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Preparation of walnut oil microcapsules employing soybean protein isolate and maltodextrin with enhanced oxidation stability of walnut oil

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ABSTRACT

This study is aimed to enhance the stability of walnut oil by microencapsulation using soybean protein isolate (SPI) and maltodextrin (MD) as wall materials. Walnut oil microcapsules were successfully prepared by spray drying method and the encapsulation efficiency (EE) was 72.2% under the optimal conditions. The effects of the wall materials concentrations, the ratio of SPI to MD, and the ratio of oil to wall materials on the EE were examined. The structure of microcapsules was characterized by various physicochemical techniques and it confirmed the walnut oil was successfully microencapsulated. The microencapsulated walnut oil showed lower oxidation values in comparison with unencapsulated oil, highlighting a protective effect of the antioxidant. The results revealed that the oxidation stability of walnut oil was enhanced significantly by microencapsulation. Microcapsules showed good application prospect for walnut oil in food industries.

1. Introduction

Walnut oil is from walnut, which is a valuable crop and nutrient-dense food owing to its lipid and protein profile (Labuckas, Maestri, & Lamarque, 2014). Walnut oil contains high amounts of monounsaturated (oleic acid) and polyunsaturated fatty acids (PUFAs, linoleic and α-linoleic acids). PUFAs are not synthesized by human body but play an essential role in physiology (Galus & Kadzinska, 2016). Epidemiological and clinical trials suggest that omega-3 PUFA might play an active role in the prevention of coronary heart disease, arrhythmia diseases and thrombotic diseases (Bucher, Hengstler, Schindler, & Meier, 2002; Harper & Jacobson, 2001).

However, PUFAs are unstable and suffer from oxidation when exposed to air or light at even low temperature, resulting in rancid odors, undesirable flavors, hydroperoxides and other forms of spoilage (Martínez, Cecilia Penci, Ixtaina, Ribotta, & Maestri, 2013; Okpala et al., 2016). It would bring great threat to the food safety and the human health. Therefore, there are more reasons to improve the stability of walnut oil during the process of production and storage. Microencapsulation is proving to be an alternative for protecting sensitive ingredients, as well as the unsaturated fatty acids (Fuchs et al., 2006; Karaca, Nickerson, & Low, 2013).

The microcapsule technology attracts much attention in the food industry as it can satisfy the needs about health and safety of consumers (Pai, Vangala, Ng, Ng, & Tan, 2015). This technology is defined as the process in which a solid, liquid or gaseous substance, namely core, is surrounded by a continuous film of polymeric material (Bakry et al., 2016; Gallardo et al., 2013; Yang, Zeng, Xiao, & Ji, 2014a). It has many advantages such as controlling the release of flavors and fragrances, protecting the active and sensitive substances, enhancing the stability and prolonging the period of storage (Champagne & Fustier, 2007; Li et al., 2015). Furthermore, the liquid grease can transfer into solid by microencapsulation, which is more convenient to the production, application, storage and transportation (Sagalowicz & Leser, 2010).

The methods for preparing microcapsules have been widely reported. Among them, the spray-drying is the most common method used in the food industry because of its relatively low cost and straightforward (Gharsallaoui, Roudaut, Chambin, Voilley, & Saurel, 2007). The main purpose of the spray-drying is to build a

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barrier between the core material and the environment (Aghbashlo, Mobli, Madadlou, & Rafiee, 2013; Calvo, Lozano, Mansilla, & Gómez, 2012). The efficiency of the barrier mainly depends on the composition and structure of the wall material and the operation conditions during the production of microcapsules. Therefore, the selection of wall material is essential for preparing the microcapsules of good properties (Zhang et al., 2015). Maltodextrin (MD) a hydrolyzed starch and commonly used in microencapsulation of food ingredients (Carneiro, Tonon, Grosso, & Hubinger, 2013). It offers advantages such as good solubility, neutral aroma and taste, low viscosity at high concentrations, and good oxidative stability to the core materials. However, it exhibit poor emulsifying capacity and low oil-retention capability (Chang, Varankovich, & Nickerson, 2016). Therefore, the MD is necessary to be used in combination with other wall materials.

In this study, the soybean protein isolate (SPI) is selected to combine with the MD to form the wall material, attributing to its good emulsification and film-forming properties (Ortiz et al., 2009; Anwara and Kunz, 2011). The SPI is produced from the defatted soy meal, which is an abundant, high nutritional, inexpensive raw material.

In our knowledge, few study has focused on the complexation behavior of the SPI and MD as wall materials for the microencapsulation, especially for walnut oil. The objectives of the present study were to prepare and evaluate the walnut oil microcapsules using SPI and MD as wall materials by spray drying. The microcapsule morphology was to be observed by scanning electron microscopy (SEM). Fourier transform infrared spectroscopy (FTIR), X-ray diffraction (XRD) and thermogravimetry (TG) methods were used to prove the successful encapsulation. The effects of various parameters on the encapsulation efficiency (EE) of walnut oil microcapsules were examined. In addition, the improved oxidative stability was also investigated.

2. Materials and methods

2.1. Materials

Soybean protein isolate and maltodextrin was purchased from Henan Qianzhi Co. Ltd., China. Walnut oil was acquired from Sun Yat-sen Nansha Research Institute (Guangzhou, China). Petroleum ether and ether were reagent grade and provided by Guangzhou Chemical Reagent Co. Ltd., China. Deionized water was used throughout the experiment.

2.2. Preparation microcapsules

Wall solution was prepared by blending different amount of the SPI and MD in deionized water and constantly stirring for 20 min (310 rpm) by IKA-WERK blender. Different amount of walnut oil was added to the wall solution gradually at 80 °C for 1 h under vigorous stirring. An aqueous emulsion was obtained from this mixture at room temperature with a Fluke homogenizer (FA25) at 10000 r·min^{-1} for 5 min. Then it was treated by high pressure homogenizer through second emulsification at 50 MPa. Walnut oil microcapsules were produced by spray drying. The operation conditions for spray dryer (JIUPIN-015) were: diameter of the atomizer nozzle was 1.00 mm, liquid flow rate was 5 ml min^{-1}, and inlet air temperatures were 190 °C and outlet air temperatures were 75 °C, the direction of hot air was co-current. The dried microcapsules collected in a cyclone were stored in plastic bottles at 4 °C for further analysis.

3. Characterization of microcapsules

3.1. Laser diffraction particle size analysis

The particle size distribution of the inclusion complexes was measured by a laser particle size analyzer (Mastersizer-2000; Malvern Instruments Ltd, Malvern, UK). The particle size was measured using the dry sample adapter and the volume mean diameter (Vd) was recorded.

3.1.1. Analysis by fourier transformed infra-red spectrometer

FTIR spectra of walnut oil, SPI, MD, and microcapsules were determined by using a Fourier transformed infra-red spectrometer (FTIR, EQUINOX 55, BRUKER) at room temperature. All spectra were recorded from 400 to 4000 cm^{-1} with a resolution of 4 cm^{-1}.

3.1.2. Scanning electron microscopy analysis

SEM was used to analyze the morphology of microcapsules on a JSM 6330F scanning electron microscope (Hitachi, Japan). Microcapsule samples were coated with a thin layer of sputtered gold prior to examination.

3.1.3. Thermogravimetric analysis

The thermogravimetric curves (TG) were performed on the thermal analysis system Nicolet 6700. A 3.0–6.0 mg of samples were placed in aluminum crucibles and heated from 30 to 500 °C at a rate of 10 °C/min under constant N_2 flow.

3.1.4. X-ray diffraction analysis

X-ray Diffraction (XRD) was imaged by X-ray diffractometer (Empyrean, Holland). The samples were tested with a scan sped at 0.04°/min the patterns were recorded over the 2θ rang from 5 to 50. The X-ray source wavelength was 1.541874 Å.

3.1.5. Determination of encapsulation efficiency

The walnut oil on the surface of microcapsules was determined as follows. 20 mL of petroleum ether was added to 2 g powder by vigorous shaking for 5 min at room temperature. The solvent was filtered and the residue was washed again with 10 mL petroleum ether. The filtrate was collected into a pre-weight evaporating dish. The solvent was evaporated at 60 °C water bath heating and dried until constant weight of evaporating dish. The surface oil was calculated according to the mass difference of the pre-weight evaporating dish.

The total walnut oil was measured by accurately weighted 2 g microcapsules and extracted by soxhlet extraction (Dima, Patrascu, Cantaragiu, Alexe, & Dima, 2016). Ether was selected as extractant, with an extraction time of 6 h. After extraction, the solvent was evaporated completely and it was weighted to obtain the quality of total oil extracted by ether. The encapsulation efficiency (EE) was calculated from the quantitative determinations detailed above according to the modified equation (Barbosa, Borsarelli, & Mercadante, 2005), as show in Eq. (1)

$$EE\% = (1 - \text{Surface oil}/\text{Total amount of oil})*100 \quad (1)$$

3.1.6. Oxidative stability

Dried microcapsules and bulk walnut oil were placed in glass-surface vessel and stored in an oven at 40, 60 °C for 7 days, respectively. The peroxide value (PV) of the unencapsulated oil and the microencapsulated walnut oil was determined as follows. Each day 2 g powder sample and 2 g walnut oil was taken out from oven for measuring. The oil extraction method of microcapsules was the