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Microencapsulation of kenaf seed oil by co-extrusion technology

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ABSTRACT

The aim of this study was to evaluate the effect of different processing parameters such as different flow rates for shell and core feed, vibrational frequencies, drying methods and shell formulations on the characteristics of microencapsulated kenaf seed oil (MKSO) by using co-extrusion technology. Optical microscopy revealed that the MKSO produced at vibrational frequency of 500 Hz and core–shell flow rate of 0.2–7.2 mL/min (for 1.5% alginate) and 0.2–7.0 mL/min (for high methoxyl pectin (HMP)-enhanced alginate) appeared to be the best processing conditions and the MKSO exhibited the highest microencapsulation efficiency at these conditions. Besides that, freeze drying presented as a better drying method to dry the MKSO than air drying. HMP-enhanced alginate seems as a better shell formulation to produce MKSO with a smaller size (450–575 μm), having a stable water activity (0.280) against oxidative deterioration, and higher microencapsulation efficiency (76.62%) but less spherical in the shape of MKSO compared to 1.5% alginate. It was demonstrated that production of MKSO by co-extrusion is possible and that the process is stable and reproducible.

1. Introduction

Kenaf (*Hibiscus cannabinus* L.), which is from the Malvaceae family is a useful fibre plant native to India and Africa (Mohamed et al., 1995). In general practice, kenaf seeds are usually discarded as industrial waste during the harvesting or processing of kenaf. More attention has been focused recently on the application of food processing by-products and wastes, in order to reduce waste disposal problems and maximize the uses of available sources. Kenaf seed oil contains a high amount of monounsaturated and polyunsaturated fatty acids, which offers a good source of essential fatty acids that will give health benefits to human (Nyam et al., 2009; Ng et al., 2013). Linoleic acid ($C_{18:2}$) was the predominant unsaturated fatty acids, which contributed to 33.7% in the fatty acid composition of kenaf seed oil, followed by oleic acid ($C_{18:1}$) that was 31.8% (Ng et al., 2013). Apart from that, kenaf seed oil seems to be a good source of lipid-soluble bioactive as kenaf seed oil possesses considerable amounts of tocopherols, polyphenols and phytosterols, which exhibited significant antioxidant activities (Nyam et al., 2009; Ng et al., 2013; Razmkhah et al., 2013).

However, kenaf seed oil is chemically unstable and easily undergoes oxidation when exposed to the condition of oxygen, light, moisture and high temperature, due to its high amount of unsaturated fatty acids. The oxidative deterioration would affect the nutritional value, shelf stability and sensory properties of the kenaf seed oil (Velasco et al., 2003). Microencapsulation is an effective technology to protect the oil in suppressing or retarding the oxidation of unsaturated fatty acids and increase the field of applications of the microencapsulated products (Calvo et al., 2010; Rubilar et al., 2012). Microencapsulation has been widely used in the food industry for a number of reasons such as stabilization of the bioactive compounds, controlled release of the bioactive compounds, masking its unpleasant taste and smell (Đorđević et al., 2015).

At present, spray drying and co-extrusion are the most commonly used methods to encapsulate oil (Shi et al., 2013). However, exposure of microencapsulated kenaf seed oil (MKSO) to

Abbreviations: HMP, high methoxyl pectin; MKSO, microencapsulated kenaf seed oil; SF, sphericity factor; MEE, microencapsulation efficiency.

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high temperature during the process of spray drying causes severe lipid oxidation on the surface of the microcapsules and loss of bioactive compounds (Ng et al., 2013). Thus, extrusion method is the mildest one among all technologies for microencapsulation (Shi et al., 2013). Co-extrusion normally uses an encapsulator equipped with a concentric nozzle which utilizes vibrating nozzle technology. Vibrating nozzle technology is based on the principle by a laminar liquid jet break-up into droplets by a superimposed vibration on the nozzle (Whelehan, 2010). However, there has been a lack of study that was conducted on the parameters to produce MKSO by co-extrusion. Alginate is the most commonly shell wall material used because of their ability to form a stronger gel in the presence of Ca^{2+}, chemical stability, low toxicity and low immunogenicity (Liu et al., 2002). Alginate is the name given to a family of linear polysaccharides found in brown algae composed by α-L-guluronic acid and β-D-mannuronic acid. However, alginate may offer limited protection to core substances as the porous structure of alginate microspheres allows diffusion of an acid in and out of microspheres easily. Thus, this disadvantage can be effectively overcome by blending alginate with other polymers (Shi et al., 2013). Pectins that are industrially extracted from apple pomace and citrus peels are mostly in the form of high methoxyl pectin (HMP) (>50% degree of esterification, DE), which often used to increase the gel strength of food products (Thakur et al., 1997). It is of interest to investigate the effect of blending of alginate with HMP on the characteristics of MKSO.

In this study, the effects of different flow rates for shell and core feed, vibrational frequencies, drying methods and shell formulations were evaluated on the characteristics of MKSO. Optimal flow rates and vibrational frequency were first selected based on the size and microencapsulation efficiency of MKSO. After that, two drying methods which were air drying and freeze drying, as well as two shell formulations which were 1.5% sodium alginate and HMP–alginate solution were compared based on the size, sphericity factor, water activity and microencapsulation efficiency of MKSO.

2. Materials and methods

2.1. Materials and chemicals

Kenaf (*H. cannabinus* L.) seeds were obtained from Malaysia Agricultural Research and Development Institute (MARDI), Selangor, Malaysia. Sodium alginate was purchased from Friendemann Schmidt, Australia. HMP was purchased from a local food ingredient supplier (VIS Food Tech Ingredient Supplies, Malaysia). All chemicals used were analytical grade (Merck, Darmstadt, Germany).

2.2. Solvent extraction of kenaf seed oil

The kenaf seeds were ground into fine powder using a food grinder (Sharp, USA). The oils were extracted from the seeds with a Soxhlet extractor using hexane at 60 °C for 3 h [sample-to-solvent ratio 1:5, w/v] (Firestone, 1993). The oil was then recovered by evaporating off the solvent using rotary evaporator Model R-205 (BÜCHI Labortechnik AG, Switzerland) at 40 °C and residual solvent was removed by flushing with 99.9% nitrogen.

2.3. Preparation of shell formulations

A sodium alginate solution (1.5% w/w) was prepared by adding the required amount of sodium alginate to distilled water at 20 °C, homogenised using a T25 digital Ultra-Turrax homogenizer (12,000 rpm for 2 min, IKA, Germany). HMP solution (1.5% w/w) was prepared by adding the required amount of HMP powder to distilled water at 20 °C, with homogenization at 7000 rpm for 1 min using the T25 digital Ultra-Turrax homogenizer. This study was investigated adding of HMP into alginate is it have a better effect, therefore one formulation with adding HMP first, and compare with the formulation with alginate alone. The HMP–alginate solution was prepared by mixing carefully the 1.5% w/w alginate solution with the 1.5% w/w HMP solution at a volume ratio of 2:1, followed by gentle stirring with a magnetic stirrer (speed at 3 for 2 min) to obtain a homogenous shell solution, and stored overnight at 4 °C. Alginate should be mixed at a higher proportion as the calcium binding mechanism formed between Ca^{2+} and guluronate and galacturonate blocks in alginate and pectin, respectively. HMP consists less galacturonate blocks.

2.4. Microencapsulation of kenaf seed oil using co-extrusion technology

The microencapsulation of kenaf seed oil using co-extrusion technology was carried out according to the previously described method with some modifications (Homar et al., 2007; Wang et al., 2013) by a Buchi Encapsulator B-390 (BÜCHI Labortechnik AG, Switzerland). Kenaf seed oil and shell formulations were taken out from the freezer and chiller respectively and return back to the room temperature. During microencapsulation, the core fluid (kenaf seed oil) and the shell fluid (1.5% alginate or HMP–alginate solution) were simultaneously pumped into the concentric nozzle (150 μm/300 μm) by the air pressure (600 mbar) to give a core–-shell fluid stream which was sprayed out through the nozzle. An additional electrostatic field of 1.5 kV was applied between the nozzle and the hardening solution and the amplitude was set at 3.

The microcapsules were incubated in 3% w/w $CaCl_2$ solution and stirred gently for 10 min for complete gel hardening. After that, the microcapsules were collected with a 100 μm nylon sieve and rinsed twice with distilled water. Then, the microcapsules were backed by tissue papers until no further moisture was found on the tissue papers. The size, shape and surface morphology of freshly prepared microcapsules were evaluated via observation using a Nikon YS100 optical microscope (Nikon Corporation, Japan). Afterward, microcapsules were dried according to different procedures which were air drying at room temperature or freeze drying. The dried microcapsules were evaluated again via optical analysis.

2.4.1. Effects of flow rates for shell and core feed

A shell feed flow rate of 7.2 and 9.2 mL/min for 1.5% alginate, and 7.0 and 10.0 mL/min for HMP–alginate and a core feed flow rate of 0.2 and 0.4 mL/min were selected in this study. The flow rate was adjusted using liquid flow regulating valve on the encapsulator. Other process variables (vibrational frequency of 1500 Hz and the produced MKSO was air dried) remained constant.

2.4.2. Effects of vibrational frequency

After the flow rates were determined, the MKSO was produced at different frequencies which were 500, 1000, 1500 and 2000 Hz for the shell formulation of 1.5% alginate and HMP–alginate and then air dried to study the effects of different vibrational frequencies.

2.4.3. Effects of drying methods

The MKSO was produced with the flow rate of the two feeds and vibrational frequency that have been determined previously and then dried using two drying methods which were air drying and freeze drying. For air drying, the MKSO obtained was washed with 100 mL of absolute ethanol to facilitate the air drying process and dried at room temperature, 25 °C for 14–18 h until constant weight