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Enhancement of storage stability of wheat germ oil by encapsulation

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ABSTRACT

Wheat germ oil which is a rich source of α-tocopherol is susceptible to oxidation. The main objective of this study was to encapsulate wheat germ oil to enhance its oxidation stability. It was also aimed to investigate the effects of different homogenization methods on physicochemical properties and storage stability of encapsulated wheat germ oil. As homogenization methods, silent crusher (SC), microfluidization (MF) and ultrasonication (US) were used. SC and MF techniques created more stable emulsions than US. The effects of maltodextrin (MD) in combination with sodium caseinate (NaCa), gum arabic (GA), chitosan (CS) or whey protein concentrate (WPC) and also WPC:CS combination at different ratios on encapsulation efficiency of capsules were studied. Sodium caseinate (NaCa) was found to be better coating material than chitosan (CS), whey protein concentrate (WPC) and gum arabic (GA) for the encapsulation of wheat germ oil in terms of encapsulation efficiency. The rate of increase in totox values of fresh oil was apparently higher than that of microcapsules. The loss of α-tocopherol in encapsulated oil was found to be lower than that in fresh oil during storage at both 15 °C and 45 °C for 24 days.

1. Introduction

Increasing concerns on human health have led to the development of functional foods. Wheat germ oil is one of the functional food ingredients since it is a rich source of α-tocopherol, phytosterols, polycosanols, thiamin, riboflavin, and niacin (Megahed, 2011; Sonntag, 1979). The high amount of tocopherols, linoleic acid, and polycosanol in wheat germ oil decrease plasma and liver cholesterol levels in animals, improve physical strength and retard aging (Wang and Johnson, 2001; Dunford, 2012). On the other hand, wheat germ oil is susceptible to oxidation which deteriorates the flavor, aroma, color and nutritional components of food (Dubois, 1995).

Encapsulation is one of the promising methods to preserve sensitive food ingredients from environmental stresses, to mask off flavors and to deliver active nutrients using coating material (Onwulata, 2013). Wheat germ oil can be protected from oxidation by means of microencapsulation.

Different types of homogenizers can be used for preparation of emulsions. Jafari et al. (2008) compared a normal mixer, a colloid mill, microfluidizer and ultrasonic homogenizer and concluded that microfluidization created the smallest emulsion droplet size and narrower distribution than ultrasonication. On the other hand, ultrasonication had an advantage over microfluidization because overprocessing was not observed during ultrasonication (Jafari et al., 2007). Klaypradit and Huang (2008) studied encapsulation of tuna oil and stated that ultrasound technology can be applied to industrial scale. Koh et al. (2014) found that similar results were obtained in whey protein solutions in the case of ultrasonic homogenization, high shear homogenization and pressure homogenization.

In literature, although there are many researches on the nutritional composition and the health promoting effects of wheat germ oil, there is only one study on microencapsulation of wheat germ oil (Yazicioglu et al., 2015). In this study, as a coating material only the effect of maltodextrin and whey protein concentrate combination was studied and the stability of wheat germ oil during storage was not investigated. In addition, the effects of different homogenization methods on wheat germ oil microcapsules were not compared. Thus, the main objective of our study was to encapsulate wheat germ oil by using different coating materials and homogenization techniques and also to investigate the effects of encapsulation on storage stability of wheat germ oil which was not studied before.

2. Materials and methods

2.1. Materials

Wheat germ oil (WGO), maltodextrin (MD) having dextrose equivalent value of 4–7, chitosan (CS), gum arabic (GA) and casein sodium salt from bovine milk (NaCa) were purchased from Sigma Aldrich Chemical Co. (St. Louis, MO, USA). The chemicals dipotassium

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phosphate (K_2HPO_4), potassium dihydrogen phosphate (KH_2PO_4), glacial acetic acid, hexane, magnesium chloride ($MgCl_2$), chloroform, potassium iodide, sodium thiosulfate, starch, isooctane (2, 2-, 4-trimethylpentane), *p*-anisidine, methanol for HPLC, acetonitrile for HPLC and DL-α-tocopherol acetate were also purchased from Sigma-Aldrich Chemical Co. (St. Louis, MO, USA). Whey protein concentrate (WPC) which contains 80 g protein/100 g dry solid was supplied from Tunçkaya Kimyevi Maddeler (Tuzla, İstanbul).

2.2. Methods

2.2.1. Preparation of coating materials

The maltodextrin (MD) and sodium salt of casein (NaCa) solutions were prepared one day before the emulsion preparation. The MD and NaCa were dissolved in distilled water at 10 g/100 mL and 30 g/100 mL concentration by weight, respectively. The solutions were mixed by magnetic stirrer (Heidolph MR 3001 K, Heidolph Instruments GmbH & Co, Schwabach, Germany) and kept in the shaking bath at 25 °C and 90 rpm overnight for complete dissolution.

Whey protein concentrate (WPC) solutions were prepared in 1 g/100 mL, 10 g/100 mL, 30 g/100 mL and 40 g/100 mL concentrations by weight, dissolving in phosphate buffer solution with pH 7.0 for 5 min by the magnetic stirrer and kept in the shaking bath at 25 °C and 90 rpm overnight for complete dissolution.

Gum arabic (GA) solution was prepared at 10 g/100 mL concentration in distilled water. Chitosan (CS) was dissolved in 0.25 g/100 mL aqueous acetic acid solution to obtain 1 g/100 mL concentration 2 h prior to emulsion preparation for complete dissolution.

2.2.2. Preparation of emulsions

After the coating materials were prepared, they were weighed in a 250 mL beaker and mixed to obtain 80 g coating material solution of ratios MD:WPC-1:3, MD:WPC-1:4, MD:GA-1:3, MD:CS-10:1, WPC:CS-10:1, WPC:CS-1:1, MD:NaCa-1:3. Wheat germ oil was added to coating material solution to obtain emulsion with core to coating ratio of 1:8.

The coating and core material mixture was first pre-homogenized in high-speed blender (IKA T25 digital Ultra-Turrax, Selangor, Malaysia) at 8000 rpm for 5 min. Then, the pre-emulsion was homogenized in Ultrasonic Homogenizer (Sonic Ruptor 400, OMNI International the Homogenizer Company, Georgia, USA) for 15 min at 40 kHz using 50% pulse. In order to prevent the temperature rise of the emulsion, the beakers were kept in ice bath during ultrasonic homogenization.

When MD:NaCa at a ratio of 1:3 was used as coating material, different homogenization techniques, which were silent crusher (Heidolph Silent Crusher S, Schwabach, Germany) and microfluidizer (Nano-dispenser NL-100, Daedeok, South Korea) were used for emulsion preparation after prehomogenization in high speed blender. The silent crusher was performed at 75,000 rpm for 15 min. In order to prevent the temperature rise in emulsion, the beakers were kept in ice bath during homogenization. Microfluidization was performed at 500 MPa for 1, 2, and 3 cycles.

After homogenization, emulsions were put into 250 mL beakers so as to be half filled and then frozen at −80 °C (CL Hetofrig, Birkrod, Denmark).

2.2.3. Freeze drying

Frozen emulsions were dried in the freeze drier (Christ, Alpha 2–4 LD plus, Osterodeam Harz, Germany) for 48 h. After drying, dried products were grinded into powders with the help of a glass rod.

2.3. Analysis of emulsions and microcapsules

2.3.1. Efficiency analysis of microcapsules

The encapsulation efficiency of microcapsules indicates the ratio of oil at the surface of the microcapsules to the encapsulated oil. It is calculated by Eq. (2.1) (Turasan et al., 2015);

$$\text{Encapsulation Efficiency}(\%) = \frac{\text{Total oil content - Surface oil content}}{\text{Total oil content}} \times 100 \quad (2.1)$$

The method to measure the surface oil of microcapsules was adapted from Calvo et al. (2010) and Millqvist-Fureby (2003). In a beaker, 5 g of microcapsules were weighed for the determination of surface oil content. The powder was mixed with 50 mL hexane using magnetic stirrer at 200 rpm for 60 s; then, it was kept in the solvent for 10 min. After 10 min, the mixture was filtered through filter paper (No. 41, Whatman, Maidstone, UK). The powder residue was again washed with 2 × 5 mL hexane. The solvent was allowed to evaporate under fume hood. Then, the extracted oil was placed in an oven at 105 °C until a constant weight was achieved for complete evaporation of solvent.

The total oil content was measured by Soxhlet extraction of microcapsules (Calvo et al., 2010). Extraction was carried out for 4 h using 250 mL hexane for 5 g of powder. The solvent in the extract was evaporated under fume hood. Then, it was dried in the oven at 105 °C until constant weight was achieved.

2.3.2. Particle size analysis of emulsions

Laser diffraction particle size analyzer was used to determine the particle size of microcapsules (Malvern Mastersizer 3000, Malvern Instruments Limited, Worcestershire, U.K.). Sauter mean diameter, D32, and span values were used to describe the particle size of emulsions. D32 is calculated by the Eq. (2.2);

$$D32 = \frac{\sum n_i d_i^3}{\sum n_i d_i^2} \quad (2.2)$$

Where, d_i represents the diameter of the particles in each size class and n_i represents the number of particles in each size class per unit volume of emulsion. Span is the width of the distribution and it is calculated by the Eq. (2.3);

$$\text{Span} = \frac{[d(0.9) - d(0.1)]}{d(0.5)} \quad (2.3)$$

where, d(0.9), d(0.5), and d(0.1) are the 90%, 50%, and 10% cumulative sizes of particle diameters respectively.

2.3.3. Storage stability of microcapsules

The storage stability of microcapsules prepared with MD and NaCa at a ratio of 1:3 using microfluidization were studied at two different temperatures of 15 °C and 45 °C. Saturated magnesium chloride solution ($MgCl_2$) was used to prepare the storage environments having the relative humidity values of 33.3 ± 0.21% and 31.10 ± 0.13% at temperatures 15 °C and 45 °C, respectively. Two identical desiccators were used to store the microcapsules at different temperatures. The microcapsules and fresh non-capsulated wheat germ oil were put into the desiccators and kept at 15 °C and 45 °C after the equilibrium is reached. Temperature and humidity data logger (EBI20 TH1, EBRO) was used to control and record the temperature and humidity values.

For the analyses of peroxide and *p*-anisidine, the samples were taken for every 10 days of 40 days storage at 15 °C and for every 7 days of 28 days storage at 45 °C. On the other hand, α-tocopherol value was analyzed for the first day and after 24 days of storage at 15 °C and at 45 °C. Then, percentage loss of α-tocopherol was calculated during storage at different temperatures.

The peroxide and *p*-anisidine values were determined to examine the oxidative stability of microcapsules during storage. Soxhlet extraction was used to extract the oil from the powder. Peroxide value (PV) and *p*-anisidine value (*p*-AV) of extracted oil samples were determined by AOCS official method (AOCS, 1998a,b).

Totox value was calculated according to Eq. (2.4);

$$\text{Totox value} = 2 \times (\text{PV}) + (\text{p-AV}) \quad (2.4)$$

α-Tocopherol analysis was done by high performance liquid