Production of Binary Phase Particles By Using Extrusion Technique

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Introduction

Encapsulation is a process in which sensitive ingredients or the core materials are entrapped in encapsulating agents or wall materials to protect them against oxygen, moisture, light, heat as well as to allow a controlled release of the entrapped ingredients. The wall materials can protect the core materials from environmental conditions and deteriorative reactions to enhance their stability. Sodium alginate is a natural polymer that has been extensively used as encapsulating agent due to its biocompatibility and simple gelation with divalent cations (e.g. calcium ions) [1]. Alginate is a polysaccharide, commonly extracted from brown seaweed *Laminaria hyperborean* [2] has a linear co-polymer composed of two monomeric units; D-mannuronic acid and L-guluronic acid.

Many of the sensitive ingredients such as anti-oxidants, nutraceutics or flavours are lipid-based compounds, which exist in liquid form at room temperature. The encapsulation of these lipophilic substances involves the emulsification or dispersion of the components in an aqueous solution. This can be performed by external gelation of the aqueous emulsion, where the hardened wall material that is contained in the aqueous emulsion entraps the dispersed oil droplets.

The objective of this study was to investigate the feasibility of encapsulating a model oil within calcium-alginate beads via extrusion method. Palm oil was used as the model oil. The first part of this study was to determine the effect of mixing on emulsion drop size and emulsion stability whereas in the second part, the effect of oil loading on encapsulation efficiency was determined.

Materials and Methods

Materials: Chemicals obtained from commercial sources were of analytical grade and were used without further purification. Sodium alginate Manugel GHB (mannuronic acid 40%, guluronic acid 60%), was supplied by ISP Technologies Inc. (U.S.A). Calcium chloride was purchased from Merck, (Germany), and the refined palm oil was purchased from Lam Soon Edible Oils Co. Ltd. (Malaysia).

Emulsion drop size: Emulsion was prepared by mixing sodium alginate (2.5% w/v) with oil loading of 30% wt, stirred with an impeller at 300 rpm and at 27°C. The emulsion drop size was assessed by taking samples every 5 minutes from the mixture and analysed under microscopic view with a microscope, which was equipped with a CCD camera. The sizes of the oil droplets were determined by using an image analyser software, Sigma Scan Pro5. A total of 200 oil droplets were measured for each sample to determine the mean diameter.

Emulsion phase stability: Emulsion samples (2 ml) were taken every 10 minutes and left in a glass vessel to study emulsion stability. Emulsion stability was assessed by measuring the height of the initial emulsion and the height of the remaining emulsion after phase separation. The emulsion stability (ES) was calculated as a percentage [3]:

Emulsion stability (ES) = (remaining emulsion height/Initial emulsion height) X 100

Oil loading: The effects of oil loading on the encapsulation efficiency of oil within calcium alginate beads were calculated based on the differences between the initial volume of oil in the oil-alginate mixture and the volume of oil remained non-encapsulated in the calcium chloride solution. The encapsulation process was carried out by using extrusion method with syringe. The mixing time for each emulsion at different oil-sodium alginate ratio was 30 minutes (i.e. selected based from the emulsion drop size and emulsion stability tests). 100 ml of the emulsions were dropped into a calcium chloride bath (1.5% w/v). Calcium-alginate beads encapsulating oil was formed and the beads were cured for 30 minutes and filtered with a filter paper in a funnel. The beads were washed with distilled water to remove excess calcium chloride and they were stored in distilled water. Finally, the volume of oil left on the surface of calcium chloride solution was measured. Encapsulation efficiency was calculated as follows:

Encapsulation efficiency = [(Total oil – non-encapsulated oil)/Total oil] X 100

Results and Discussion

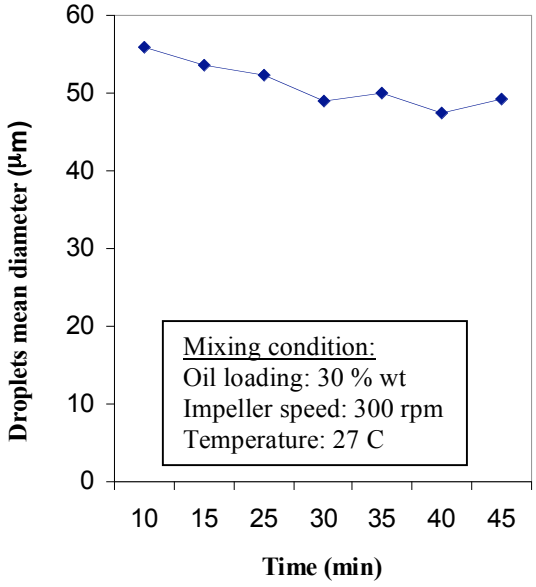

Figure 1: Mean diameter of oil-sodium alginate emulsion droplets over mixing time.

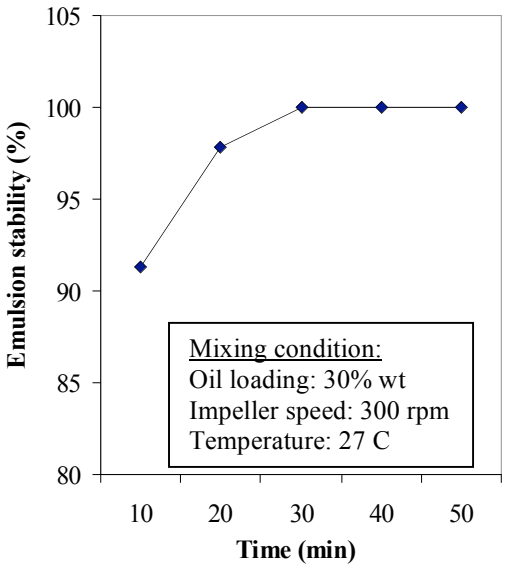

Figure 2: Emulsion stability over mixing time.

Emulsion stability: The effect of mixing time on emulsion droplet size is shown in Figure 1. The mean diameter of the emulsion droplets decreased gradually from 55.80 µm to 49.10 µm on increasing the mixing time from 10 to 30 minutes. However, above 30 minutes the droplets mean diameter only changed marginally. In the phase separation analysis, two separated phases were obtained, where the oil was at the top layer and the emulsion at the bottom layer. The stable state of an emulsion is in the form of its phases layers separated by interfaces [4]. The emulsion stability was plotted over mixing time as shown in Figure 2, where it can be seen that the emulsion stability significantly increased from 91.3% to 100% with increasing mixing time. A further increase in the mixing time from 30 to 50 minutes had no effect on the position of the emulsion interface; the emulsion stability reached constant at 100%, indicating that a mixing time of 30 minutes was sufficient to achieve stable emulsion.

Encapsulation efficiency: Figure 3 shows the effect of oil loading on encapsulation efficiency of oil within ca-alginate beads. It was found that the encapsulation efficiency was about 99% with oil loading of up to 30% wt. When the oil loading was higher than 30% wt, the encapsulation efficiency decreased significantly with increasing oil loading. The logical explanation for this observation is that at higher oil loading, there was less alginate, which was available at the surface of the extruded emulsion droplet. This inevitably reduced the site for cross-linking. Therefore, once the emulsion droplet dropped into the gelation bath, there was insufficient cross-linking at the surface to form a ca-alginate hydrogel barrier to contain the oil from leaking out. This observation is in good agreement with Adamiec et. al. [5], whose findings showed that the microencapsulation efficiency of peppermint oil in maltodextrin decreased with an increase of the initial oil content and Tan et. al. [6] whose findings also showed that an increase in the initial fish oil content encapsulated in modified food starch, resulted in lower microencapsulation efficiency.

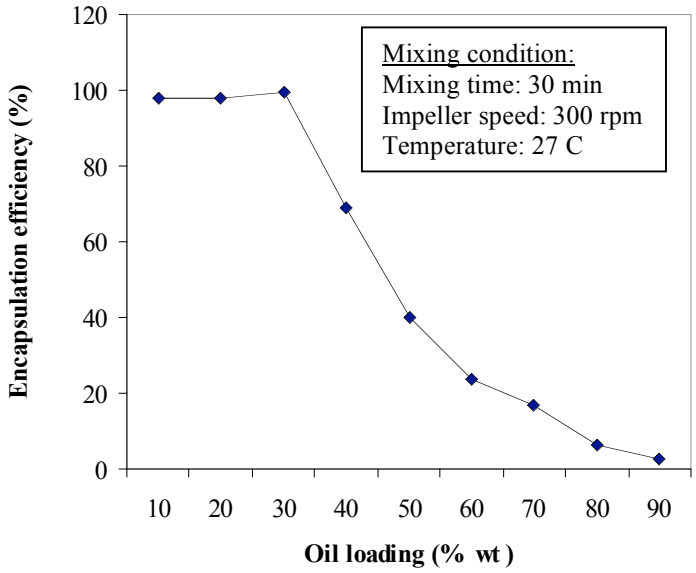

Figure 3: Effect of Oil loading on the encapsulation efficiency.

Conclusion:

In conclusion, this preliminary study shows that it is possible to encapsulate oil compounds within ca-alginate beads by using simple extrusion technique. However, future work has to concentrate on increasing the encapsulation efficiency in order to make this technique feasible for commercial application.

References:

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