Biocatalysis and Agricultural Biotechnology 4 (2015) 250–258

Contents lists available at ScienceDirect

Biocatalysis and Agricultural Biotechnology

journal homepage: www.elsevier.com/locate/bab

Physicochemical characterization of five types of citrus dietary fibers

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ARTICLE INFO

Article history:
Received 27 September 2014
Received in revised form
9 February 2015
Accepted 21 February 2015
Available online 28 February 2015

Keywords:
Citrus fruit peels
Dietary fiber
Chemical composition
Physicochemical properties
In vitro binding capacity

ABSTRACT

The chemical composition, physicochemical and *in vitro* biochemical properties of dietary fibers (DFs) from five types of citrus fruit peels (orange, grapefruit, lemon, gonggan and ponkan) were investigated. The contents of total, soluble and insoluble dietary fibers in citrus fruit peels had no significant difference ($p > 0.05$) and the binding capacities of soluble dietary fibers (SDFs) of orange, lemon, gonggan and ponkan for sodium cholate and cholesterol were significantly ($p < 0.05$) lower than those of grapefruit SDF. The more sponge-like porous network structure of grapefruit SDF could lead to its higher binding capacities. All the SDFs of citrus fruit peels exhibited a near-Newtonian fluid behavior, and grapefruit SDF solution had higher viscosity than that of others. The main monosaccharide of orange SDF was glucose (33.87%), while arabinose was the main monosaccharide in the SDFs from grapefruit, lemon, gonggan and ponkan (38.67–44.83%). To the water-holding, oil-holding and swelling capacities of insoluble dietary fiber (IDF), the IDF from grapefruit and orange were the highest and lowest one respectively.

1. Introduction

Citrus is one of the most important fruit crops in the world with an annual production exceeding 122.5 million tons and one-third of the crop is processed (Jiang et al., 2014). Oranges, lemons, grapefruits and mandarins account for nearly 98% of the entire industrialized crops, and oranges are the most relevant crop with approximately 82% of the total cultured for industry (Marín et al., 2007).

Worldwide industrial citrus byproducts are estimated to be more than 1.5 million tons, as the amount of residues account for 50% of the whole fruit mass (Laufenberg et al., 2003; Montgomery, 2004). During the processing of citrus fruits, peels are the primary byproduct, and a potential burden to the environment without further treatment (Wang et al., 2008; Ramful et al., 2011). The byproducts generated by the citrus juice industries are sources of dietary fiber (DF) but are commonly used in animal feed or fertilizer. In recent year, DF attracts a great deal of attention from researchers, the food industry, and consumers, due to its health benefits (Tanaka et al., 2012).

The functional properties of DF include the bulk volume, the hydration, hydrocolloidal and rheological properties, which contribute to application in food formula design and food manufacturing (Bodner and Sieg, 2009; Gómez-Ordóñez et al., 2010). DF mainly consists of soluble and insoluble fiber fractions which show different physiological effects on human health. Beneficial effects of soluble dietary fiber (SDF) include lowering blood lipid and glucose levels (Benavente-Garcia and Castillo, 2008), reducing risks from cardiovascular and colorectal cancer diseases (González-Molina et al., 2010; Adibelli et al., 2009), increasing satiety of host (Bengtsson et al., 2011; Brownlee, 2011), and enhancing gastrointestinal immunity (Anderson et al., 2009; Gunness and Gidley, 2010). While cellulose, hemicellulose and lignin were the main components of the insoluble fraction of dietary fiber which prevents or relieves the constipation due to the absorption of water from the digestive tract. SDF could lower blood cholesterol and regulate blood glucose level. Several studies indicated that SDF was more important than IDF in many health aspects (Galisteo et al., 2008; Kethireddipalli et al., 2002). Therefore, DF was not only desirable for their nutritional value but also potential in food formulation with its functional and physicochemical properties (Fabek et al., 2014). Compared with other alternative sources, such as cereals, DF from citrus fruit peels is in rich of higher proportion of SDF, which is important and exhibit several functional properties, such as glucose retardation index, water-holding capacity (WHC) and oil-holding capacity (OHC). These properties are more useful for understanding the chemical composition and physiological effects of DF (Jing and Chi, 2013; Kendall, et al., 2010).

Chemical and physical properties of citrus fibers have been widely studied (Lundberg, et al., 2014; Sudha et al., 2007). Some researchers primarily analyzed the DF content of citrus fruits (Gorinstein, et al., 2001), while others mainly focused on DF components (de Almeida Costa et al., 2006). In addition, functional properties of DF were extensively evaluated by Fernández-López et al. (2004). Nevertheless,

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http://dx.doi.org/10.1016/j.bcab.2015.02.003
1878-8181/

the composition and functional properties of DFs from citrus fruit peels have not yet been compared for further application in food industry.

The objective of this study is to characterize the physicochemical properties of five types of citrus dietary fibers based on their contents of TDF, SDF and IDF, the monosaccharide composition of SDF, *in vitro* binding capacities to cholesterol and sodium cholate of SDF, rheological properties and microstructure of SDF as well as hydration capacities (water-holding capacity, oil-holding capacity and swelling capacity) of IDFs.

2. Materials and methods

2.1. Plant materials and sample preparation

The orange [*Citrus sinensis* (L.) Osbeck], grapefruit [*Citrus grandisi* Marc.], lemon [*Citrus limon* (L.) Burm.f.], gonggan (*Citrus reticulate* × *Citrus sinensis*) and ponkan [*Citrus reticulate* (L.) Blanco] were purchased from the commercial orchards located in China. The citrus 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 from (5.83 ± 0.73) g/kg to (7.02 ± 0.83) g/kg. The dried samples were grounded into powder with a particle diameter below 0.38 mm (40 mesh). Samples were kept in a desiccator at ambient temperature (ca. 21 °C) until used.

2.2. Separation of SDF and IDF from citrus fruit peels

To determine SDF and IDF content in citrus fruit peels, the extraction was carried out following an enzymatic-gravimetric procedure (AOAC 985.29, 2001) with minor modifications. In brief, peel sample was thoroughly dispersed in 4 times volume of deionized water, and the pH of peel dispersion was adjusted to 6.0 with 0.1 mol/L NaOH, 0.1% (w/w) heat-stable α-amylase was added, and hydrolyzed at 95 °C with constant stirring at 120 rpm for 30 min. After the temperature of the hydrolysate was cooled down to 60 °C, 0.016% (w/w) neutral protease was added and further hydrolyzed for 30 min with constant stirring at 120 rpm. At the end, the enzymatic hydrolysis reaction was quenched at 95 °C for 5 min and the hydrolysate was centrifuged at 3800 × *g* for 20 min after cooled down to room temperature, the supernatant and sediment were collected. The supernatant was condensed to one-tenth with a vacuum rotary evaporator (Model R203B, Shanghai Senco Technology Co. Ltd., Shanghai, China). Afterwards, the concentrated supernatant was mixed with 95% (v/v) ethanol at 4 °C for 12 h and then subjected to centrifugation at 3800 × *g* for 15 min. The precipitated flocculate was dried at 60 °C for 48 h. The dried flocculate was SDF, which was milled and passed through a 60-mesh sieve and stored at 4 °C. The sediment (IDF) was washed for three times with 70 °C water, dried at 60 °C for 48 h and milled into powder and passed through a 60-mesh sieve and stored at 4 °C. TDF was the sum of IDF and SDF.

2.3. Analysis of SDF and IDF

The SDF was purified by dialysis in order to prevent error caused by precipitating dietary fiber with ethanol, the supernatant containing SDF was sequentially dialyzed with a dialysis tube (12,000–14,000 molecular weight cutoff, Spectrum Laboratories, Inc., Rancho Dominguez, CA., USA) in 1 L of deionized water in the refrigerator. Deionized water was changed at 4, 16, 28, 30, and 36 h, and the dialysis was finished at 48 h of which the conductivity of dialysate was around 2–6 μS/cm. The dialyzed SDF preparation was freeze-dried and then acid hydrolyzed in 6% sulfuric acid at 121 °C for 1 h (Bravo and Saura-Calixto, 1998). Uronic acids (UAs) in the resulting hydrolysate were quantified by colorimetry method as described by Anthon and Barret (2008). Neutral sugars (NS) were determined by HPLC following neutralization by addition of calcium carbonate. D-glucose, D-xylose, D-galactose, L-arabinose, D-fructose, L-rhamnose and D-mannose were employed as standards. HPLC was done using a Shimadzu 20A series instrument equipped with a Shimadzu-LTII evaporative light scattering detector (ELSD, Shimadzu, Columbia, MD, USA). The nitrogen pressure of the ELSD was maintained at 50.6–52.0 psi and column temperature at 85 °C. Deionized water was used as the mobile phase with a flow rate of 0.6 mL/min and injection volume of 20 μL (Sluiter et al., 2008). The sum of NS and UA was taken as the amount of SDF in citrus fruit peels.

The dry residue (IDF) was hydrolyzed in a two-stage manner. The first stage involves the addition of 3 mL 72% (v/v) sulfuric acid to the residue, with stirring, and subsequent incubation at 30 °C for 1 h. The second stage consists of diluting the first-stage hydrolysate to 2.5% sulfuric acid, followed by incubation of the resulting suspension at 121 °C for 1 h. The hydrolyzed mixture is then filtrated (fritted crucible; Pyrex 30 mL M, Corning, Inc., USA). The filtrate is used for quantifying UA and NS as described above. The crucible containing the residue is used for the gravimetric determination of Klason lignin (KL) as described by Sluiter et al. (2008). KL was the weight of residue after drying at 105 °C for 16 h, ashing for 5 h at 525 °C, and subtracting resistant protein (RP). RP in the citrus fruits peels powder, defined as the protein after protease treatment and acid hydrolysis in this study, was determined by the micro-Kjedahl method using a nitrogen-to-protein conversion factor of 6.25 (AOAC 960.52, 1995). IDF was the total content of NS, UA and KL.

2.4. Determination of SDF and IDF purity

The purity of SDF and IDF was determined according to the method described by Deng et al. (2011) and it was calculated with the following equation:

$$\text{Purity (SDF or IDF, \%)} = \frac{W_1}{W_2} \times 100$$

where W_1 is equal to the contents of NS and UA in SDF or NS, UA and KL in IDF (g); W_2 is the content of SDF or IDF extracted from citrus fruit peels (g).

2.5. Determination of monosaccharide composition

Monosaccharide composition of SDF was measured according to the method described by Wu et al. (2014) with some modifications. Briefly, 0.15 g SDF was mixed with 7 mL sulfuric acid solution (6 mol/L) in a stoppered test tube and stayed at ambient temperature for 1 h for dissolution. The SDF solution was diluted with 5 times volume of deionized water and hydrolyzed at 100 °C for 1 h. Afterwards the pH of hydrolysate was adjusted to 7.0 with NaOH (2 mol/L) and then the volume of hydrolysate was made up to 50 mL with deionized water.

The chemical composition of SDF was determined by high performance anion exchange chromatography coupled with a pulse amperometric detector (HPAEC-PAD) on a Dionex ICS-3000 chromatographic system (Dionex Co., Sunnyvale, USA). Hydrolysates were separated on a Dionex CarboPac™ PA20 column (150 mm × 3 mm, i.d., 5 μm) with a constant elution at a flow rate of 0.4 mL/min. 2 mmol/L of NaOH was the mobile phase for detecting galactose, glucose, xylose, mannose, and fructose. The elution was changed to 10 mmol/L of NaOH when rhamnose and arabinose were analyzed.