



Analytical Methods

Preparation and physicochemical properties of soluble dietary fiber from orange peel assisted by steam explosion and dilute acid soaking

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ABSTRACT

The coupled pretreatment of orange peel with steam explosion (SE) and sulfuric-acid soaking (SAS) was investigated to enhance the yield and improve the functionality of soluble dietary fiber (SDF). When orange peel was pretreated by SE at 0.8 MPa for 7 min, combined with 0.8% SAS, the content of SDF was increased from 8.04% to 33.74% in comparison to the control and SDF prepared with SE–SAS showed the high water solubility, water-holding capacity, oil-holding capacity, swelling capacity, emulsifying activity, emulsion stability and foam stability. SDF from orange peel treated by SE–SAS exhibited significantly ($p < 0.05$) higher binding capacity for three toxic cations (Pb, As and Cu) and smaller molecular weight ($M_w = 174$ kDa). Furthermore, differential scanning calorimetry (DSC) measurement showed that SDF from orange peel treated by SE–SAS had a higher peak temperature (170.7 ± 0.4 °C) than that of the untreated sample (163.4 ± 0.3 °C). Scanning electron micrograph (SEM) images demonstrated that the surface of SDF from orange peel treated by SE–SAS was rough and collapsed. It can be concluded that SDF from orange peel treated by SE–SAS has the higher potential to be applied as a functional ingredient in food products.

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1. Introduction

Worldwide industrial citrus byproducts are estimated to be more than 15×10^6 tons, as the amount of residues accounted for 50% of the whole fruit mass. During the processing of citrus fruits, peels were the primary byproduct, and a potential burden to environment without further treatment (Ramful, Tarnus, Aruoma, Bourdon, & Bahorun, 2011).

Byproducts from citrus juice extraction had a potential use as a source of dietary fiber (DF). In recent years, DF received a great deal of attention from researchers, the food industry and consumers due to the health benefits that were associated with the consumption of fiber-rich products. Beneficial effects included lowering blood lipid and glucose levels, reducing risks from cardiovascular and colorectal cancer diseases, increasing satiety of host, and enhancing gastrointestinal immunity (Gunness & Gidley, 2010). Among those health benefits, soluble dietary fiber (SDF) was thought to play a major role. In addition, DF had hydrocolloidal properties that contributed technological implications in food manufacturing and final food products (Kosmala et al., 2013).

Therefore, DF was not only desirable for its nutritional value but also important in food formulation with its functional and physicochemical properties (Fabek, Messerschmidt, Freeport, & Goff, 2014). The main advantage of DF from orange peel, compared with other alternative sources, such as cereals, was its higher proportion of soluble dietary fiber. This was very important for the development of fiber-containing products, considering that the requirement for DF intake should be balanced, i.e. the water-soluble fraction should represent between 30% and 50% of the total dietary fiber (Jing & Chi, 2013).

Steam explosion (SE) is an innovative processing technology, which is used for the pretreatment of lignocellulosic materials. This technology is based on exposing the samples to high-temperature pressurized steam for a short period and forcing the steam into the tissues and cell of samples, followed by explosive decompression completed in millisecond (Yu, Zhang, Yu, Xua, & Song, 2012). During the explosion, most of the steam and hot liquid water in the samples quickly expands and breaks free of the structure. Compared with other pretreatment methods, the advantages of SE included significantly lower energy consumption, lower capital investment and less hazardous process chemicals (Alvira, Tomás-Pejó, Ballesteros, & Negro, 2010). In the previous publication, the results showed that SE could significantly improve the extraction yield and physicochemical properties of cellulose from

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Lespedeza stalks (*Lespedeza crytobotrya*) (Wang, Jiang, Xu, & Sun, 2009). Chen and Chen (2011) had reported that SE extraction was an effective method for extracting and conversing flavonoids from sumac fruits. However, the application of SE in SDF extraction from orange peel has not yet been reported.

Dilute sulfuric-acid soaking (SAS) pretreatment is an effective and inexpensive way of hydrolyzing hemicellulose, reducing cellulose crystallinity, and increasing surface area and pore volume of the substrate (Fang, Deng, & Zhang, 2011). A few available researches demonstrated that the dilute-acid soaking treatment influenced the structure and function of soy protein. The improvement of functional properties of acid-modified soy protein was due to its decreased molecular size and increase in surface hydrophobicity induced by deamidation (Zhang, Yang, Zhao, Hua, & Zhang, 2013; Zhang, Zhao, et al., 2013).

Based on the aforementioned viewpoint, a combination of SE and SAS pretreatment can simultaneously influence the structural and functional properties of SDF from orange peel. The objective of this study was to optimize the parameters of SE–SAS in which the maximum SDF extraction yield was obtained. In addition, the physicochemical properties and microstructures of SDF were also evaluated.

2. Materials and methods

2.1. Materials

The oranges [*Citrus sinensis* (L.) Osbeck] were purchased from the commercial orchards located in China. The one batch of oranges was used for independent replicates. The orange fruits were separated into edible and inedible portions (peel) and the peels were dried in an air-oven at 50 °C for 24 h. The moisture content of the dried peel samples was $5.83 \pm 0.73\%$. The dried samples were then finely ground to pass through a 10-mesh screen and stored in polyethylene bags until used. All other reagents and chemicals were of analytical grade.

2.2. Acid soaking treatment

The ground orange peel was treated with dilute sulfuric acid solution (orange peel/solution ratio = 1:2) in a stainless steel container immersed in a temperature-controlled water bath maintained at 80 °C for 2 min. The container was equipped with a stirrer to ensure proper mixing of orange peel with the acid solution. The sulfuric acid concentrations were examined in a range between 0% and 1.0% (w/v). Soaking in deionized water was referred to as a non-acid treatment condition. Following the acid and non-acid treatment, the slurry was filtrated through the four-fold gauze to separate excess sulfuric acid (Zhang, Yang, et al., 2013; Zhang, Zhao, et al., 2013). After filtration, the moisture content of soaked orange peel meal was about 65%.

2.3. Steam explosion treatment

About 500 g of acid soaked and non-acid soaked orange peels were loaded into a 5 L reactor of the SE system. It adopts a structure in catapult explosion mode that is principally composed of a cylinder and piston. The force of the piston drive system, which is composed of a linear actuator and a solenoid valve, comes from compressed air (Yu et al., 2012). The reactor is equipped with a high-pressure autoclave with gas inlet. When the saturated steam was quickly allowed to enter the reactor and steam pressure was maintained for given time, the steam inlet was shut off and the piston device was triggered. The explosion was completed in about

millisecond. The samples were carefully recovered and sealed in plastic bags.

2.4. Description of experimental design

In the acid soaking concentrations (0–1.0%) experiments, acid soaked and non-acid soaked orange peels were treated at 0.6 MPa for 9 min, and the SDF extraction yield of orange peel was examined. In the optimization of steam pressure, the acid-soaked orange peel was then treated at 0.4 MPa, 0.6 MPa, 0.8 MPa, 1.0 MPa and 1.2 MPa for 7 min, and the SDF extraction yield of SE-treated orange peel was examined. In the optimization of residence time of SE, the acid-soaked orange peel was treated at 0.8 MPa for different residence times (3 min, 5 min, 7 min, 9 min and 11 min), and the SDF extraction yield of orange peel was examined. All experiments were carried out using the individual amount (500 g) of dried orange peels.

All experimental set points were carried out in triplicate. For each experiment, the materials recovered from receiver were carefully mixed together and constituted a unique batch (Zhang, Yang, et al., 2013; Zhang, Zhao, et al., 2013).

2.5. Preparation of SDF from orange peel

To determine SDF content in orange peel, SDF was extracted according to the AOAC method 985.29, an enzymatic–gravimetric procedure (AOAC, 2001) with slight modifications. The pretreatment orange peel, dispersed in 4 times volume of deionized water, adjusted pH to 6.0 with 0.1 mol/L NaOH, added 0.1% (w/w) heat-stable α -amylase, hydrolyzed at 95 °C under constant stirring of 120 rpm for 30 min, and 120 °C in oil bath for 5 min to inactivate. After the temperature of the hydrolysate was decreased to 60 °C, 0.016% (w/w) neutral protease was added and went further hydrolysis for 30 min under constant stirring of 120 rpm. At the end, the enzymatic hydrolysis reaction was quenched at 95 °C for 5 min and the hydrolysate was centrifuged at 4200 rpm for 20 min, the supernatant and sediment were collected, respectively. The supernatant was condensed to one-tenth in vacuum rotary evaporation system. Afterwards, the concentrated supernatant was mixed with 95% (v/v) ethanol at 4 °C for 12 h and subjected to centrifugation at 4200 rpm for 15 min. The precipitated flocculate was dried in a laboratory oven (DHG-9140A, Yiheng, Shanghai, China) at 60 °C for 48 h. The dried flocculate was milled (Thomas Scientific, Swedesboro, NJ, USA) and passed through a 60-mesh sieve and stored at 4 °C, the powder obtained was SDF.

2.6. Determination of soluble dietary fiber

The contents of soluble dietary fiber (SDF) in untreated and treated samples were measured according to the AOAC (AACC Method 32–45, 2010).

2.7. Physicochemical properties of SDF

2.7.1. Solubility, water- and oil-holding capacities and swelling capacity

The water solubility (WS) was determined in triplicate according to the method described by Zhang, Liang, Pei, Gao, and Zhang (2009) with minor modification. Dry sample (1.0 g) was gently mixed with 50 mL of distilled water in a beaker. Subsequently, the mixture was stirred at 90 °C for 30 min in thermostat water bath followed by centrifugation at 4200 rpm for 10 min. The supernatant was collected, freeze-dried and weighted. The WS was calculated as follows:

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