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Extraction, structure, and emulsifying properties of pectin from potato pulp

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ABSTRACT

Effects of HCl, H_2SO_4, HNO_3, citric acid, and acetic acid on the yield, structure, and emulsifying properties of potato pectins were investigated. Results showed that the highest yield (14.34%) was obtained using citric acid, followed by HNO_3 (9.83%), HCl (9.72%), H_2SO_4 (8.38%), and acetic acid (4.08%). The degrees of methylation (37.45%) and acetylation (15.38%), protein content (6.97%), and molecular weight (3.207×10^5 g/mol) were the highest for pectin extracted using acetic acid, and (galactose + arabinose)/rhamnose was 33.34, indicating that it had a highly branched rhamnogalacturonan I domain. Fourier transform infrared spectroscopy showed a specific absorbance peak at 1064 cm^{-1}, which corresponds to the acetyl groups in potato pectins. SEM showed that all potato pectins are morphologically different. The emulsifying activity (EA, 44.97%–47.71%) and emulsion stability (ES, 36.54%–46.00%) of the pectins were influenced by acid types, and were higher than those of commercial citrus and apple pectin.

1. Introduction

China is the largest producer of potato in the world. In 2014, the yield of fresh potato in China reached 96 million tonnes (FAO, 2014). Potato is commonly used in starch processing, which results in a large quantity of waste pulp. In China, approximately 4.5–5.0 tonnes of fresh potato pulp are generated for every tonne of starch produced. However, while a small amount of the potato pulp byproduct is used as low-value animal feed, most of it is disposed, which means that it is a major contributor to environmental pollution. Previous studies have indicated that potato pulp consists of starch (37%), pectin (17%), cellulose (17%), hemicellulose (14%), and protein (4%) (dry basis) (Mayer, 1998), and potato pulp is rich in pectin which can be used as a good raw material for pectin extraction, however, there is little information about potato pectin.

Pectin is a complex acidic macromolecular polysaccharide found in primary cell walls and the middle lamella. It is generally composed of homogalacturonan (HG), rhamnogalacturonan I (RG-I), and rhamnogalacturonan II (RG-II). HG is a linear polymer that consists of α-(1, 4)-linked galacturonic acids (GalA) and is a major domain of pectin (Caffall & Mohnen, 2009). The GalA residues in the HG backbone can be methyl esterified at C-6 and can also be acetylated at O-2 and/or O-3. According to the degree of methylation (DM), pectin can be classified as high methoxyl pectin (HMP) (DM > 50%) or low methoxyl pectin (LMP) (DM < 50%) (Yapo, 2011). The RG-I region is composed of 100 repeating disaccharide units ([→4)-α-D-Gal*p*A-(1 → 2)-α-L-Rhap-(1→]), and 20%–80% of the rhamnosyl residues in the backbone may be substituted with neutral sugar side chains (galactan, arabinan, and arabinogalactan) at O-4; further, GalA can also be acetylated at C-2 and/or C-3 (Albersheim, Darvill, Oneill, Schols, & Voragen, 1996; Ridley, Oneill, & Mohnen, 2001). The RG-I domain contains at least seven α-(1, 4)-linked GalA in the backbone, and the side chains are mainly composed of sugars such as apiose, rhamnose, xylose, galactose, and fucose (Pellerin & Oneill, 1998). The proportions of HG, RG-I, and

Abbreviations: HG, homogalacturonan; RG-I, rhamnogalacturonan I; RG-II, rhamnogalacturonan II; GalA, galacturonic acids; DM, degree of methylation; HMP, high methoxyl pectin; LMP, low methoxyl pectin; DA, degree of acetylation; HPP, pectin extracted by HCl; SPP, pectin extracted by H_2SO_4; NPP, pectin extracted by HNO_3; CPP, pectin extracted by citric acid; APP, pectin extracted by acetic acid; Mw, weight-average molecular weight; MF, mass fraction; FTIR, Fourier transform infrared spectroscopy; SEM, scanning electron microscopy; EA, emulsifying activity; ES, emulsion stability

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RG-I are related to the pectin source; for example, the proportions of the HG and RG-I regions in commercial citrus and apple pectins are 65% and 20%–35%, respectively, whereas potato pectin has a high proportion of the RG-I region (75%) and a relatively low proportion of the HG region (20%). Furthermore, β-(1, 4)-linked galactan side chains are abundant in the RG-I domain of potato pectin. The degree of acetylation (DA) of potato pectin is about 14%, which is higher than that of commercial citrus pectin (2%) and apple pectin (4%) (Mohnen, 2008; Sorensen et al., 2000; Vincken et al., 2000; Voragen, Schols, & Pilnik, 1986). Commercial citrus and apple pectins are often used as gelling agents in the food processing industry because of their high DM, molecular weight, and high proportion of the HG region. In comparison, potato pectin is richer in acetyl groups and neutral sugar side chains but HG domain is shorter than commercial citrus and apple pectins, and thus, potato pectin does not have good gelling ability. On the other hand, previous studies have showed that acetyl groups and neutral sugar side chains have positive effects on the emulsifying properties of pectin (Funami et al., 2011; Leroux, Langendorff, Schick, Vaishnav, & Mazoyer, 2003). Therefore, the unique structure of potato pectin may allow it to be used as an emulsifier. However, little attention has been paid to the properties of potato pectin which limits its application in the food industry.

The alkaline, enzymatic, and acid methods are the most common ways to extract pectin from sources such as potato, apple, citrus fruits, sugar beet, and cocoa husk. The alkaline extraction method can retain the neutral sugar side chains in pectin; however, the methyl ester and acetyl groups of pectin are hydrolyzed by the β-elimination reaction (Rombouts & Thibault, 1986). The enzymatic extraction method can decrease the emission of waste acid or alkali solutions, and results in pectins with a higher molecular weight and degree of esterification (DE) than the acid method, but it takes longer time than acid method (Wikiera, Mika, Starzynskajaniszewska, & Stodolak, 2016). The acid extraction method is often used to extract pectin in the food industry because of its convenient and easy operation. Several studies have indicated that the use of different acid extractants can have different effects on pectin yield, structure, and physicochemical properties (Chan & Choo, 2013; Ma et al., 2013). In recent years, some studies have mainly focused on the application of endo-polygalacturonase and KOH/NaOH to extract the RG-I domain from potato pulp (Khodaei & Karboune, 2016; Khodaei, Karboune, & Orsat, 2016). As far as we know, few studies have reported the effects of different types of acids on the yield, structure, and emulsifying properties of potato pectin.

In the present study, the effects of three mineral acids (HCl, H_2SO_4, and HNO_3) and two organic acids (citric acid and acetic acid) on the yield, structure, and emulsifying properties of potato pectins were clarified. The purpose of this study was to provide a theoretical basis for the industrial extraction of pectin from potato pulp, and to evaluate the potential of potato pectin as a natural emulsifier in the food industry.

2. Materials and methods

2.1. Materials and reagents

Potatoes (Kexin No. 1) were bought from Inner Mongolia Huaou Starch Industry Co., Ltd. (Inner Mongolia, China). The commercial citrus and apple pectins and the carbazole reagent (GC reagent) were purchased from Sigma–Aldrich (St. Louis, MO, USA). The rhamnose, arabinose, galactose, glucose, xylose, mannose, GalA, and glucuronic acid were HPLC grade, and were also purchased from Sigma–Aldrich. H_2SO_4 was guaranteed reagent, and was bought from Beijing Chemical Works (Beijing, China). The trifluoroacetic acid was chromatographic grade, and was purchased from Shanghai Aladdin Biochemical Technology Co., Ltd. (Shanghai, China). Deuterium oxide (99 atom % D) was purchased from Shanghai Macklin Biochemical Co., Ltd. (Shanghai, China). Sodium-3-(trimethylsilyl) propionate-2, 2, 3, 3-d4 (TSP) was bought from J & K Scientific Ltd. (Beijing, China), and the purity was > 98 atom % D. The corn oil was food grade, and was bought from a local supermarket. All other reagents were of analytical grade.

2.2. Preparation and extraction of pectins from potatoes

The preparation and extraction process of pectins from potatoes was shown in Fig. S1. Briefly, fresh potatoes were washed, peeled, cut into pieces, and mashed with water (1:1, w/v), afterwards, 0.05% (w/v) sodium bisulfite solution was added to prevent the potato pulp from browning. The wet pulp was dried at 60 °C overnight, and then ground and sieved with an 80-mesh screen. To remove the high content of residual starch, the above potato pulp powder was dissolved in distilled water (1:30, w/v) and enzymatically hydrolyzed using thermostable α-amylase (100 μL/g, enzyme activity was 120 KNU; Novozymes, Bagsværd, Denmark) at pH 6.25 ± 0.02, 95 °C for 30 min. The resulting slurries were cooled down to room temperature and centrifuged at 7000 × *g* for 10 min (GL-21M Refrigerated Centrifuge, Changsha Xiangyi Centrifuge Instrument Co., Ltd., Changsha, China). The precipitate was washed once with 85% (v/v) ethanol and recentrifuged, followed by drying at 50 °C overnight. The final dried potato pulp was ground and passed through a 40-mesh screen before pectin extraction.

The potato pectin was extracted according to the method of Wan (2008), with some modifications. Five grams of potato pulp was dissolved in distilled water (1:15, w/v) and adjusted to pH 2.04 ± 0.02 with HCl, H_2SO_4, HNO_3, citric acid, or acetic acid, respectively. The resulting solutions were heated at 90 °C for 60 min, and centrifuged at 7000 × *g* for 30 min. The supernatants were collected and treated with three volumes (v/v) of absolute ethanol at 4 °C overnight. The pectin precipitates were collected by recentrifugation, and then were washed twice with 70% (v/v), 80% (v/v), and 90% (v/v) ethanol, respectively. Finally, the pectin was dispersed in distilled water and freeze dried (SIM-FD5, the Siemon Company, Los Angeles, America).

Hereafter, the potato pectins extracted by HCl, H_2SO_4, HNO_3, citric acid, and acetic acid are referred to as HPP, SPP, NPP, CPP, and APP, respectively. The pectin yield (%, wet basis) was calculated as the ratio of the mass of dried pectin (Wp) to the mass of potato pulp after enzymatic treatment (We):

$$\text{Yield (\%, wet basis)} = \frac{Wp(g)}{We(g)} \times 100\% \quad (1)$$

2.3. Proximate composition of potato pulp

The protein content was determined by the Kjeldahl method, with a nitrogen conversion factor of 6.25 (AOAC 955.04). Crude fat (AOAC 920.39), total and soluble dietary fiber (AOAC 991.43), moisture (AOAC 925.09), ash (AOAC 942.05), and starch (AOAC 996.11) contents were analyzed using AOAC methods. The pectin content was determined according to the method described by Donaghy and Mckay (1994) with slight modifications.

2.4. Pectin characterization

2.4.1. Moisture and protein content of potato pectin

The moisture content of the pectin was analyzed according to AOAC 925.09. The protein content was determined by the modified Lowry method (Markwell, Haas, Bieber, & Tolbert, 1978; Peterson, 1977). Different concentrations of BSA solutions (10–100 g/mL) were used as standards. The concentration of the potato pectin solutions was 1 mg/mL, and the protein contents are presented on a dry basis.

2.4.2. Neutral sugars and galacturonic acid content of potato pectin

The individual neutral sugars of potato pectin were quantified by