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Extraction of pectin from the peels of pomelo by high-speed shearing homogenization and its characteristics

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ABSTRACT

High-speed shearing homogenization extraction (HSHE) was applied to extract pectin from the peels of pomelo using a high-speed shearing extractor for the first time and the extraction conditions were optimized using response surface methodology with Box-Behnken design. The optimal extraction conditions were determined: pH of extraction solvent 1.24, extraction voltage 156 V, extraction time 240 s, with the predicted extraction yield was 209 g/kg, which was very consistent with the experimental value (209 ± 2 g/kg) and significantly higher than traditional thermal extraction method (175 ± 6 g/kg at 85 °C for 80 min). Furthermore, pectin extracted by HSHE has a higher viscosity, intrinsic viscosity and viscosity-average molecular weight than traditional thermal extracted pectins. The results demonstrated that HSHE has a great potential for the extraction of pectin with higher viscosity efficiently.

1. Introduction

Pomelo (*Citrus grandis* Osbeck, *Citrus* genus, Rutaceae family), one of the most well-known and staple commercial fruits native to Southeast Asia, is widely cultivated and consumed all over the world currently due to its distinct flavor and unique taste (Foo & Hameed, 2011; Sârbu et al., 2012). During the processing of Citrus fruits for juice, marmalade or canning productions, peels represent about 50%–65% of the total weight of the fruits and remain as the primary byproduct. Massive amounts of the pomelo peels (as waste products) are disposed as industrial wastes, causing severe problems in the community (Sârbu et al., 2012). Therefore, there have been great interests in searching for a proper disposal methods of pomelo peels. Pectin, as a family of complex heteropolysaccharides, has been widely used in the food, pharmaceutical and cosmetic industries as gelling, thickening or stabilizing agent, etc. (Thakur, Singh, & Handa, 1997). Its structure is based on 1, 4-linked α-D-galacturonic acid, interrupted by L-rhamnose residues with side-chains of neutral sugars (mainly D-galactose and L-arabinose) (Mohnen, 2008; Ridley, O'Neill, & Mohnen, 2001). At present, traditional thermal extraction (TTE) was the most common method for pectin extraction by using hot acid with the pH and temperatures generally in the range of 1.3–3, 60–100 °C, respectively (Levigne, Ralet, & Thibault, 2002; Liu, Shi, & Langrish, 2006; Yeoh, Shi, & Langrish, 2008). Several kinds of new technologies, such as ultrasound, high hydrostatic pressure, microwave heating at ambient and moderate pressures, as well as enzyme-assisted extraction are currently investigated in the extraction of pectin from various natural resources to give the increased extraction yield, higher quality, less time or energy consumption than the traditional thermal extraction (TTE) (Fishman, Chau, Cooke, & Hotchkiss, 2008; Fishman, Chau, Hoagland, & Hotchkiss, 2006; Fishman, Chau, Hoagland, & Ayyad, 2000; Guo et al., 2012; Wang et al., 2007, 2015; Wikiera, Mika, & Grabacka, 2015). High-speed shearing homogenization extraction (HSHE) is an emerging novel extraction technique originally used for the extraction of biologically active ingredients from various natural resources or Chinese

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herbs with lower extraction temperature, higher efficiency, less time and energy consumption, etc. (Lee et al., 2011; Liu, Liu, Rong, & Rong, 2011; Liu, Wang, Liu, Wang & Liu, 2010; Liu, Zheng, Wang, Zou, & Li, 2011; Zhang et al., 2008; Zhu, Lin, Chen, Xie, & Wang, 2011; Zu, Zhao, Li, Shi, & Jiao, 2005). The principle of the HSHE is based on a rotating inner cutter driven by a high-speed motor, which produces a powerful shear force between the inner and external cutters to disrupt and mix the samples. Meanwhile, the high rotate speed results in the pressure differences between the inside and outside the cavities and causing the occurrence of the mass transfer between the samples and solvents. With the combination of high-intensity shearing force, fierce collision, pressure differential relief and other forces, the extraction process could complete in a few minutes, even dozens of seconds by HSHE (Liu et al., 2010; Liu et al., 2011; Zhang et al., 2008; Zhu et al., 2011). Most of the studies currently are focused on the application of HSHE for the extraction of small molecule ingredients with biological activity from various natural resources or Chinese medicinal herbs. A recent study using an ultraturrax equipment, similar with HSHE, for the extraction of cell wall ingredient from 26 food waste streams to study their pectin content and composition (Müller-Maatsch et al., 2016). However, there is no investigation reported on the difference of the pectin extracted by HSHE with other extraction technologies.

In the present study, HSHE was used to explore its potential utilization in the extraction of pectin from the peels of pomelo and its effects on physicochemical characteristics of pectin were also investigated. The optimum extraction conditions were well established by using response surface methodology (RSM) with Box-Behnken design (BBD). In addition, the comparative analysis on the extraction yield and physicochemical characteristics of the pectin extracted by HSHE, TTE, as well as two types of commercialized pectin, were also investigated.

2. Material and methods

2.1. Material and chemical reagents

Honey pomelo (*Citrus grandis* Osbeck) cultivated in Guanxi, Fujian province, was purchased from a local market in Beijing. The collected peels of pomelo were first cut into pieces and steamed at 100 °C for 2 min to inactivate the enzymes, and then dried by a vacuum freeze dryer (LGJ-25C, Four-ring Science Instrument Co., Ltd, Beijing, China) with the shelf temperature at 18 °C until the water content was reduced to ca. 80 g/kg. Then the sample was milled by an electric grinder (HY-04A, Beijing Huanya Tianyuan Machine Technology Co., Ltd, Beijing, China) and filtrated by a filter sieve (ca. 250 μm). Finally, the sample was vacuum-packed and stored at 4 °C for subsequent extraction. Standard D-galacturonic acid (Sigma-Aldrich, St. Louis, USA) and *m*-hydroxybiphenyl (Sigma-Aldrich, St. Louis, USA) were purchased from Beijing Biodee Biotechnology Co., Ltd (Beijing, China). Two types of commercial pectin SP9135 (SP) (Sigma-Aldrich, St. Louis, USA) and commercial pectin AP102 (AP) (Andre Pectin Co. Ltd, Yantai, China) extracted from citrus peel and apple pomace, respectively, using traditional thermal extraction were also purchased from Beijing Biodee Biotechnology Co., Ltd (Beijing, China). Deionized water with different contents of hydrochloric acid was selected as extraction solvent according to our preliminary study.

All chemical reagents, including ethanol, hydrochloric acid, sulphuric acid, sodium tetraborate, coomassie brilliant blue, bovine serum albumin (BSA), sodium hydroxide, etc, used in the experiments were analytical grade and purchased from the Lanyi reagent company (Beijing, China).

2.2. Apparatus

The HSHE was carried out by a JHBE-50 system (Jinnai Sci-tech Development Ltd, Zhengzhou, China) consisting of the rotating cutter, high-speed motor, lifting system, control system and sample container (Fig. 1). The rotate-speed of the inner cutter could be regulated in the range from 0 to 11000 rotation/min linearly by controlling the extraction voltage from 0 to 220 V.

2.3. Experimental design

Based on the results of single factor experiment, response surface methodology with Box-Behnken design was applied to optimize the working conditions of HSHE for the extraction of pectin from the peels of pomelo. The range of independent various X_1 (pH of extraction solvent), X_2 (extraction voltage) and X_3 (extraction time) and their levels are presented in Table 1. The relationships between the responses and variables were approximated and fitted by the following second order polynomial function (Li, Jia, Wei, & Liu, 2012):

$$Y = \beta_0 + \sum_{i=1}^{\alpha} \beta_i X_i + \sum_{i=1}^{\alpha} \beta_{ii} X_i^2 + \sum_{i,j=1(i\neq 1)}^{\alpha} \beta_{ij} X_i X_j \quad (1)$$

where Y is the calculated response function and X_i is the corresponding actual value of variable; β_0 is the estimated regression coefficient of the fitted response at the center of the design; β_i is the regression coefficient for liner terms; β_{ij} is interaction effects; and β_{ii} is quadratic effects. All calculations and graphics were performed by using the Design-Expert 8.0 (Stat-Easy Inc., Minneapolis, USA).

2.4. Extraction and purification of pectin

2.4.1. HSHE procedure

Six grams of the sample mixed with 300 mL of the extraction solvent (pH from 0.8 to 1.6) was placed in the sample container and pre-heated at 85 °C for 5 min by a constant temperature water-bath (HH-601, Ronghua experimental apparatus Co., Ltd, Jintan, China), and then the extraction was carried out by JHBE-50 under various extraction voltage (120–180 V) and extraction time (2–4 min) in sequence as presented in Table 2. After the extraction process, the mixture was centrifuged by high speed freezing centrifuge using 12000*g* and filtered by vacuum filtration using medium speed filter paper for subsequent purification immediately.

2.4.2. TTE procedure

Pectin extracted by traditional thermal extraction (TTE) was according to the method described by Kratchanova et al. with slight modification (Kratchanova, Panchev, Pavlova, & Shtereva, 1994). The sample (6 g) was mixed with 300 mL of extraction solvent (pH = 1.24) and then heated to 85 °C for 80 min with continuous stirring using a constant temperature bath oscillator (DSHZ-300A, Peiying experimental apparatus Co., Ltd, Taicang, China). After the extraction process, the mixture was centrifuged by high speed freezing centrifuge using 12000*g* and filtered by vacuum filtration using medium speed filter paper for subsequent purification immediately.

2.4.3. Pectin purification procedure

One volume of crude pectin filtrate extracted by the two different extraction methods was precipitated using double volumes of absolute ethanol, respectively, and kept overnight without stirring at 4 °C. The precipitated pectin was filtered through a filter cloth (37 μm) and then washed three times by using pure alcohol to