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Structural characteristics and physicochemical properties of okara (soybean residue) insoluble dietary fiber modified by high-energy wet media milling

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ABSTRACT

Changes in structural characteristics and physicochemical properties of okara insoluble dietary fiber (IDF) during high-energy wet media milling process were analyzed. Particle size of the IDF was effectively reduced from 66.7 μm to 544.3 nm after 6 h of milling and kept constant ($p > 0.05$) as the milling process prolonged. As the particle size decreased, lightness and whiteness of the IDF significantly ($p < 0.05$) increased, zeta potential decreased continuously ($p < 0.05$). Swelling power, water solubility index and apparent viscosity extensively increased ($p < 0.05$) after 1 h of milling and then steadily decreased ($p < 0.05$). Electron microscopy observations showed that the IDF was changed from regular and compact rods to aggregates with puffed morphology. Fourier transform infrared spectroscopy (FTIR) and X-ray diffraction (XRD) analysis revealed that intermolecular hydrogen bonds and crystalline structure of the IDF polysaccharides were disturbed after milling.

1. Introduction

Okara (soybean residue) is the by-product produced from soy milk and soy curd (tofu) production process. Okara consists of 50–60 g/100 g dietary fiber on dry weight basis, of which >90 g/100 g is insoluble dietary fiber (IDF) (Vong & Liu, 2016). The okara IDF is mainly composed of cellulose, hemicellulose (40–60 g/100 g on dry weight content) and lignin (Guermani et al., 1992). The monomers in polysaccharides of okara are mainly glucose, uronic acid, galactose, arabinose, xylose, fucose, rhamnose and mannose (Mateos-Aparicio, Mateos-Peinado, & Rupérez, 2010; Redondo-Cuenca, Villanueva-Suarez, & Mateos-Aparicio, 2008). Okara has been reported to possess various functional properties, for instance, reduction in caloric intake and maintaining healthy serum and hepatic lipid profile (Pŕestamo, Ruṕerez, Espinosa-Martos, Villanueva, & Lasuncíon, 2007; Villanueva, Yokoyama, Hong, Barttley, & Rupérez, 2011). However, currently, it is mainly used for animals feed or dumped in the landfills, resulting in economic loss.

IDF from by-product is not commonly incorporated in food products due to its adverse effects on food quality, like sensory and functionality (Aravind, Sissons, Ergan, & Fellows, 2012). Different modification methods such as chemical, enzymatic, and physical methods have been employed to modify physicochemical and health related properties of IDF (Chau, Wang, & Wen, 2007; Chen, Gao, Yang, & Gao, 2013; Liu et al., 2016; Qi et al., 2016; Wen, Niu, Zhang, Zhao, & Xiong, 2017). Physical methods own advantages over the other methods, like simple technology, low cost and sustainability (Sangnark & Noomhorm, 2003). The physical methods, for instance high-pressure homogenization, micro-fluidization and ultrafine grinding, have been reported to effectively modify physicochemical and functional properties of IDF from okara, oat, peach and wheat, etc., mainly by decreasing their particle size (Auffret, Ralet, Guillon, Barry, & Thibault, 1994;

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Chen et al., 2013; Liu et al., 2016; Zhu, Huang, Peng, Qian, & Zhou, 2010). Zhu, Du, Li, and Li (2014) reported that ultrafine milling of buckwheat hull IDF to micrometer range effectively enhanced its hydration capacity and free radical inhibition activity. Dry media milling was shown to be an efficient tool for the reduction of wheat IDF particle size to submicron level with increase in its surface area and modification of functional properties (Zhu et al., 2010). Particle size reduction of IDF using different micronization technologies may affect breakage efficiency, physicochemical (e.g., water-holding capacity, swelling capacity, oil-holding capacity, and cation-exchange capacity) and functional properties (e.g., glucose adsorption capacity, amylase inhibitory activity, pancreatic lipase inhibitory activity, and cholesterol-lowering activity) of the end products (Chau et al., 2007; Sangnark & Noomhorm, 2003).

Wet media milling technique has been successfully utilized in paint, biomaterials, pharmaceutical, and food industries for the preparation of ultrafine/nanometer rang particles, providing good surface properties like dispersibility and solubility (Chen, Shen, & Yeh, 2010; Zhang, Zhang, & Xia, 2012; Zhang et al., 2016). During wet media milling, materials in the suspension are downsized to nanometer range by the shearing forces provided by collision among media beads, walls of the milling chamber, and the material itself. Chen et al. (2010) and Zhang et al. (2012) applied media milling to starch and chitosan, respectively, and reported that wet media milling could effectively reduce the particle size to nanometer range with significantly enhanced water solubility, swelling power and viscosity of the material. However, high-energy wet media milling has not been investigated for the modification of okara IDF as so far.

Therefore, the objective of this study was to explore the possibility for preparation of nanometer range okara IDF particles by high-energy wet media milling and to investigate the related changes in structure and physicochemical properties during the milling process.

2. Materials and methods

2.1. Materials

Soybean was purchased from a local market near Huazhong Agricultural University. Okara was obtained from the production of tofu processing according to the Chinese traditional method. Briefly, the obtained soybean was soaked in water for 16 h at a weight ratio of 0.1–1. The soaked soybean was crushed using a kitchen blender (JYL-G12E, Jiuyang Co., Ltd., Hangzhou, China) for three times and 30 s each time. The slurry was further crushed using a colloidal mill. Okara obtained after filtering the slurry with linen cloth was washed with tap water three times, dried at 55 °C for 48 h, coarsely milled, packaged and stored in a desiccator. Contents of moisture, ash, fats, crude protein and crude fiber were determined to be 4.9, 3.6, 11.4, 25.3 and 53.8 g/100 g okara, respectively (AOAC, 2002).

2.2. Preparation of IDF

IDF was extracted from the okara as prepared above according to the method of Liu et al. (2016) with slight modification. Briefly, the okara was mixed with sodium hydroxide solution (pH 11) at a weight ratio of 1–15 and subsequently stirred at 1200 rpm for 2 h under 50 °C. After filtration with linen cloth, the solid residue was neutralized by washing several times with distilled water prior to draining off. The drained solid residue was added with hydrochloric acid solution (pH 2) at a weight ratio of 1–15 and then stirred at 1200 rpm for 2 h under 60 °C. After filtration, pH of the residues was adjusted to neutral by washing with distilled water, followed by drying in an oven at 60 °C for 48 h. Moisture, ash, protein, and dietary fiber content were determined to be 6.7, 5.9, 0.5 and 81.4 g/100 g, respectively. Fat content was less than 0.1 g/100 g (AOAC, 2002). The yield of the IDF was 33.3 g/100 g dried okara.

2.3. Media milling of IDF

The dried okara IDF was subjected to coarse milling for 30 s using a pulverizer (BJ-500A, Baijie Electrical Appliance Co. Ltd., Hangzhou, China) and screened through a sieve (105 μm). The sieved IDF was added with deionized water and the total solid content was adjusted to 3 g/100 g. The IDF suspension was loaded into milling chamber of a high-energy wet media mill (MINI ZETA 03E, Netzsch-Geratebau GmbH, Selb, Germany) through the hopper and milled under following operation parameters: media (yttria-stabilized tetragonal zirconia) diameter of 0.8 mm, filling ratio of 80%, and agitation speed of 3000 rpm. The media milling treatment was performed in triplicate and the obtained samples were subsequently characterized.

2.4. Measurement of particle size

Particle size of the IDF during wet media milling was determined according to Zhang et al. (2016). The IDF particles in micrometer range (1–1000 μm) (MIDF) were analyzed by laser light scattering using a Mastersizer-2000 (Malvern Instruments Ltd., Malvern, U.K.). And the IDF particles in nanometer range (1–1000 nm) (NIDF) were measured by Malvern Nano ZS90 Zetasizer (Malvern Instruments Ltd., Malvern, U.K) based on dynamic light scattering.

2.5. Electron microscopy

Morphological characterization of the MIDF was observed using a scanning electron microscope (JSM-6390LV, NTC, Tokyo, Japan). Before scanning, sample was dried in natural air circulation and sputter-coated. The NIDF particles prepared by wet media milling were imaged for morphological study using a transmission electron microscope (H-7000, Hitachi Ltd., Tokyo, Japan).

2.6. Fourier transform infrared spectroscopy (FT-IR)

The IDF with different milling time was freeze-dried (FD-1A-50, Boyikang Laboratory Instruments Co., Ltd, Beijing, China) and changes in molecular structure was measured according to Ma and Mu (2016a) using a FT-IR spectrometer (NEXUS 470, Thermo Fisher Scientific, Waltham, MA, USA) at 400 to 4000 cm^{-1} wavenumbers with 32 scans per sample.

2.7. X-ray diffraction (XRD)

Crystalline structural analysis of the freeze-dried samples was performed on an advance X-ray diffractometer (D8, Bruker AXS, Karlsruhe, Germany) using Cu Kα radiation ($\lambda = 0.154$ nm) at generator voltage of 40 kV and an incident current of 40 mA. The pattern was recorded with a scanning speed of 1°/min over a diffraction angle (2θ) range of 5-70°. The degree of crystallinity was calculated according to Ma and Mu (2016b) by calculating area under the curve using Peak Fit v4.12 according to the following equation: