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Properties and extraction of pectin-enriched materials from sugar beet pulp by ultrasonic-assisted treatment combined with subcritical water

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ABSTRACT

Pectin-enriched material (PEM) was extracted from sugar beet pulp using subcritical water combined with ultrasonic-assisted treatment. Optimisation of the reaction parameters for maximum extraction yield of PEM was carried out using response surface methodology. Optimum modification conditions were as follows: liquid/solid ratio 44.03, extraction temperature 120.72 °C, extraction time 30.49 min and extraction pressure 10.70 MPa. Under optimal conditions, the maximum yield of PEM was 24.63%. The composition of the PEM was determined. The data showed that the contents of galacturonic acid and arabinose were 59.12% and 21.66%, respectively. The flow behaviours were investigated by a rheometer. The effects of PEM on the pasting and thermal properties of maize starch were also conducted. The results showed that the addition of PEM increased pasting temperature and decreased other pasting parameters. Increasing PEM concentrations resulted in increased gelatinisation temperature and enthalpy.

1. Introduction

Sugar beet (*Beta vulgaris*) pulp (SBP), a by-product of the sugar-refining industry, is mainly used as a feed formulation, with few other commercial uses. Moreover, the drying process requires high energy and often presents an environmental problem. Therefore, numerous attempts have been made to utilise this waste as a biosorbent for the removal of heavy metal ions (Reddad et al., 2002), a source of pectins (Phatak, Chang, & Brown, 1988; Rombouts & Thibault, 1986), feruloylated oligosaccharides (Ralet, Faulds, Williamson, & Thibault, 1994), dietary fibres (Michel, Thibault, Barry, & de Baynast, 1988), and biofuels (Zheng et al., 2012). Specifically, a focus has been placed on pectin which exhibits superior emulsifying properties compared with commercial pectins (Ma et al., 2013).

Pectin, extracted from cell walls in most plants, is an anionic polysaccharide, which consists mostly of polymers rich in D-galacturonic acid (GalA) and often contains significant amounts of L-rhamnose (Rha), D-arabinose (Ara) and D-galactose (Gal) as well as 13 other monosaccharides (Vincken et al., 2003). In the industry, pectin is obtained from apple pomace and citrus peels chemically using conventional acid extraction, with strong acids such as sulphuric acid (Garna et al., 2007) and hydrochloric acid (Lv, Wang, Wang, Li, & Adhikari, 2013). This generally results in degradation of the arabinan side-chains and therefore in a loss of feruloyl groups which are the key factors in cross-linking pectins (Oosterveld, Beldman, Schols, & Voragen, 1996). Furthermore, such

Abbreviations: PEM, pectin-enriched material; SBP, sugar beet pulp; GalA, galacturonic acid; S/L, solid/liquid; DM, degree of methylation; DA, degree of acetylation; AMW, average molecular weight; SCW, subcritical water; Fuc, L-fucose; Rha, L-rhamnose; Ara, L-arabinose; Xyl, D-xylose; Gal, D-galactose; Man, D-mannose; GalA, D-galacturonic acid; FA, ferulic acid; RSM, response surface methodology; AIR, alcohol insoluble residue; DSC, differential scanning calorimetry; BD, back down value; SB, setback value; PV, peak viscosity; FV, final viscosity; HPV, hot paste viscosity; T_o, onset temperature; T_p, peak temperature; T_c, completion temperature; ΔH, enthalpy change; CCD, Box–Behnken central composite design.

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methods may cause environmental problems by producing hazardous contaminants. Therefore, new environmentally and human-friendly technologies are badly needed by the food industry.

Subcritical water (SCW), also known as hot compressed water, is an effective solvent for both polar and non-polar compounds and has been developed as an environmentally benign extraction technology for natural materials. Pectin with high yield (80%) is separated from the flavedo of *Citrus junos* with SCW, which demonstrates that extracting pectin using SCW is a feasible method (Ueno, Tanaka, Hosino, Sasaki, & Goto, 2008). However, Maillard reaction may happen between pectin and protein at high temperature for a long time. Another environmentally-friendly method, ultrasound, is reported to be capable of increasing extraction yield or reaction rate as well as reducing extraction time (Bagherian, Zokaee Ashtiani, Fouladitajar, & Mohtashamy, 2011). Ultrasonic-assisted extraction technology has been successfully applied in extraction of components such as polysaccharides (Bagherian et al., 2011), oils (Riera et al., 2004) and protein (Moulton & Wang, 1982).

Starch has important applications in the food industry. However, it has numerous negative properties such as synaeresis, retrogradation, and high viscosity during paste formation. This is often controlled by complex chemical modification or simple mixing with hydrocolloids (Sudhakar, Singhal, & Kulkarni, 1996). There are many reports on interactions between starches and hydrocolloids (Lee, Baek, Cha, Park, & Lim, 2002; Shi & Bemiller, 2002). However, no published information is available on the interaction between starch and sugar beet pulp pectin.

The aim of the present work was to optimise the preparation conditions of PEM and further to study the properties of PEM/starch mixtures. For this aim, sugar beet pulp was pre-treated with ultrasound. Subsequently, response surface methodology (RSM) was conducted to optimise the levels of the preparation variables (extraction temperature, extraction time, liquid/solid (L/S) ratio (w/w) and extraction pressure) for maximum yield of pectin-enriched material. We further investigated the chemical and rheological characteristics of the product and the effect of PEM on pasting and thermodynamic properties of normal maize starch. This work may provide an eco-friendly method to extract PEM, which could be expected to be well used as a stabilising agent in the food industry.

2. Materials and methods

2.1. Materials

Pressed SBP was supplied by Luyuan Sugar Industry Co., Ltd (Xinjiang, China). The pulp was oven-dried at 40 °C for 24 h to reach a moisture content of about 2%. The dried pulp was then ground using a grinder (model M20; IKA Werke GmbH & Co., Staufen, Germany) and the powder obtained was sieved (mesh No. 60). The ground powder was then stored in sealed polyethylene bags at room temperature for further experiments. The enzyme used, Viscozyme L9, was a commercial preparation obtained from Novo Nordisk (Copenhagen, Denmark). Because it was a multi-enzyme complex, purification was needed before use (Garna, Mabon, Wathelet, & Paquot, 2004). 2-Deoxy-D-glucose, *myo*-inositol, L-fucose (Fuc), L-rhamnose (Rha), L-arabinose (Ara), D-xylose (Xyl), D-galactose (Gal), D-mannose (Man), D-galacturonic acid (GalA), ferulic acid (FA), succinic acid, glacial acetic acid and methanol were purchased from Sigma–Aldrich Chemical Co. (St. Louis, MO). Trifluoroacetic acid was purchased from Aladdin Reagents Co., Ltd (Shanghai, China). All other chemicals were of analytical grade unless otherwise mentioned.

2.2. Ultrasonic pre-treatment

SBP was first washed with tap water to eliminate the bulk of sand and other inorganic materials. Then SBP was immersed in 96% (v/v) boiling ethanol for 20 min and washed with 70% ethanol to exclude low molecular weight sugars, amino acids, organic acids, inorganic salts, and also inactivate enzymes. The residue was dried at 40 °C to give an alcohol insoluble residue (AIR). Ultrasonication was carried out using the SCIENTZ Electronics ultrasound device (JY98-3, Ningbo, China) at sonic frequency 25 kHz and sonication time of 10 min. All experiments were performed on SBPs (12.19 g, dry basis) dispersed in 1000 mL of distilled water at the same ultrasonic intensity of 1 W/cm^2 without additional stirring.

2.3. Preparation of PEM by subcritical water

After ultrasonic pre-treatment, SBP slurry was transferred to the subcritical water extractor. The pressure in the extractor was controlled at a pre-set value. After SCW extraction at a defined temperature for a given period of time, the residue was separated by filtration through a Millipore 20-μm nylon filter. The pectin-enriched extract was cooled down in an ice bath for about 30 min and was precipitated by adding 4 volumes of isopropyl alcohol at room temperature for 60 min. Precipitated pectin was recovered by centrifugation at 9418*g* for 20 min. The precipitate was collected and dried by freeze drying.

2.4. Response surface methodology

Response surface methodology comprises a group of empirical techniques devoted to the evaluation of relations existing between a series of controlled experimental factors and measured responses, according to one or more selected criteria (Gao, Luo, Fu, Luo, & Peng, 2012). Four extraction variables considered for this research were extraction temperature, extraction time, liquid/solid (L/S) ratio (w/w) and extraction pressure, and the proper range and centre point value of four independent variables were confirmed on the basis of a single-factor experiment (Table 1). According to Box–Behnken central composite design, 24 experimental runs were carried out and the zero experiment was repeated five times. The Design Expert 8.0.6 software package (Stat-Ease, Inc., Minneapolis, MN) was used to establish the mathematical progress. In developing the regression equation, the test factors were coded according to the equation:

$$x_i = \frac{X_i - X_i^x}{\Delta X_i} \quad (1)$$

where x_i is the coded value of the ith independent variable, X_i is the real value of the ith independent variable, X_i^x is its value of in the centre point of the interval and ΔX_i is the step change value:

$$Y = b_0 + \sum_i b_i x_i + \sum_i \sum_j b_{ij} x_i x_j + \sum_i b_{ii} x_i^2 \quad (2)$$

where Y is the observed response, b_0 is an intercept, b_i is the first-order model coefficient, b_{ii} is the quadratic coefficient for the factor i, b_{ij} is the linear model coefficient for the interaction between factors i and j. The variable $x_i x_j$ represents the first-order interactions between x_i and x_j ($i < j$).

2.5. Determination of sugar composition

The alcohol precipitate was hydrolysed with combined chemical and enzymatic hydrolysis as described in the work of Garna et al. (2004). Precipitate (100 mg) was hydrolysed with 0.2 M