



## Rapid Communication

# Characterisation of the turbid particles in the extraction of sugar beet pectins



Xiao-ming Guo<sup>a</sup>, Si-ming Zhu<sup>a</sup>, Qiang Tang<sup>a</sup>, Shu-juan Yu<sup>a,b,c,\*</sup>

<sup>a</sup> College of Light Industry and Food Sciences, South China University of Technology, Guangzhou 510640, China

<sup>b</sup> State Key Laboratory of Pulp and Paper Engineering, Guangzhou 510640, China

<sup>c</sup> Guangdong Province Key Laboratory for Green Processing of Natural Products and Product Safety, Guangzhou 510640, China

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## ABSTRACT

This paper was aimed at characterising the insoluble substances (IS) responsible for the turbidity of the extract and impurity of the resulting pectins. Results showed that the IS caused a significant increase in the turbidity of the extract. The IS had bi-pyramidal shapes under the SEM observation. The observed XRD peaks for the IS were similar to those of calcium oxalate dihydrate (COD). Moreover, the IS consisted of mainly oxalate and calcium. Results from the present study indicate the IS is COD. The influence of the IS on the purity of pectin was also studied. The presence of the IS in the pectins indicated the IS can precipitate with pectins during the alcohol precipitation, thereby contaminating the resulting pectins.

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## 1. Introduction

Pectic saccharides are a complex family of heteropolysaccharides in the plant cell wall. Chemically, pectins are characterised by a linear backbone of  $\alpha$ -(1→4)-linked D-galacturonic acid units which are partly methyl esterified and acetylated (Buchholt, Christensen, Fallesen, Ralet, & Thibault, 2004; Thakur, Singh, Handa, & Rao, 1997). The galacturonic units of the backbone can be substituted with 2-O-linked- $\alpha$ -L-rhamnose residues which can bear neutral sugar side-chains with arabinose and galactose as the major sugar constituents.

Pectins can be extracted from various pectin-rich materials using the hot acid extraction, which is convenient for the industrial processing (May, 1990). This extraction is usually performed under the conditions ranging from pH 1.3–3, 60–100 °C, and 20–360 min (Levigne, Ralet, & Thibault, 2002; Yapo, 2009). This hot acid extraction is advantageous for obtaining a good yield, because it is capable of releasing pectic saccharides from the starting material (Yapo, 2009). But this conventional extraction has some limitations, which might provoke the undesirable co-extraction of non-pectic compounds (Garna et al., 2007).

Sugar beet pulp is the by-product of the beet sugar industry, and it has a complex chemical composition. Besides a considerable amount of polysaccharides including pectins, semicellulose and cellulose (Weibel, 1989), sugar beet pulp also contains some non-pectic compounds such as proteins, minerals and organic acids (Joy, 1964; Murray, Longland, Hastie, Moore-Colyer, & Dunnett, 2008; Nuñez, Fishman, Fortis, Cooke, & Hotchkiss, 2009). Once these compounds were solubilised into the extractant, the separation of pure pectins from it will be difficult.

As food additives, pectins must meet the standards relating to the purity and quality. Also, it is very important to investigate and control the potential impurities which may impair the quality of pectins.

There have numerous studies on the effect of extraction conditions on the structural characteristics and purity of sugar beet pectins (Garna et al., 2007; Kalapathy & Proctor, 2001; Yapo, Robert et al., 2007), and the effects of purifying mode on the purity of the sugar beet pectins from pulp (Yapo, 2009). They focused on how the extracting and purifying conditions influence the chemical features and purity of the obtained pectins. However, few studies were directed toward understanding of the fine structure of the impurities in sugar beet pectins.

This paper was aimed at characterising a non-pectic compound responsible for the turbidity of the extract and the impurity of sugar beet pectins.

\* Corresponding author at: College of Light Industry and Food Sciences, South China University of Technology, #381, Wushan road, Guangzhou, China. Tel./fax: +86 20 87113668.

E-mail addresses: [lfshjyu@scut.edu.cn](mailto:lfshjyu@scut.edu.cn), [shujuanyu8@gmail.com](mailto:shujuanyu8@gmail.com) (S.-j. Yu).

## 2. Materials and methods

### 2.1. Materials

Sugar beet pulp was a gift from Lvxiang Sugar Company (Xinjiang, China). Ash and moisture content of the pulp was 6.9% w/w, 3.4% w/w respectively. It was ground and kept at 25 °C until used.

### 2.2. Pectin extraction

Hot acid extraction was applied to extract pectins from the pulp. Extractions were performed by heating the samples at 80 °C under stirring for 2 h. Sugar beet pulp suspension (solid–liquid ratio 1:20, w/v) was dispersed in water and set to pH 1.5 with hydrochloric acid. Extractions were stopped by cooling to room temperature with running tap water. Then the slurries were filtered through nylon cloth (23 µm) and centrifuged (8000 rpm, 20 min) to remove the residue. After centrifugation, the clarified sugar beet pectin extract (SBPE) was pooled and aliquots were taken for the pH adjustment.

### 2.3. Particle size distribution, and turbidity of the SBPE

#### 2.3.1. Inline particle size distribution

Particle size distribution of the SBPE was recorded using a focused beam reflectance measurement (FBRM) (Mettler Toledo, USA) equipped with a Lasentec focused beam reflectance probe (Model D6001-C22-K). Prior to determination, the probe was washed with distilled water (400 mL × 3) to ensure no contaminations. For the determination, the extract was set to target pH 1.5, 2.5, 3.5 and 4.5 in sequence using 0.5 M K<sub>2</sub>HPO<sub>4</sub> as pH adjustment. Then the particle size distribution of the extracts was recorded with stirring (400 rpm). Data from the probe were acquired and processed by the intelligent control FBRM (IC-FBRM) software (Mettler Toledo Auto Chem Inc., USA). The so obtained pectin extract with pH adjusted to 1.5, 2.5, 3.5 and 4.5 was referred to SBPE1.5, SBPE2.5, SBPE3.5 and SBPE4.5, respectively.

#### 2.3.2. Turbidity analysis

Turbidity of the extracts was measured by using a turbidimeter (2100AN, HACH, USA). Prior to determination, the turbidimeter was calibrated with six standard turbidity solutions ranging from 0.1 NTU to 7500 NTU. 25 mL of each SBPE with pH set to 1.5, 2.5, 3.5 and 4.5 using NaOH and K<sub>2</sub>HPO<sub>4</sub>, respectively (Levigne et al., 2002; Yapo, Wathélet, & Paquot, 2007), was then made up to 100 mL with distilled water. Turbidity of each sample was directly read from the turbidimeter. Measurements were carried out in duplicate.

### 2.4. Isolation and characterisation of the insoluble substances

#### 2.4.1. Isolation

The substance in the turbid extract was isolated by the centrifugal procedure. 200 mL turbid extract was centrifuged at 8000 rpm for 20 min. After centrifuging, the IS deposited at the bottom of the centrifuge tube was thoroughly washed with water to remove the residue pectins. The supernatant was discarded, and the IS was air-dried at 45 °C until constant weight.

The separation of the IS from the crude pectins was also conducted using the centrifuging procedure. A 0.5% w/w pectin solution was prepared by dissolving sugar beet pectins (SBP) in water. The isolation of the IS from the pectin solution was carried out as described above. However, in this case, the pectic substances

were recovered by precipitating with 3 volume of 95% ethanol, and then air-dried at 45 °C until constant weight.

#### 2.4.2. Characterisation

X-ray diffraction of the IS was obtained with a Bruker diffractometer (Bruker D8 Advance, Germany) equipped with a Cu anticathode. Analysis was performed in the 2θ angle range (5–100°) with a step size of 0.04 2θ angle.

Scanning electron microscope (SEM) measurement was performed using a TM-3000 tabletop microscope (Hitachi, Japan). Prior to the observation, samples were stuck on stubs with double-face tape and coated with a gold–palladium layer. Determination was operated at an accelerating voltage of 15 kV.

Fourier transform infrared (FTIR) spectra (mid-infrared region, 4000–400 cm<sup>-1</sup>) were recorded using a vector 33 spectrometer (Bruker, Germany). FTIR spectra were measured in the transmission mode, with a resolution of 4 cm<sup>-1</sup>. Samples used for analysis were pressed in pellets with potassium bromide.

Determination of oxalate and cations was performed by using an ion chromatography (IC) system (ICS-5000, Dionex, USA), equipped with a conductivity detector and a 25-µL injection loop. All determinations were carried out at 30 °C. The separation of oxalate was performed using an IonPac-AS23 column (4 × 250 mm) in combination with an IonPac-AG23 guard column (4 × 50 mm). Oxalate was eluted isocratically by 4.5 mM Na<sub>2</sub>CO<sub>3</sub> and 0.8 mM NaHCO<sub>3</sub> at the flow rate 1 mL/min. An anion suppressor ASRS (4 mm) was used. A calibration curve was constructed using oxalic acid dissolved in distilled water. Samples were run in triplicate.

The separation of cations was performed using an IonPac-CS12A column (4 × 250 mm) in combination with an IonPac-CG12A guard column (4 × 50 mm). Mineral ions were eluted isocratically by 20 mM methanesulfonic acid at the flow rate 1 mL/min. Calibration curves were constructed using a standard solution obtained from Dionex. An anion suppressor CSRS (4 mm) was used. Samples were run in triplicate.

## 3. Results and discussion

### 3.1. Turbidity and particle size distribution of the turbid SBPE

#### 3.1.1. Turbidity

Turbidity indexes of acidic extracts are presented in Fig. 1. A rapid increase in turbidity was observed when pH increased from 1.5 to 3.5. It was shown that the type of reagent used for pH adjustment (NaOH and K<sub>2</sub>HPO<sub>4</sub>) had no significant effect on the turbidity. In order to isolate the turbid substances, the turbid extract was centrifuged at 8000 rpm for 10 min. Then, turbidity was found to have substantially decreased to the initial level (data not shown). This observation revealed that the IS originally suspended in the extract was responsible for the turbidity. It is added that the

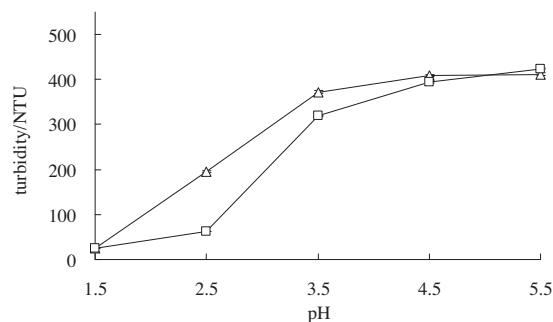


Fig. 1. Turbidity of various sugar beet extracts at different pH, adjusted by NaOH (triangles) and K<sub>2</sub>HPO<sub>4</sub> (squares) respectively.

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