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Quality analysis and microencapsulation of chili seed oil by spray drying with starch sodium octenylsuccinate and maltodextrin

Yuxiao Wang, Bin Liu, Xin Wen, Mo Li, Kunli Wang, Yuanying Ni *

College of Food Science and Nutritional Engineering, China Agricultural University, Beijing 100083, China
National Engineering Research Center for Fruit and Vegetable Processing, Beijing 100083, China
Key Laboratory of Fruit and Vegetable Processing, Ministry of Agriculture, Beijing 100083, China

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ABSTRACT

Chili seeds, which are a byproduct of the production of chili powder and chili paste, contain a variety of substances that can lead to useful products. The oxidation of the unsaturated fatty acids in chili seed oil can lead to off flavor for these products, so it is important to develop procedures for reducing the oxidation. In this paper, we analyzed the quality of chili seed oil extracted with supercritical fluid CO_2, studied the viscosity and stability of different emulsion formulations, and investigated a way of protecting the chili seed oil by microencapsulation with a composite wall material composed of starch sodium octenylsuccinate and maltodextrin. The oil, which was extracted with supercritical carbon dioxide, CO_2, was of high quality with an iodine value (IV) of 130.50 ± 1.75 g/100 g and a peroxide value (PV) of 2.81 ± 0.29 mmol/kg. Gas chromatography was used to identify ten different fatty acids in the chili seed oil, and 82.84% of them were found to be unsaturated. An emulsion containing 30% oil was stabilized by 10% composite wall material and spray drying using an inlet air temperature of 160 ± 2 °C and an outlet air temperature of 80 ± 2 °C. Scanning electron microscopy revealed a particle size of polyhedral microcapsules from 3 to 20 μm. The loading efficiency of the microcapsules was up to 94.35%, which suggested good liquidity and uniformity. We found that using starch sodium octenylsuccinate and maltodextrin as a composite wall material with spray drying is an effective way to encapsulate and protect chili seed oil.

1. Introduction

The chili (*Capsicum annuum* L.) is widely used in culinary preparations [1]. The fruit is consumed as fresh, dried, pickled, or powdered. The research on chili has mainly focused on the cultivation [2,3,4], color stability [5], capsaicinoids [6,7], and fungaltoxin [8,9,10,11], but there has been less research on the uses of the byproducts of chili food processing, such as the seeds generated in abundance during chili paste or powder processing. Previous chemical investigations indicate that chili seeds contain sterols, functional protein, fatty acids, and capsaicinoids [12]. There is about 20% to 25% oil in chili seeds, and over 80% of them are unsaturated. There are also capsaicinoids in seeds that are responsible for the sensory attributes of flavor, taste, and pungency. Therefore, it is important to protect the fatty acids in chili seeds from oxidation and develop procedures for producing high added value products using the ingredients from the seeds.

Microencapsulation with spray drying is an effective technique for protecting food ingredients [13]. Spray drying is widely used in food industries to deliver emulsified food ingredients, such as lipophilic bioactive ingredients [14,15]. The creation of spray dried systems based on oil-in-water emulsions involves making emulsions using homogenization, followed by the addition of a wall material, such as gums [13,16,17,18], protein [19,20,21,22], and natural and modified polysaccharides [23,24,25,26]. Starch sodium octenylsuccinate (SSOS) has been widely studied as an excellent emulsifier. This is mainly due to the introduction of hydrophobic alkenyl and hydrophilic carboxyl groups to the relatively hydrophilic structure of starch molecules. Compared with gum arabic and gelatin, SSOS has an advantage of being an inexpensive and abundant material resource.

When producing emulsions with a high percentage of solids, lower viscosity is important for spray drying [27,28]. There is very little in the scientific literature on encapsulating the chili seed oil with SSOS. Therefore, the objectives of the present work were to analyze the quality of chili seed oil extracted with supercritical fluid CO_2, to study the viscosity and stability of different emulsion formulations, and to encapsulate the chili seed oil with SSOS by spray drying.

* Corresponding author at: College of Food Science and Nutritional Engineering, China Agricultural University, Box 303, No. 17 Tsinghua East Road, Beijing 100083, China.
E-mail address: niyycau@hotmail.com (Y. Ni).

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2. Materials and methods

2.1. Materials

The chili (*Capsicum annuum* L.) seed was kindly offered by Chenguang Biotech Group Co. Ltd. (Hebei, China). Acetic acid, cyclohexane, potassium iodide, sodium thiosulfate, anhydrous sodium carbonate, starch, salicylic acid, hydrochloric acid, and sodium sulfate monohydrate were obtained from the Sinopharm Chemical Reagent Co. Ltd. (Shanghai, China). Isooctane (for the high performance liquid chromatography, HPLC), tridecane acid methyl ester (for HPLC, ≥ 99.0%), fatty acid methyl ester mixture (FAME) as an analytical standard, and iodine monochloride were bought from Sigma-Aldrich (St. Louis, MO, USA). Starch sodium octenylsuccinate (SSOS), soybean protein isolated (SPI), gelatin (GA), and maltodextrin (MA) were kindly provided by Yundong Jiang who works in Cargill (Beijing, China). Unless otherwise mentioned, the chemicals and reagents used in this experiment were analytical grade.

2.2. Extracting chili seed oil with supercritical CO_2

Before supercritical fluid extraction, the chili seed was ground into powder, sifted with a 30 mesh sieve, and stored at −4 °C. Extraction was used to separate compounds in the ground chili seeds. The extraction rate and the yield of chili seed oil were determined by temperature, pressure, extraction bed size, and solvent flow rate [29]. The supercritical fluid extractions (SFE) were done at a pressure of 25 ± 0.5 MPa, a temperature of 45 ± 2 °C, and an extraction time of 70 min.

2.3. Physicochemical properties of chili seed oil

Physicochemical properties of chili seed oil were measured according to the national standard methods of China (GB). The GB/T 5528-2008 official method was used to determine the moisture content and amount of volatile matter by measuring the loss of mass after heating the samples in a glass container at 103 ± 2 °C until the mass remained constant. Iodine value (IV), peroxide value (PV), and levels of capsaicinoids were measured according GB/T 5532-2008, GB/T 5530-2005, GB/T 5538-2005, and GB/T 30389, respectively. Briefly, Iodine value (IV) was determined by adding 25 mL Wijs reagent to the sample and making the solution in the dark. Subsequently, 20 mL of 10% KI and 100 mL water were added. The solution was titrated with standard $Na_2S_2O_3$ 0.1 N using some drops of starch solution as indicator, since it gives an intensely blue complex with iodine. Peroxide value (PV) was measured by adding 50 mL organic solvent mixture (chloroform: acetic acid, 2:3) to the sample. After vigorously shaking, 0.5 mL of saturated KI solution was added to the mixture. The mixture was kept in the dark for 1 min and 30 mL of distilled water were added. The mixture was added 0.5 mL of starch solution (1%, w/v) as an indicator. All determinations were carried out three times.

2.4. Analysis of fatty acids with gas chromatography

The methyl esterification of fatty acids was performed by using the boron trifluoride method according to the AOAC Official Method 969.33. The analytical conditions for the gas chromatograph (GC) were based on what was reported by Zhu [30] with some modifications. After the dilute solution containing the chili seed oil's FAMEs was filtered through a 0.45 μm organic membrane filter, 1.0 μL of the filtrate was injected into an Agilent 7890A network gas chromatograph system with an FID detector. The separation of the FAMEs was performed in a fused silica capillary column HP-FFAP (30 m × 0.25 mm i.d., 0.25 μm; Agilent Technologies, J&W Scientific, USA). The temperature of the injector was set at 230 °C. The oven temperature was programmed to yield an initial column temperature of 180 °C for 5 min, which was increased at 2 °C/min to 210 °C and then increased again at 5 °C/min to 250 °C with a split ratio of 1:50. Peaks were identified by taking into account the retention times of FAME standards. Results were reported in relative peak area percentages. All analyses were performed three times.

2.5. Emulsion of chili seed oil

Starch sodium octenylsuccinate (SSOS), soybean protein isolated (SPI), gelatin (GA), and maltodextrin (MA) were selected as single wall materials. SSOS/MA, SPI/MA and GA/MA mixed with a mass ratio of 1:1 were used as the composite wall materials. The wall materials were dissolved in Milli-Q (EMD Millipore, Billerica, MA, USA) water with magnetic stirring at 500 rpm and at room temperature (25 ± 1 °C) for 5 h before preparing the emulsion for drying. Chili seed oil was gradually added to the wall material solution, stirring at 10,000 rpm for 3 min using a disperser (T18 digital Ultra Turrax, IKA). The emulsions, after high speed shearing, were immediately subjected to a homogenization (GEA Niro, Italy) to ensure complete emulsification of the chili seed oil. The SSOS/MA ratios (SSOS and MA mixed at the weight ratio from 1:1 to 5:1 wt/wt), contents of wall material (concentration of the wall material dissolved in water from 5 wt.% to 25 wt.%), and core material load (concentration of oil by wall material from 20 wt.% to 45 wt.%) were used to determine the optimum compositions that yield a relatively low viscosity and high stability.

The viscosity and stability of the emulsions were measured according Piriyaprasarth [31] with some modifications. The viscosity was determined with a rotational viscometer (Fungilab, Spain) with a spindle LCP at ambient temperature (25 °C). The emulsion was transferred into a glass tube and then placed in an accelerated storage apparatus at 40 °C for 24 h. After the accelerated storage step, some formulations separated into a "cream layer" at the top (optically opaque) and a "serum layer" at the bottom (transparent or turbid). The total height of the emulsions (H_E) and the height of the serum layer (H_S) were measured. The extent of creaming was characterized by creaming index using the following equation:

$$\text{Stability}(\%) = \left(1 - \frac{H_S}{H_E}\right) \times 100 \quad (1)$$

2.6. Spray drying

The spray-drying process was performed in a laboratory-scale Mini Spray Dryer Buchi B-290 (Buchi Labortechnik AG, Switzerland) with a two-fluid nozzle with a cap orifice diameter of 0.5 mm. The air atomizing pressure was 4.4 L/h, the air inlet temperature was 160 ± 2 °C, the air outlet temperature was 80 ± 2 °C, the atomized air flow rate was 538 L/h, the pump setting was 15%, and aspirator setting was 80%. The powder was collected in aluminized foil mailers and stored at 4 °C. Scanning electron microscopy was used to observe the particle size and microstructure of the chili seed oil microcapsules.

The surface oil was determined by using hexane to extract the free oil. The powder was extracted with hexane in a flask, which was shaken by hand for the extraction of free oil. After 2 min, the solvent mixture was filtered through a filter paper and the powder collected on the filter was rinsed three times. The loading efficiency (LE) of the microcapsules was determined by using the soxhlet extraction method according to China GB/T 5512-2008. Petroleum ether was used to extract the left powder at 50 °C for 6 h with a Soxhlet extractor (SZF-06A, Hangzhou Huier Instrument Equipment Co., LTD, China), and the extra petroleum ether was evaporated (R-100, BUCHI). The LE was calculated using Eq. (2) according to Feng [32]:

$$\text{LE}(\%) = \frac{\text{Total amount of oil encapsuled}}{\text{Total amount of oil added}} \times 100 \quad (2)$$