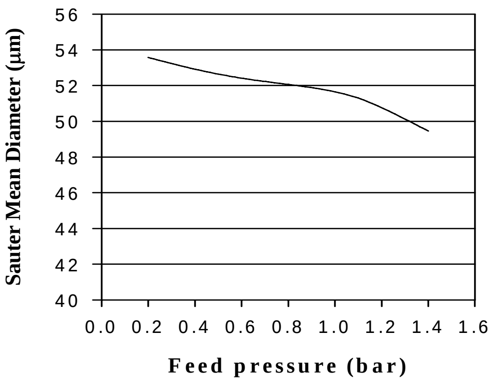


Figure 1: Effect of feed pressure on Sauter mean diameter of microbeads. Experimental condition: Sodium alginate concentration 2wt%, $CaCl_2$ concentration 1.5wt%, Sodium alginate flow rate 8.36 ml/min, and spraying distance 14cm.

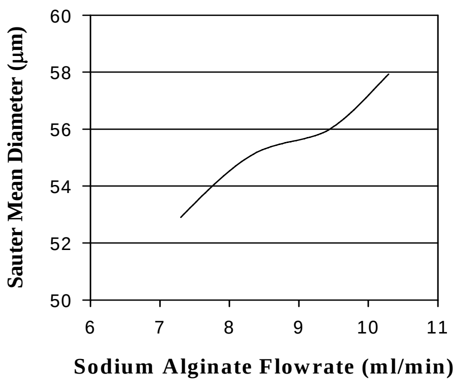


Figure 2: Effect of sodium alginate flow rate on Sauter mean diameter of microbeads. Experimental condition: Sodium alginate concentration 2wt%, $CaCl_2$ concentration 1.5wt%, Feed pressure 1.7bar, and spraying distance 14cm.

Effect of Spraying Distance: The effect of spraying distance on Sauter mean diameter of microbeads is shown in Figure 3. The Sauter mean diameter of the microbeads increased significantly when the spraying distance increased in the range of approximately 8cm to 30cm. This phenomenon could be resulted from the aggregation of the sprayed-droplets before hardened in the $CaCl_2$ bath. However, when the spraying distance was further increased to above 30cm, the Sauter mean diameter of the microbeads started to show reduction in size. In addition, deformation of alginate microbeads was observed when the collecting distance was increased, expressed in shape factor as shown in Figure 4. The deformed microbeads formed 'tails' as shown in Figure 5. The shape factors of the alginate mircobeads were in the range of 0.75 to 0.90 as the spraying distance was increased from 8cm to 52cm. These deformed microbeads with 'tails' had reduced their diameter size as the microbeads were not spherical in shape.

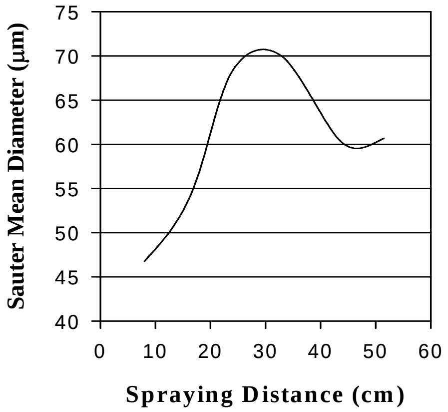


Figure 3: Effect of spraying distance on Sauter mean diameter of microbeads. Experimental condition: Sodium alginate concentration 2wt%, $CaCl_2$ concentration 1.5wt%, Feed pressure 1.7bar, Sodium alginate flow rate 8.36 ml/min.

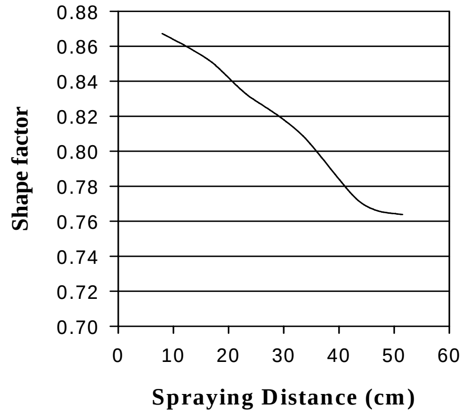
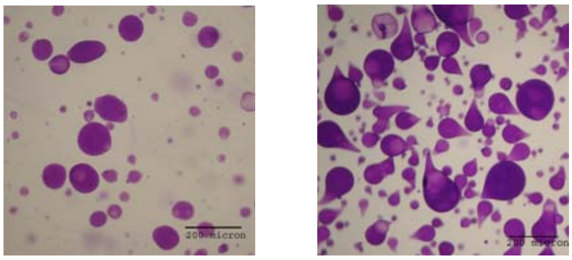


Figure 4: Effect of spraying distance on the shape factor of microbeads. Experimental condition: Sodium alginate concentration 2wt%, $CaCl_2$ concentration 1.5wt%, Feed pressure 1.7bar, Sodium alginate flow rate 8.36 ml/min.



(a) **(b)**

Figure 5: Microscopic view (100X magnification) of microbeads at spraying distance (a) 8.0cm (b) 51.5cm

Effect of Air Liquid Mass Ratio (ALMR): An increase in the ALMR had significantly decreased the mean size diameter as shown in Figure 6. The ALMR is one of the most important variables to affect mean droplet size [6,7]. In air atomization system, low-speed liquid jets are accelerated by the surrounding of high-speed gas flow, usually in the spray flow direction whereby the liquid is subjected to both tensile and shearing stresses and subsequently promotes disintegration of liquid [5]. An increase in air flow rate increases the tensile and shearing stresses of the sodium alginate and this has caused the formation of smaller microbeads with high disintegration rate. These results were in accordance with the theoretical explanation by Rizt *et al.*[8] who studied the mechanism of sheet disruption and drop formation. They proved that an increase in air velocity, had expedited the liquid sheet disintegration to form droplets.

Effect of Sodium Alginate Concentration: The Sauter mean size diameters of the microbeads decreased with an increase in the alginate concentrations, which is shown in Figure 7. When the concentrations of the sodium alginate increased, the number of binding sites for Ca^{2+} also increased. As a result, a more densely cross-linked gel structure formed [9] and this might had reduced the size of microbeads in the tight chains. These results are in very good agreement with the experimental results of Cui *et. al.* [3]. However, an inverse correlation between alginate concentration and size of microbeads was observed as described in Herrero *et al.*[5], Ge Jiang *et al.*[10] and Kwok *et al.*[11].

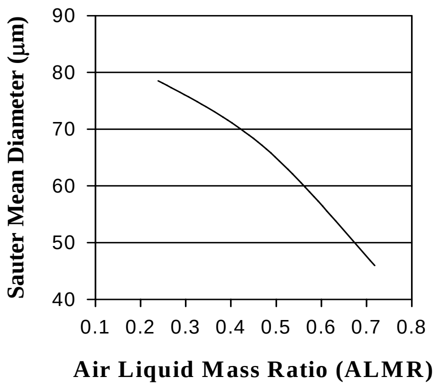


Figure 6: Effect of ALMR on mean size diameter of microbeads. Experimental condition: Sodium alginate concentration 2wt%, $CaCl_2$ concentration 1.5wt%, Feed pressure 1.7bar, Sodium alginate flow rate 8.36 ml/min, and spraying distance 14cm.

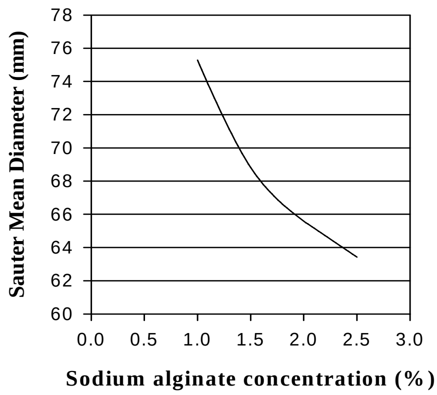


Figure 7: Effect of sodium alginate concentration on Sauter mean diameter of microbeads. Experimental condition: $CaCl_2$ concentration 1.5wt%, Feed pressure 1.7bar, Sodium alginate flow rate 8.36 ml/min and spraying distance 14cm.

Conclusion

The results presented in this work cover parameters to take into account when employing air atomisation method in producing calcium alginate microbeads. The air atomisation method reported has produced small-sized calcium alginate microbeads in the size range of Sauter mean diameter of 30-80 μm. Generally, the Saunter mean diameter decreased as the feed pressure and sodium alginate concentration decreased, but increased spraying distance. The results also indicated that the microbeads size of the spray was influenced by ALMR, whereby the microbeads size was easily adjusted by choice of ALMR with a higher ALMR resulted in a smaller microbeads size. This microencapsulation process via air atomisation method has provided an alternative to encapsulate biomaterials, which are in micron size and produced in mild conditions.

References

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