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Effect of total solids content in feed emulsion on the physical properties and oxidative stability of microencapsulated kenaf seed oil

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ABSTRACT

Kenaf (*Hibiscus cannabinus* L.) seed oil has high potential to be used as edible oil. Despite the high content of polyunsaturated fatty acids, one of the major complications in commercialisation kenaf seed oil is its rapid oxidation, which leads to the production of undesirable toxic substances such as peroxides. The objective of this study was to investigate the effect of total solids content (TSC) on the oxidative stability of microencapsulated kenaf seed oil (MKSO) and to compare its oxidative stability with bulk kenaf seed oil upon accelerated storage. Microcapsules with 20%, 30% and 40% total solids content were prepared. Physical properties, such as the emulsion characteristics and microcapsules characteristics were also studied. Results showed that bulk kenaf seed oil was oxidised to a greater extent compared to the microencapsulated samples. The results showed that 40% total solids content microcapsules had the lowest peroxide value (PV), p-anisidine value (AnV), total oxidation (TOTOX) value and free fatty acid (FFA) value, which were 3.70 ± 0.83 meq O_2/kg oil, 16.12 ± 0.19, 23.52 ± 1.67 and 2.54 ± 0.06%, respectively. The microencapsulation of kenaf seed oil showed protective effect against lipid oxidation.

1. Introduction

Kenaf (*Hibiscus cannabinus* L.) seed oil has a relatively high amount of monounsaturated and polyunsaturated fatty acids, which are nutritionally beneficial for human health (Coetzee, Labuschagne, & Hugo, 2008) and seems to be a good source of lipid-soluble bioactives (Nyam, Tan, Lai, Long, & Che Man, 2009). Despite the high content of polyunsaturated fatty acids, one of the major complications in commercialisation kenaf seed oil is its rapid oxidation, which leads to the production of undesirable toxic substances such as peroxides (O'Brien, 2009). For food industry, the oxidation level of lipid is a crucial quality criteria, as the oxidation products formed not only causes rancidity but also degrades the nutritional quality and safety of the lipid (Kanazawa, Sawa, Akaike, & Maeda, 2002). Microencapsulation is a process that makes it possible to transform the oil into a powder, where the small droplets of oil are surrounded by a shell coating of proteins and/or carbohydrate resulting in small dry granules that have powder like flow characteristics (Wan, Bechtel, & Sathivel, 2011). Microcapsules with low microencapsulation efficiency can lead to breakage of the capsules, dissolution and losses of the core material as well as lead to higher amount of surface oil. Based on the previous studies conducted, one of the major factors that influence encapsulation efficiency of microencapsulated oils is the properties of the in feed emulsion, such as total solids content, viscosity and droplets size of the emulsion (Jafari, Assadpoor, He, & Bhandari, 2008).

To the best of our knowledge, no information has been reported on the effects of total solids content in feed emulsion on the microencapsulated kenaf seed oil. Therefore, the main objectives of this study were to study the influence of total solids content (TSC) in feed emulsion on the physico-chemical properties as well as oxidative stability of microencapsulated kenaf seed oil (MKSO) upon accelerated storage. The oxidative stability of microencapsulated kenaf seed oil and bulk (uncapsulated) kenaf seed oil under accelerated storage were also compared. Peroxide value (PV), p-anisidine value (AnV), total oxidation value (TOTOX), and free fatty acid (FFA) value of the oil were determined to analyse the oxidative stability of the kenaf seed oil.

2. Materials and methods

2.1. Materials and chemicals

Kenaf (*H. cannabinus* L.) seed was obtained from Malaysian Agricultural Research and Development Institute (Selangor, Malaysia).

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Wall materials, such as sodium caseinate (China), maltodextrin DE10 and soy lecithin (used as an emulsifier) were purchased from a local food ingredient supplier (VIS Food Tech Ingredient Supplies, Selangor, Malaysia). All chemicals used were analytical grade (Merck, Darmstadt, Germany).

2.2. Methods

2.2.1. Oil extraction

The kenaf seeds were ground into fine powder using a coffee grinder (National, Osaka, Japan). The oils were extracted from the seeds with soxhlet extractor using hexane at 60 °C for 8 h (AOCS, 1997). The oil was then recovered by evaporating off the solvent using rotary evaporator Model N-1 (Eyela, Tokyo Rikakikal Co., Ltd., Japan) and residual solvent was removed by flushing with 99.9% nitrogen.

2.2.2. Preparation of emulsion

Kenaf seed oil was homogenised with wall material solutions (sodium caseinate, maltodextrin DE10) at a fixed core/wall ratio of 1:3 and a protein/carbohydrate ratio of 1:9, whereas soy lecithin was added as an emulsifier at the ratio of 0.1:1 (w/w) with respect to the protein (Lim, Tan, Bakar, & Ng, 2012). The wall material mixtures were dispersed in deionised water and kept overnight at 4 ± 2 °C for rehydration. Kenaf seed oil was added into the mixture and the pre-homogenised in a shear homogeniser (Silverson L4R, Buckinghamshire, UK) for 5 min at 3000 rpm until complete dispersion was observed. The resultant emulsions were further homogenised in a high-pressure homogeniser (APV, Crawley, UK) at pressure of 50 MPa for 4 cycles. In this study, emulsions with different total solids content (20%, 30%, 40% w/w) were used in the microencapsulation of kenaf seed oil. The experiment design was shown in Table 1.

2.2.3. Emulsion characterization

2.2.3.1. Emulsion droplet size. The determination of emulsion droplet size was carried out according to the method of James-Smith, Alford, and Shah (2007). Malvern Mastersizer X (Malvern Instruments, UK) was used. The refractive index of the dispersed phase is input into the software. The refractive index of kenaf seed oil was set at 1.465q. The emulsion sample was measured 5 min after high pressure homogenizing to cancel any creaming effect and it was diluted by 10% with water. The droplet diameter is determined by means of light scattering.

2.2.3.2. Emulsion viscosity. The apparent viscosities of the emulsions were measured immediately after the sample preparation at 25 °C using a rheometer equipped with concentric cylinder measuring system (Rheolab QC, Anton Paar, USA). The viscosity measurement of the rheometer appears in the unit of mPa s.

2.2.4. Spray drying of emulsion

The slurry was spray-dried in a Buchi B-290 model mini spray dryer (Buchi Labortechnik AG, Switzerland) equipped with a 0.7 mm standard diameter nozzle. Airflow through the chamber was set to the equipment maximum. The compressed air at a pressure of 600 kPa was used for atomisation and the atomiser pressure was 450 ± 10 kPa. Emulsion feed temperature was 25.0 ± 0.5 °C, and the feed rate was 1 L/h. Inlet temperature was set at 160 °C and the outlet temperature was 85 ± 2 °C. The dried microencapsulated kenaf seed oil (MKSO) was collected and sealed in high-density polyethylene (HDPE) plastic bags and stored at −20 °C in freezer for further analyses.

Table 1
Experiment design for the spray drying tests.

Total solids %	Sodium caseinate (g)	Maltodextrin (g)	Soy lecithin (g)	Deionised water (mL)	Kenaf seed oil (g)
20	10	90	1.0	Top up to 466.67	33.33
30	15	135	1.5	Top up to 450.00	50.00
40	20	180	2.0	Top up to 433.33	66.67

2.2.5. Physico-chemical properties of microencapsulated kenaf seed oil

2.2.5.1. Moisture content. The moisture contents of the microcapsules (4 g) were determined gravimetrically by oven drying at 103 °C to constant weight (Hogan, Mcnamee, O'Riordan, & O'Sullivan, 2001).

2.2.5.2. Water activity. The a_w of the microcapsules was determined by dew point method (Aqua-Lab Water Activity Meter, Series 3, Decagon Devices Inc., USA) at 25 °C.

2.2.5.3. Scanning electron microscopy. A scanning electron microscope was used to examine the morphology and surface appearance of the microcapsules. MKSO samples were mounted on specimen stubs with double sided adhesive carbon tapes and then gold-coated in a putter coater (BAL-TEC, SCD 005, Witten, Germany). The coated microcapsules were examined in a LEO 1455 VPSEM attached with EDX (FEI Co., Eindhoven, the Netherlands) at 20.0 kV.

2.2.5.4. Particle size analysis. The particle size of the MKSO was determined by laser light scattering (Mastersizer 2000G equipped with a Scirocco dry powder dispersion unit, Malvern Instruments, Worcestershire, UK) with measurement time 5 s and background time 10 s.

2.2.5.5. Microencapsulation efficiency (MEE) test. Microencapsulation efficiency (MEE) was calculated using Eq. (1) according to the previously described method (Pauletti & Amestoy, 1999).

$$\text{MEE} = [(\text{total oil} - \text{extractable oil}) \times 100]/\text{total oil} \quad (1)$$

Total oil content of MKSO was determined according to the previously described method (Lim et al., 2012). In the preparation of de-emulsifier, 10 g of sodium salicylate and 10 g of sodium citrate were dissolved separately in deionised water, followed by mixing these solutions together with 18 mL of *n*-butanol, and top up to 90 mL with deionised water. 10 g of MKSO was mixed with 20 mL water at 50 °C in an Erlenmeyer flask with a stopper. 15 mL of the pre-prepared de-emulsifier was added in, and the mixture was shaken vigorously and left to stand in a 70 °C water bath for 6 min. The resulting mixture was then centrifuged at 3000 × g for 10 min, and the total oil was collected.

The extractable oil was measured according to the method described by Velasco, Marmesat, Dobarganes, and Márquez-Ruiz (2006). 200 mL of light petroleum ether (60–80 °C) was added to 10 g of the MKSO in an Erlenmeyer flask with a stopper and stirred at 25 °C in the dark for 15 min. The resulting mixture was filtered by passing through a Buchner funnel with a Whatman No. 4 filter paper, collected in a round-bottom flask, and evaporated using a rotary evaporator in a water bath at less than 30 °C to minimize the influence of heating on lipid oxidation.

2.2.6. Oxidative stability under accelerated storage

The oxidative stability of MKSO was tested under accelerated storage conditions by the Schaal oven test (Wanasundara & Shahidi,