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Modification of deoiled cumin dietary fiber with laccase and cellulase under high hydrostatic pressure

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ABSTRACT

In this study, we evaluated the effects of high hydrostatic pressure (HHP) and enzyme (laccase and cellulase) treatment on the structural, physicochemical, and functional properties and antioxidant activity of deoiled cumin dietary fiber (DF). HHP–enzyme treatment increased the contents of soluble dietary fiber (SDF) (30.37 g/100 g), monosaccharides (except for glucose), uronic acids, and total polyphenol. HHP–enzyme treatment altered the honey-comb structure of DF and generated new polysaccharides. DF modified by HHP–enzyme treatment exhibited improved water retention capacity (10.02 g/g), water swelling capacity (11.19 mL/g), fat and glucose absorption capacities (10.44 g/g, 22.18–63.54 mmol/g), α-amylase activity inhibition ration (37.95%), and bile acid retardation index (48.85–52.58%). The antioxidant activity of DF was mainly correlated to total polyphenol content ($R = 0.8742$). Therefore, DF modified by HHP–enzyme treatment from deoiled cumin could be used as a fiber-rich ingredient in functional foods.

1. Introduction

Cumin (*Cuminum cyminum*), the second largest aromatic crop after pepper in the world, is used as flavoring and seasoning agent in foods. Essential oil and oleoresin, which take up 20–25% of whole cumin, are mainly extracted for exportation in industry and have extensive biological activities, such as antibacterial, anti-tumor, anticancer, immunoregulation, etc. (Sowbhagya, 2013). Deoiled cumin, which generates from essential oil and oleoresin extraction, is an important source of total dietary fiber (TDF, 62.10%), which is associated with reduced risk of obesity, diabetes, cardiovascular diseases, and certain types of cancers (Galisteo, Duarte, & Zarzuelo, 2012; Sowbhagya, Suma, Mahadevamma, & Tharanathan, 2007). Soluble dietary fiber (SDF) appears to be more bioactive than insoluble dietary fiber (IDF) due to its fermentability and viscosity; approximately 30% SDF should be consumed on a daily basis (Galisteo et al., 2012; Mateos-Aparicio, Mateos-Peinado, & Rupérez, 2010). Deoiled cumin dietary fiber (DF) consists of 12.26% SDF and 71.92% IDF; the low SDF content of deoiled cumin DF limits its applications in the food industry.

Chemical, biological, and physical methods have been developed to modify TDF and increase SDF content in foods. Chemical methods with limited reaction conditions lead to low SDF conversion efficiency and introduce detrimental chemical groups (Sangnark & Noomhorm, 2003). Biological methods are expensive

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because they require purified enzymes and bacterial strains. Additionally, the fermentation conditions are difficult to control (Santala, Kiran, Sozer, Poutanen, & Nordlund, 2014). Physical methods, e.g., micro-fluidization, ultrafine grinding, high-pressure homogenization, and blasting extrusion, improve the physicochemical and functional properties of DF by decreasing particle size rather than by increasing SDF content (Chen, Gao, Yang, & Gao, 2013; Jing & Chi, 2013; Raghavendra et al., 2006; Wennberg & Nyman, 2004). Therefore, it is important to develop a modification method that improves DF quality and increases SDF content.

High hydrostatic pressure (HHP) treatment is an emerging non-thermal physical modification method that improves the appearance, flavor, texture, and nutritional quality of foods (Balny, 2002; Mateos-Aparicio et al., 2010). Moreover, pressure levels <300 MPa enhance enzyme activity and shorten reaction time by reducing substrate particle size, cell wall and membrane degradation, and cell deformation (Eisenmenger & Reyes-De-Corcuera, 2009; Kim & Han, 2012). In light of this, HHP–enzyme treatment may represent a high-efficiency and environmental friendly method for improving DF quality.

The objective of this study was to increase the SDF content of deoiled cumin DF by HPP–enzyme treatment using commercial laccase and cellulase for the enzymatic hydrolysis. The chemical, structural, physicochemical, and functional properties of DF were evaluated, and antioxidant activities were measured.

2. Materials and methods

2.1. Materials

Cumin (Dunyu No. 1 variety) was purchased from Dunhuang Seed Co., Ltd (Gansu, China). Deoiled cumin DF was prepared by the method reported by Ma et al. (2015).

Alcalase 2.4 L (enzyme activity: ≥3000 U/mL) was obtained from Novozymes (Copenhagen, Denmark); cellulase (enzyme activity: ≥10,000 U/g) was purchased from Pharmaceutical Ind. Co., Ltd (Tokyo, Japan); laccase (enzyme activity: ≥50 U/mg), rhamnose, arabinose, galactose, glucose, xylose, galacturonic acid, glucuronic acid, trifluoroacetic acid, taurocholic acid sodium salt hydrate, fluorescein sodium, Folin–Ciocalteu reagent, and AAPH were purchased from Sigma–Aldrich (Louis, USA). The commercial glucose (GOPOD) assay kit was obtained from Megazyme (Wicklow, Ireland). All other chemicals were of analytical grade.

2.2. Preparation of modified DF

Initially, the optimum temperature, pH, enzyme-to-substrate ratio (E/S), pressure, and reaction time were evaluated by single-factor experiments (data not shown). DF was mixed with 0.2 M phosphate buffer solution (PBS) at pH 6.5 (1:10, w/v) and laccase (E/S, 15 U/g), sealed in a polyethylene bag, and subjected to 200 MPa at 30 °C for 25 min in an HHP system (L2-600/2, Huataisenmiao Biological Engineering Technology Co., Ltd, Tianjin, China). Cellulase (E/S, 210 U/g) was added to the bag, and the mixture was subjected to 200 MPa at 50 °C for 15 min. Subsequently, the mixture was heated in a boiling water bath for 15 min to terminate enzymatic hydrolysis and allowed to cool to room temperature. Thereafter, the mixture was centrifuged at 7000 *g* for 15 min to remove supernatant, and resulting precipitate was freeze-dried at −60 °C (FD5-3, American Intl Group, USA).

2.3. TDF, IDF, and SDF determination

Moisture, TDF, IDF, and SDF contents were measured by AOAC official methods (method 925.09 and 991.43; 2000).

2.4. Monosaccharide and uronic acid determination

Neutral sugars (rhamnose, arabinose, galactose, glucose, and xylose) and uronic acids (galacturonic and glucuronic acids) were analyzed following acid hydrolysis of TDF with 4 M trifluoroacetic acid at 121 °C for 2 h. Individual sugars were quantified by high-performance anion-exchange chromatography with pulsed-amperometric detection (HPAEC-PAD) as reported by Mei, Mu, and Han (2010).

Standard solutions containing neutral sugars and uronic acids (0.2–20 μg/ml) were prepared to confirm the linearity of the detector and to determine the relative response factors.

2.5. Particle size distribution

Particle size distribution of unmodified and five modified DFs was determined by Laser Diffraction in a Baite Particle Size Analyzer (BT-9300, Dandong Baite Instrument Co., Ltd, Dandong, Liaoning, China). 1 g dehydrated sample (after freeze-drying) was mixed with 10 mL deionized water to make a 10% suspension which was used for analysis. Particle size distribution parameters recorded included median diameter [D_{50} (μm)], volume–surface mean diameter [$D_{3,2}$ (μm)], and volume-weighted mean diameter [$D_{4,3}$ (μm)]

2.6. Scanning electron microscopy (SEM)

The surface and microstructure of DFs were observed under a scanning electron microscope (S-3400, Hitachi, Ltd., Tokyo, Japan) at 15 kV. Dehydrated samples were ground into fine powder using a grinder (RT-04, Beijing Kaichuangtonghe Technology Development Co., Ltd, Beijing, China) at medium speed for 2 min and then were placed on double-sided conducting adhesive tape and coated with a 10-nm gold layer. Representative micrographs were taken at 1000× magnification.

2.7. Fourier-transformed infrared spectroscopy (FTIR)

DFs were thoroughly mixed with KBr (1:250, w/w) and pelletized. The IR spectra of DFs were recorded in a Tensor 27 spectrometer (Bruker, Germany) from 400 to 4000 cm^{-1} with 32 scans and 4 cm^{-1} resolution.

2.8. Physicochemical and functional properties

2.8.1. Physicochemical properties

2.8.1.1. Water retention capacity (WRC). Samples (1 g) were hydrated with 30 ml distilled water at room temperature for 18 h and centrifuged at 3000 *g* for 20 min. The weight of residue was recorded both prior to drying (fresh weight) and after drying at 105 °C (dry weight) until a constant weight was obtained. WRC was calculated by Eq. (1) (Sowbhagya et al., 2007):

$$\mathrm{WRC(g/g)} = \frac{m_f - m_d}{m_d} \quad (1)$$

where m_f is the weight of the fresh residue (g) and m_d is the weight of the dry residue (g).

2.8.1.2. Water swelling capacity (WSC). Samples (0.2 g) were hydrated with 10 ml distilled water in a graduated test tube at room temperature for 18 h. The bed volume was recorded. WSC was expressed as volume of water (ml) per sample weight (g) (Sowbhagya et al., 2007).

2.8.1.3. Fat adsorption capacity (FAC). FAC was determined by the method reported by Abdul-hamid and Luan (2000) with slight modifications. Samples (0.2 g) were mixed with 30 ml sunflower oil for