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Physical stability of emulsion encapsulated in alginate microgel particles by the impinging aerosol technique

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ABSTRACT

Emulsion filled alginate microgel particles can be applied as carrier systems for lipophilic actives in pharmaceutical and food formulations. In this study, the effects of oil concentration, emulsifier type and oil droplet size on the physical stability of emulsions encapsulated in calcium alginate microgel particles (20–80 μm) produced by a continuous impinging aerosol technique were studied. Oil emulsions emulsified by using either sodium caseinate (SCN) or Tween 80 were encapsulated at different oil concentrations (32.55, 66.66 and 76.68% w/w of total solids content). The emulsions were analysed before and after encapsulation for changes in emulsion size distribution during storage, and compared to unencapsulated emulsions. The size distribution of encapsulated fine emulsion (mean size ~0.20 μm) shifted to a larger size distribution range during encapsulation possibly due to the contraction effect of the microgel particles. Coarse emulsion droplets (mean size ~18 μm) underwent a size reduction during encapsulation due to the shearing effect of the atomizing nozzle. However, no further size changes in the encapsulated emulsion were detected over four weeks. The type of emulsifier used and emulsion concentration did not significantly affect the emulsion stability. The results suggest that the rigid gel matrix is an effective method for stabilising lipid emulsions and can be used as a carrier for functional ingredients.

1. Introduction

Lipid carrier systems have been extensively studied for potential applications in the pharmaceutical and food industries. These carrier systems are highly valued for their control release properties of functional lipophilic components such as drugs. These systems include liposomes, solid-lipid particles, self-emulsifying drug delivery systems (SEDDS) and filled polymeric particles (McClements, 2010; Nanjwade, Patel, Udhani, & Manvi, 2011). Among these, emulsions represent the most conventional lipid delivery system.

Conventional emulsions are prone to destabilisation by environmental factors such as ionic strength, pH, and temperature. The complex chemical environment present in different foods limits the use of emulsions as a bioactive carrier system. In order to reach the small intestine where nutrients are absorbed, bioactive solubilised in emulsion needs to pass through the low pH condition of the stomach. Emulsions made with commonly used food grade emulsifier such as di- and triglycerides as well as surfactants like caprylocaproyl polyoxylglycerides (Labrasol), polyethylene glycol (25)-cetostearyl ether (Cremophor) and polyoxyethylene (20) sorbitan monooleate (Tween 80) are susceptible to enzymatic degradation in the gastrointestinal tract thus resulting in an unstable emulsion and lower bioactive bioavailability (Fatouros, Karpf, Nielsen, & Mullertz, 2007; Singh, Ye, & Horne, 2009).

The stability of an emulsion used as a carrier system is especially crucial in determining bioactive bioavailability. A number of studies have shown that lipid digestion and bioavailability of emulsions and their bioactive content are affected by factors such as droplet size, emulsion interfacial composition, lipid molecular structure and lipid physical state (Golding & Wooster, 2010; McClements, Decker, & Park, 2009). Emulsion size is a critical factor in determining the bioavailability of lipids, drugs or bioactives delivered as an emulsion (Hauss et al., 1998; Nielsen, Petersen, & Mullertz, 2008). Fine emulsion droplets provide a larger surface area for pancreatic lipases to attack resulting in increased rate of lipolysis compared to coarse emulsion droplets. Raatz, Redmon, Wimmergren, Donadio, and Bibus (2009) showed that the bioavailability of emulsified fish oil is significantly higher compared to unemulsified oil. Similarly, Haug et al. (2011) showed that fish oil delivered by a gelatin based emulsion microgel was readily absorbed by the human body resulting in a 105.6% increase in blood plasma docosahexaenoic acid (DHA) and eicosapentaenoic acid (EPA) levels when compared to fish oil delivered in soft gel capsules (unemulsified).

The encapsulation of emulsion in a polysaccharide gel matrix (emulsion filled gel) may potentially help stabilise the emulsion droplets. Polysaccharide gels, for example ionic alginate gel, are able to protect the entrapped bioactives against digestive enzymes and low pH conditions in the gastric juice by the acid-induced formation of a compact and highly viscous alginic acid layer, which is resistant to

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Table 1
Sample formulation of alginate-emulsion mixture prior to gelation. The sample names are given based on the concentration of oil per g dry solid mass.

Sample name	Oil conc. in dry solid basis[a] (% w/w)	Sodium alginate in alginate-emulsion mixture solution (% w/w)	Oil in alginate-emulsion mixture solution (% w/w)
32_{Oil}	32.25	2	1
67_{Oil}	66.66	2	5
77_{Oil}	76.68	2	10

[a] Determined from the amount of oil emulsion added to the alginate-emulsion mixture.

erosion and contraction (Klinkesorn & Julian McClements, 2010; Krasaekoopt, Bhandari, & Deeth, 2004; You et al., 2001). Alginate, a widely used polysaccharide, is made up of β-D-mannuronate and α-L-guluronate monomers. Gelation of alginate occurs by the ionic interaction between multivalent cations such as Ca^{2+} and carboxyl groups on the alginate monomers to form a gel network.

In this study, we attempt to study the effects of oil concentration, emulsifier type and emulsion size on the physical stability of emulsion encapsulated in alginate microgel particles. Past studies have investigated emulsion filled alginate gel particles containing lipids such as canola oil (Sun-Waterhouse, Wang, & Waterhouse, 2014), peppermint oil (Koo, Cha, Song, Chung, & Pan, 2014), durum oil (Durante, Lenucci, Laddomada, Mita, & Caretto, 2012), essential oils (Chan, Lim, & Heng, 2000; Soliman, El-Moghazy, Mohy El-Din, & Massoud, 2013), and palm oil (Chan, 2011). These studies have mainly focused on the methodology and chemical stability but not on the physical stability of the emulsion. These groups produced mm-sized alginate particles with the lab scale syringe method. In this research we utilised the newly developed impinging aerosol technique, which is a continuous and scalable method, to produce the emulsion filled microgel particles (Bhandari, 2009). Results from this experiment will provide a better understanding of the emulsion filled alginate microgel system in practical applications and give an early insight into understanding how encapsulated emulsions behave upon release in the gastric tract of the human body.

2. Materials and methods

Calcium alginate microgel particles were produced with sodium alginate (GRINDSTED® Alginate FD 155, Danisco, Australia) and calcium chloride. Sodium caseinate (SCN) (NatraPro, MG Nutritionals, Australia) or Tween 80 (Chem-Supply, Australia) was used as emulsifier. Canola oil was purchased from local supermarkets. Nile red (Sigma Aldrich, Australia) (0.1% v/w in acetone), a lipid soluble fluorescence stain, was used to stain the emulsion droplets. Deionised water was used as sample diluent throughout the experiment. Sodium azide (Sigma Aldrich, Australia) (0.001% w/w) was used as a preservative.

2.1. Preparation of emulsion

To prepare the emulsion filled alginate microgel particles, an oil emulsion was firstly made up followed by encapsulation in the alginate microgel particles. To create coarse emulsion, 10% (w/w) canola oil was emulsified in a 1% (w/w) emulsifier solution with the Silverson (UK) mixer at medium speed for 5 min. To create fine emulsion (mean size 0.22 μm), the coarse emulsion was passed through a two-stage homogeniser (Twin Panda, GEA, Australia) (1st stage — 25 MPa, 2nd stage — 0.5 MPa) twice. After emulsification, different proportions of emulsion, water and sodium alginate were mixed with a lab mixer (T25 IKA Labortechnik, Malaysia) to make up solutions with different oil contents (Table 1).

2.2. Preparation of emulsion-filled alginate microgel and macrogel particles

The oil emulsion was encapsulated in calcium alginate microgel particles based on the spray aerosol method described in the International Provisional Patent No. 062254, 2009 (Fig. 1a) (Bhandari, 2009; Ching, Bhandari, Webb, & Bansal, 2015). A fine aerosol mist of 0.1 M calcium chloride solution was created in the cylindrical encapsulation chamber using an air atomizing nozzle operated at liquid and air pressure of 0.15 and 0.2 MPa. Pressurized (0.5 MPa) mixture of sodium alginate and emulsion was counter currently atomized (25 °C) in the chamber using compressed air at 0.5 MPa.

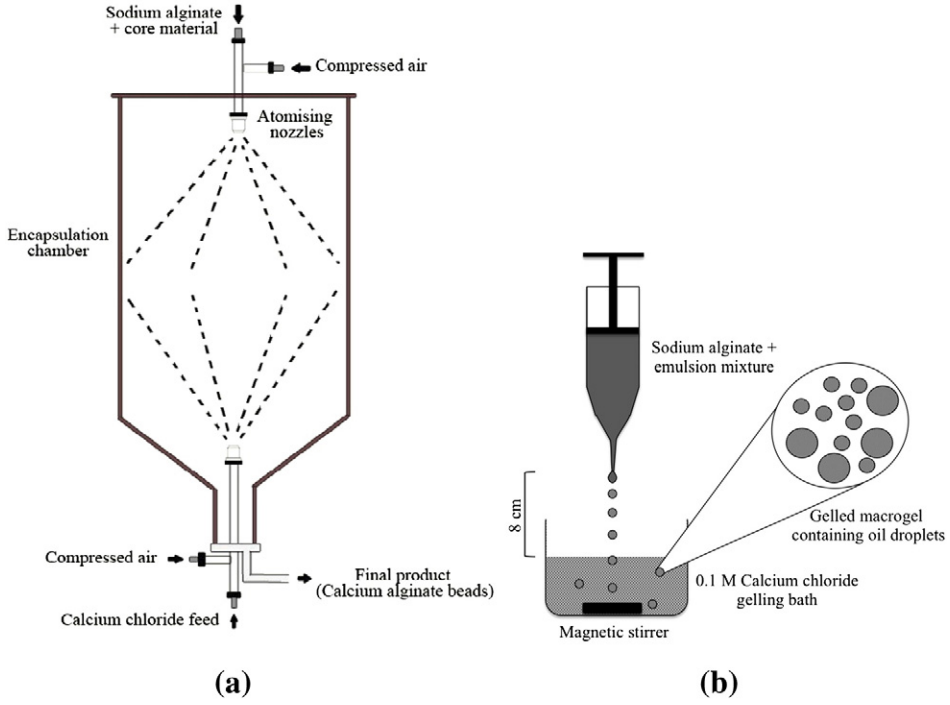

Fig. 1. Techniques used in producing (a) micron-sized alginate microgel particles (spray aerosol method) (modified from Bhandari (2009)), and (b) mm-sized macrogel particles (extrusion method) (modified from Ching, Bansal, and Bhandari (2015)).