



Physicochemical and functional properties of coconut (*Cocos nucifera* L.) cake dietary fibres: Effects of cellulase hydrolysis, acid treatment and particle size distribution

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ABSTRACT

Effects of cellulase hydrolysis, acid treatment and particle size distribution on the structure, physicochemical and functional properties of coconut cake dietary fiber (DCCDF) were studied. Results showed that both the cellulase hydrolysis and acid treatment contributed to the structural modification of DCCDF as evident from XRD, FT-IR and SEM analysis. Moreover, the cellulase hydrolysis enhanced soluble carbohydrate content, water holding capacity (WHC) and swelling capacity (WSC), α -amylase inhibition activity (α -AAIR), glucose dialysis retardation index (GDRI) and cation-exchange capacity (CEC) of DCCDF; but it had undesirable effects on colour, oil holding capacity (OHC) and emulsifying capacity (EC). On other hand, acid treatment decreased the WHC, WSC and GDRI, but improved the colour, CEC, OHC and emulsion stability of DCCDF. Furthermore, the WHC, WSC and EC of DCCDF increased as the particle size reduced from 250 to 167 μm , while the GDRI, OHC, α -AAIR and emulsion stability decreased with decreasing particle size.

1. Introduction

Many reports indicate numerous health benefits associated with an increase intake of dietary fiber (DF), including reduced risk of coronary heart disease, diabetes and some forms of cancer (Elleuch et al., 2011). In the food industry, DF is usually added to improve the nutritional characteristics as well as the texture, colour, flavour and taste of foods. DFs with high antioxidant activity can be added to fatty foodstuffs to improve oxidative stability and prolong shelf life (Peerajit, Chiewchan, & Devahastin, 2012). Recently, DFs from food industry byproducts have received more and more attention for their low cost, extensive availability and health benefits, including their antioxidant, emulsifying and texture modifying properties (Elleuch et al., 2011).

The extent to which specific DF preparations exert their physiological and technological effects is dependent on the chemical composition, interrelated structural and physicochemical properties (Izydorczyk, Chornick, Paulley, Edwards, & Dexter, 2008). DFs with a high content of soluble dietary fiber (SDF) can regulate and improve the intestinal microflora, prevent diabetes and other cardiovascular diseases (Galisteo, Duarte, & Zarzuelo, 2008). Meanwhile DFs with a high water holding capacity (WHC) and water swelling capacity (WSC) can enhance satiety, increase fecal volume and shorten defecation time,

thereby reducing the risk of obesity, constipation and colon cancer (Peerajit et al., 2012). It was also found that DFs with a high binding ability can effectively accelerate the excretion of bile acids, in turn preventing epithelial cell and DNA damage (Ma & Mu, 2016).

Most of the dietary fiber from plant sources is classified as insoluble dietary fiber (IDF), which contains functional groups like phenolics, carboxylic acids, aldehydes, ketones and ether linkages (Elleuch et al., 2011). These groups have a strong affinity to bind water, oil or toxic metal ions. However, typical processing methods of DFs like alkali extraction and enzymatic hydrolysis cannot adequately expose these groups or binding sites. Thus, several physical, chemical and enzymatic treatments, such as micronization, shear emulsifying, acidic treatment, as well as cellulase and/or xylanase hydrolysis, have been tested (Jing et al., 2015; Zhu, Du, Zheng, & Li, 2015; Ma & Mu, 2016). It was evident that enzymatic extraction assisted with cellulase could increase SDF content of DFs, and also improved some physicochemical and functional properties of DFs, such as WHC, emulsifying properties, entrapment capacity and adsorption ability (Cheng et al., 2017). Jing et al. (2015) found that acidic treatment could improve the porosity and crystallinity, and in turn enhance oil holding capacity of rice bran dietary fiber. Daou and Zhang (2013) reported that cellulase could increase the glucose dialysis retention index and cation exchange capacity

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of rice bran.

Moreover, the extent of physical, chemical and enzymatic treatment strongly depends on the particle size of DFs. Reduction in particle size may influence the structure, porosity and surface structure of DFs, resulting in modification of physicochemical and functional properties (Peerajit et al., 2012). Sangnarka and Noomhomb (2003) reported that reduction in particle size increased the WRS and WSC of sugarcane bagasse DFs; whereas Ma and Mu (2016) found that cumin DFs with particle size > 125 µm exhibited better functional properties than those with smaller particle sizes.

Coconut cake is a good source of dietary fiber for its high content of DF (14%) and low cost (Thaiphanit & Anprung, 2016). However, coconut cake dietary fiber is also classified as IDF since its SDF content is only 4% (Yalegama, Nedra Karunarathne, Sivakanesan, & Jayasekara, 2013). Although some physicochemical properties of coconut cell wall polysaccharides from virgin coconut oil residue and coconut milk residue have been studied (Yalegama et al., 2013), there is little information on the effect of acidic treatment and enzymatic hydrolysis on the structural, physicochemical and functional properties of coconut cake dietary fiber.

The aims of this paper are: (1) to evaluate the effects of acidic treatment and cellulase hydrolysis on physicochemical and functional properties of defatted coconut cake dietary fiber (DCCDF) and (2) to study the modifications of physicochemical and functional properties induced by changes in particle sizes.

2. Materials and methods

2.1. Materials

Coconut cake was obtained from the Nanye coconut food Co., Haikou, China. Papain (1.0×10^4 U), α -amylase (2.0×10^4 U), glucoamylase (5.0×10^4 U) and cellulase (3.0×10^5 U) were purchased from Shanghai DINGUO Biotech. Co., Ltd. (China). The monosaccharide standards including l-arabinose, d-galactose and aminoglucose were purchased from Sigma. Other chemicals and reagents were of analytical grade.

2.2. Defatted coconut cake preparation

One kilogram of coconut cake was dried at 50 °C for 4 h in a dryer, grated into a crude powder with a mill and then defatted three times with *n*-hexane (1:10, g/ml). The defatted powder was milled again until more than 90% material passed through a US # 40 mesh sieve to get a fine powder of defatted coconut cake (DCC).

2.3. Dietary fiber extraction, acidic treatment and enzymatic hydrolysis

2.3.1. Preparation of defatted coconut cake dietary fiber

Following the method of Ma and Mu (2016), 100 g of DCC was suspended in 1000 ml phosphate buffer (0.1 M), 1 g of α -amylase was added and the solution was stirred gently at 90 °C for 2 h. The mixture was cooled and adjusted to pH7.0, and 0.5 g papain was added. After incubation at 50 °C for 2 h, the reaction solution was adjusted to pH2.0, mixed with 0.5 g glucoamylase and incubated at 60 °C for 2 h. The mixture was incubated at 100 °C for 10 min to inactivate the enzymes, and cooled to room temperature (RT) and filtered using 100-mesh linen. The residue was collected, washed with deionized water, dried at 50 °C for 12 h, and then DCCDF was obtained.

2.3.2. Acidic treatment

Based on the modified method described by Jing et al. (2015), with few modifications, 50 g of DCCDF was suspended in 500 ml of 1 M NaOH. After stirring at 60 °C for 2 h, the suspension was filtered through linen, and the residue was collected. This residue was continuously soaked in 1 M HCl at 60 °C for 30 min, then neutralized,

filtered, washed with deionized water and dried in a forced air-oven at 60 °C for 8 h. Then DCCDF with acidic treatment (DCCDF-A) was obtained.

2.3.3. Enzymatic hydrolysis

Fifty grams of DCCDF described above were suspended in 500 ml of phosphate buffer (0.1 M, pH5.0) and mixed with 0.3 g cellulase. After incubation at 50 °C for 1 h, the mixture was heated at 100 °C for 10 min and then cooled to RT and filtered using 100-mesh linen (Daou & Zhang, 2013). The residue was collected, dried at 50 °C for 12 h and named as DCCDF-C.

2.4. Chemical and monosaccharide composition analysis

Moisture (method 925.09), protein (method 955.04), fat (method 920.39), starch (method 996.11), ash (method 942.05), total dietary fiber (TDF), IDF, and SDF (method 991.43) contents were measured by American Association of Cereal Chemists official methods (AACC 1986, 1995). Polyphenol content was determined by the Folin-Ciocalteu colorimetric method (Chandrasekara & Shahidi, 2010).

The monosaccharide composition of DCC and the DFs (DCCDF, DCCDF-A and DCCDF-C) was measured by a modified method reported by Chau and Huang (2003). The fiber was hydrolyzed in 12 M H₂SO₄ at 40 °C for 1 h. The hydrolysate was diluted with deionized water to make the final concentration of H₂SO₄ 1 M and autoclaved at 121 °C for 1 h. The obtained hydrolysate was neutralized with KOH, filtered through a 0.45 µm membrane and then injected into an ICS-5000 HPLC system (DIONEX Co., Sunnyvale, USA) equipped with a pulse ampere detector. The mobile phase was H₂O/NaOH/NaAc:78.2/1.8/20 at a flow rate of 0.5 ml/min, at 25 °C.

2.5. Particle size distribution

The obtained DCC and DCCDF, DCCDF-A and DCCDF-C were ground into a fine powder and separated by a sieve shaker (Model VE 100, Retch, Germany) with a series of sieves (mesh No. 40, 60, 80 and 100). Each sample was placed in the top sieve with the largest mesh and shaken for 5 min at an amplitude setting of 2 mm, disassembled and stirred lightly, and then shaken for an additional 5 min. The residue remaining on each sieve was weighed and expressed as a percent of the original sample weight (g/100 g). Moreover, the particle size distribution parameters expressed as Sautermean diameter $D_{3,2}$ (µm) and specific surface area of the DFs obtained from the sieving mesh were determined by a Laser Diffraction Particle Size Analyzer (MS3000, Malvern instruments Ltd., UK).

2.6. Colour

Colour values of DCC and the DFs were measured using a NS800 spectrophotometer (Shenzhen 3NH Technology Co. Ltd., China). The colour values were expressed using CIE Lab* coordinates where L^* represents the luminosity (0 = black; 100 = white), a^* the redness ($a^* > 0$) or greenness ($a^* < 0$) and b^* the blueness ($b^* > 0$) or yellowness ($b^* < 0$). The DCC with particle size of more than 250 µm, which did not show any apparent browning was taken as a reference. The total colour difference (ΔE) was then determined using the following equation:

$$\Delta E = \sqrt{(L-L_0)^2 + (a-a_0)^2 + (b-b_0)^2} \quad (1)$$

The subscript "0" in the equation refers to the reference.

2.7. Scanning electron microscopy

The surface and microstructure of DFs were observed by Scanning electron microscopy (S-3400 scanning electron microscope, Hitachi,

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