Journal of Food Engineering 225 (2018) 34–41

Contents lists available at ScienceDirect

Journal of Food Engineering

journal homepage: www.elsevier.com/locate/jfoodeng

In-vitro digestion of refined kenaf seed oil microencapsulated in β-cyclodextrin/gum arabic/sodium caseinate by spray drying

Sook Chin Chew [a], Chin Ping Tan [b], Kar Lin Nyam [a, *]

[a] Department of Food Science and Nutrition, Faculty of Applied Sciences, UCSI University, 56000 Kuala Lumpur, Malaysia
[b] Department of Food Technology, Faculty of Food Science and Technology, Universiti Putra Malaysia, 43400 Serdang, Selangor, Malaysia

ARTICLE INFO

Article history:
Available online 30 January 2018

Keywords:
Kenaf seed oil
Spray drying
Oil release
Antioxidant activity
Tocopherol
Phytosterol

ABSTRACT

Refined kenaf seed oil was microencapsulated by spray drying using the wall materials of β-cyclodextrin (β-CD), gum arabic (GA), and sodium caseinate (SC) to produce three different models (SC:β-CD, GA:β-CD, GA:SC:β-CD) of microencapsulated refined kenaf seed oil (MRKSO). An *in-vitro* digestion was used to simulate the human gastrointestinal digestion to examine the oil release behavior of MRKSO, changes in antioxidant activity and bioactive compounds of undigested oil, digested oil, and digested MRKSO samples. The results showed that three models of the MRKSO offered good protection by a lower percentage of oil released (1.43–6.44%) in the simulated gastric fluid and a high percentage of oil released (81.10–91.19%) after simulated gastric and intestinal phases digestion. The degree of lipolysis was in the order of SC:β-CD > GA:SC:β-CD > GA:β-CD > un-encapsulated oil. Among three models of MRKSO, GA:SC:β-CD offered better bioaccessibility by showing an increase in DPPH (20.0% increase) and ABTS (5.0% increase) values, phenolic content (130.4% increase), tocopherol and tocotrienol contents (147.7% increase), as well as slower degradation of phytosterol contents (59.4% decrease) after *in-vitro* digestion, compared to the undigested kenaf seed oil.

1. Introduction

Currently, the interest in functional and nutritious oil has increased due to its abundance of unsaturated fatty acids and bioactive compounds (Chew et al., 2015). Kenaf (*Hibiscus cannabinus* L.) is a valuable fiber plant native to India and Africa from the Malvaceae family. Kenaf seeds have been widely studied to yield kenaf seed oil that is high in monounsaturated and polyunsaturated fatty acids. Moreover, abundance of lipophilic bioactive compounds is presented in the kenaf seed oil, such as tocopherols and phytosterols, which can offer antioxidant activity (Chew et al., 2016). Thus, refined kenaf seed oil has been suggested to be used as edible and functional oil due to its unique composition (Chew et al., 2016, 2017a). However, the incorporation of these functional ingredients into foods, maintaining their stability and functionality to the gastrointestinal (GI) tract for absorption by the human body is a major challenge faced by the food industry. Presence of high amount of oxygen in the GI tract can favor the oxidation of unsaturated fatty acids (Chew et al., 2015). Microencapsulation has been focused recently to provide a physical barrier to protect the functional oils against degradation of unsaturated fatty acids and bioactive compounds (Chew and Nyam, 2016). Microencapsulation offers controlled release property by delivering the functional ingredients to the small intestine where they are absorbed into the bloodstream (Chew et al., 2015; Timilsena et al., 2017). Spray drying is the most common and economically feasible technology by using the hot gas stream to transform the fluid product into dry powder form to encapsulate functional ingredients in powder form for food applications (Daza et al., 2016; Edris et al., 2016; Goyal et al., 2015).

Spray drying has been used to encapsulate kenaf seed oil in the previous study by using maltodextrin, sodium caseinate, and soy lecithin as the wall materials (Ng et al., 2013). However, high glycemic index (>130) and potentially unhealthy effects of maltodextrin have discouraged its application as wall material (Sun-Waterhouse and Waterhouse, 2015). Dietary fiber has been encouraged to be used as wall material in the encapsulation process due to their health benefits and protective effect on the core substances (Chew et al., 2015). Cyclodextrins (CDs) are dietary fibers that have low glycemic index and can act as prebiotics in the GI tract. CDs are beneficial in controlling human body weight and

* Corresponding author.
E-mail address: nyamkl@ucsiuniversity.edu.my (K.L. Nyam).

https://doi.org/10.1016/j.jfoodeng.2018.01.018
0260-8774/

blood lipid profile (Fenyvesi et al., 2016). β-CD is the most commonly used among the CDs in the encapsulation process due to their ability to bind the hydrophobic oil in the interior cavity and the hydrophilic exterior surface can bind with water or hydrophilic head of other wall materials to form inclusion complex (Cheong and Nyam, 2016; Hundre et al., 2015). Cheong et al. (2016) reported that the kenaf seed oil encapsulated in nanoemulsion by β-CD, sodium caseinate and tween 20 offered a good lipid digestion and good bioaccessibility of antioxidants, as well as low degradation rate of phytosterols during the simulated digestion. It is of interest to study the β-CD to encapsulate kenaf seed oil in a powder form in order to develop the functional product.

In this context, β-CD will be blended with other wall materials, such as gum arabic and sodium caseinate to encapsulate refined kenaf seed oil. Gum arabic (GA) is a dietary fiber that can ferment in the colon to produce short-chain fatty acids that offer prebiotic effect and also possesses high solubility, low viscosity and good emulsifying properties (Babiker et al., 2012; Fernandes et al., 2013). Sodium caseinate (SC) is a water-soluble milk protein that could offer good emulsifying and encapsulation properties (Wang et al., 2016). To the best of our knowledge, no information has been reported on the microencapsulation by using the formulation of β-CD/GA/SC by spray drying. Hence, in order to ensure the delivery of the refined kenaf seed oil to the targeted sites of the human GI tract, it is important to test the stability and the release behavior of the oil during *in-vitro* digestion using simulated digestive fluids. Previous study reported the formation of a secondary emulsion by addition of β-CD to the primary emulsion had remarkably increased its stability by binding of the hydroxyl group of β-CD with the hydrophilic head group of other wall materials via hydrogen bonding in the emulsion (Cheong and Nyam, 2016). Thus, β-CD would add as a secondary layer into the primary emulsion which contained the required wall materials and oil as the secondary emulsion in this study.

In this study, microencapsulated refined kenaf seed oil (MRKSO) was subject to dissolution tests in gastric fluid and intestinal fluid to understand the release behavior of refined kenaf seed oil from MRKSO during their passage through the human GI digestion system. The bioaccessibility of the released refined kenaf seed oil from MRKSO was evaluated by compared its antioxidant activity and bioactive compounds after the simulated digestion with undigested and digested un-encapsulated kenaf seed oils.

2. Materials and methods

2.1. Materials

Kenaf seeds (variety: V36) were purchased from the National Kenaf and Tobacco Board (Kelantan, Malaysia). β-CD was purchased from Zibo Qianhui Fine Chemical Co., Ltd. (Shandong, China). Gum arabic and sodium caseinate were purchased from VIS Food Tech Ingredient Supplies (Kuala Lumpur, Malaysia). Bile salts, 2,2'-azino-bis(3-ethylbenzothia zoline-6-sulfonic acid) diammonium salt (ABTS), 2,2'-diphenyl-1-picrylhydrazyl radical (DPPH•, 95%), 5α-cholestane, 6-hydroxy-2,5,7,8-tetram-ethylchroman-2-carboxylic acid [Trolox (TE), 97%], N,O-bis (trimethylsilyl) trifluoroacetamide (BSTFA) with 1 mL/100 mL trimethylchlorosilane (TMCS), phytosterols standard (β-sitosterol, campesterol, and stigmasterol), pepsin from porcine gastric mucosa (P7125), and pancreatin from porcine pancreas (P3292) were purchased from Sigma-Aldrich (St. Louis, MO, USA). Tocopherols and tocotrienols standards (α-, β-, γ-, and δ-) were the products of ChromaDex, Inc. (California, USA). Tween 20 and other chemicals used were purchased from Merck Co., Ltd. (Darmstadt, Germany).

2.2. Refining of kenaf seed oil

Crude kenaf seed oil was extracted using Soxhlet extractor at 60 °C for 3 h from the kenaf seeds according to the previously reported method (Chew et al., 2017a). The remaining solvent was evaporated off using Buchi Multivapor P-6 (Büchi Labortechnik AG, Switzerland) to recover the crude kenaf seed oil. A chemical refining process, which includes degumming, neutralization, bleaching, and deodorization was carried out to produce refined kenaf seed oil (Chew et al., 2017a). The crude oil was pre-treated with the 0.09% of phosphoric acid (85% concentration) for 10 min, followed by treated with 22.4% of Milli-Q water for 30 min at 40 °C with 175 rpm. Then, the degummed oil was neutralized by adding 3.75% w/w of an excess of the stoichiometric ratio of NaOH solution (16 °Be) at 40 °C for 20 min. The neutralized oil was reacted with 1.5% w/w of acid-activated bleaching earth at 70 °C for 40 min. Finally, the bleached oil was deodorized with a lab-scale glass deodorizer at 220 °C with a reduced pressure of 9–12 mbar for 90 min.

2.3. Preparation of emulsions

Three models of MRKSO were employed in this study, which were SC:β-CD (ratio 2:1), GA:β-CD (ratio 2:1) and GA:SC:β-CD (ratio 4:1:1). The wall material concentration was 20% w/w for SC:β-CD model, and 30% w/w for GA:β-CD and GA:SC:β-CD models of MRKSO. Tween 20 (1% w/w) was added into each formulation. Required amounts of wall materials (gum Arabic, sodium caseinate and Tween 20) were dissolved in distilled water at 50 °C using T25 digital Ultra-Turrax homogenizer (IKA, Germany) and stored overnight at shaker (SK-300, Lab Companion, Korea) to ensure full hydration. After that, required amount of refined kenaf seed oil was added drop-wise into the solution under magnetic stirring at 1000 rpm and continued stirred for 10 min after all the oil was incorporated into the aqueous phase to form the primary emulsion. The oil-to-wall ratio was kept constant at 1:4 in each model in order to evaluate the effect of different wall constituents. Then, required amount of β-CD was dissolved into the primary emulsion using T25 digital Ultra-Turrax homogenizer (IKA, Germany) at 9000 rpm for 5 min to form a secondary emulsion. The emulsion was then homogenized using a Labsonic®P ultrasonic homogenizer (Sartorius AG, Germany) at the amplitude of 100% for 5 min to get the final emulsion. The selection of the wall materials and the ratio was based on the initial screening carried out in the laboratory (data not shown).

2.4. Microencapsulation by spray drying

The emulsion was spray dried using Mini Spray Dryer B-290 (Büchi Labortechnik AG, Switzerland) with 0.45 m in height and 0.14 m in diameter of the glass dryer chamber. The spray dryer consisted of a two-fluid nozzle composed of an internal tip with an opening of 0.7 mm in diameter and an external ring with an opening of 1.5 mm in diameter. The inlet temperature was 160 °C and the emulsion was fed into the main chamber through a peristaltic pump at a pump setting of 20% with a feed rate of 8 ± 2 g/min. The compressor air pressure was 600 kPa and the atomiser pressure was 450 ± 10 kPa. The resultant powder was collected from both the drying chamber wall and from the cyclone. The powder was stored in freezer at −20 °C for further analysis.

2.5. In-vitro release study

2.5.1. Preparation of simulated digestive fluids

Simulated gastric fluid (SGF) was prepared by following the