Carbohydrate Polymers 88 (2012) 926–930

Contents lists available at SciVerse ScienceDirect

Carbohydrate Polymers

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

Structure characteristics and rheological properties of acidic polysaccharide from boat-fruited sterculia seeds

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ARTICLE INFO

Article history:
Received 12 December 2011
Received in revised form
31 December 2011
Accepted 13 January 2012
Available online 21 January 2012

Keywords:
Boat-fruited sterculia seed
Pectic polysaccharides
Rheological properties

ABSTRACT

An acidic polysaccharide (APS) was extracted from the boat-fruited sterculia seeds (*Semen Sterculiae Lychnophorae*). It consisted mainly of galacturonic acid (40.1%, w/w) along with rhamnose, arabinose, galactose, xylose and glucose as minor components, indicating a pectic polysaccharide which was confirmed by FT-IR spectra. The degree of esterification of APS determined by FT-IR method was 68%. The shear-thinning and viscoelastic behaviors of APS have been investigated by steady shear and small amplitude oscillatory experiments, respectively. Steady-shear rheological measurement in a range of shear rate (1–1000 s^{-1}) showed increase in pseudoplasticity (or non-Newtonian shear-thinning flow behavior) with the increase in APS concentration (1.0–10%, w/v). APS itself could not form a gel; however, thermal irreversible gels were obtained in the presence of sucrose at low pH (pH < 3.5). The changes in storage modulus G' and loss modulus G'' during heating and cooling cycles indicated that G' was nearly reversible at temperature >30 °C while G'' exhibited thermal hysteresis.

1. Introduction

As a major structural component of the primary cell walls of plants, pectic polysaccharides are widely studied natural anionic biopolymers with useful and versatile properties and applications. Their gelling capacity has provided the basis for most applications as fine chemicals in the food and pharmaceutical industries. They have been used as extrusion material in the formation of pellets, employed in formulations intended for colon drug delivery and as film coatings and tablet matrix in the pharmaceutical industry (Kjøniksen, Hiorth, & Nyström, 2005). In food systems pectic polysaccharides are used as thickeners, stabilizers, gelling agents, emulsifiers, flavor fixation agents and texture modifiers (Fu & Rao, 2001; Wang, Pagan, & Shi, 2002). All the functional properties of these types of biopolymers are closely related to their rheological properties.

Boat-fruited sterculia seed, *Semen Sterculiae Lychnophorae*, is a tropical herb of the *Sterculiaceae* family, mainly distributed in Vietnam, Thailand, Malaysia, Indonesia, as well as South China (Wang et al., 2003). The aqueous extracts from this plant exhibited some biological functions and are commonly used for the treatment of many diseases such as clearing phlegm (by "clearing heat from the lungs" as explained in Chinese medicine) and relieving sore throat to restore the voice on the upper respiratory tract, relaxing the bowels to relieve constipation (Xiao, 2002). In previous studies, we optimized the extraction process of crude polysaccharides (Wu, Cui, Tang, & Gu, 2007) and identified that the acidic polysaccharide component was the active component responsible for the anti-inflammatory properties for the boat-fruited sterculia seeds (Wu, Cui, Tang, Wang, & Gu, 2007). Although some studies on the preparation, sugar composition and biological activity of water-soluble polysaccharides from boat-fruited sterculia seeds have been reported, there is limited information currently available in the literature on the structural and rheological properties of this material (Chen, Li, Shen, Peng, & Xu, 1994; Chen, Cao, & Song, 1996; Somboonpanyakul, Wang, Cui, Barbut, & Jantawat, 2006). In order to explore further the applications and understand the functional properties of this material, the objectives of current work were to study the flow behavior and viscoelastic properties of this acidic polysaccharide.

2. Materials and methods

2.1. Plant material and chemicals

The boat-fruited sterculia seeds harvested in Vietnam were provided by Shanhe Pharmaceutical Co. Ltd. (Wuxi, China). Pectin

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doi:10.1016/j.carbpol.2012.01.035

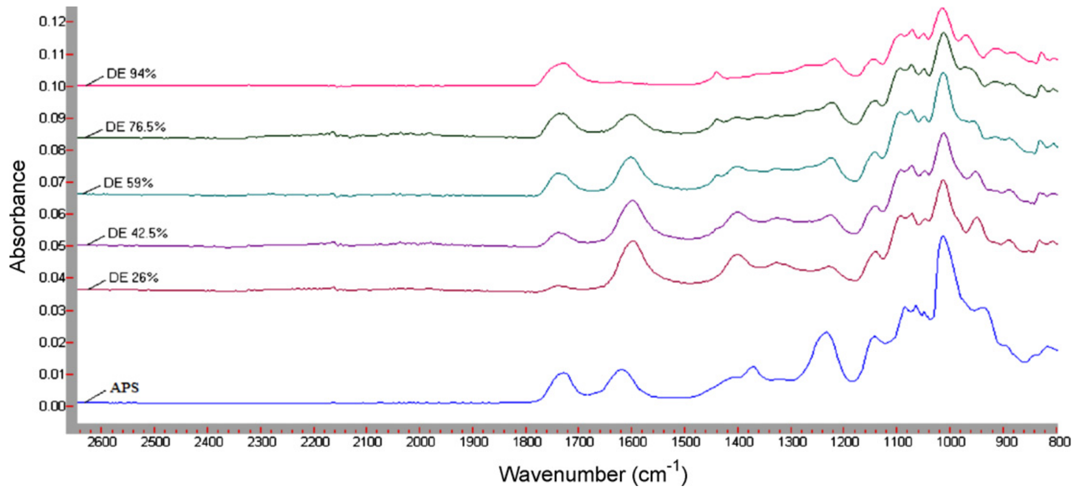

Fig. 1. Fourier transform infrared spectra of APS and pectin standards.

standards with known degree of esterification (DE) (26, 59 and 94%) were obtained from Sigma–Aldrich. Pectin standards (DE: 42.5% and 76.5%) were prepared by mixing accurate amounts of the three commercial pectins.

2.2. Extraction and fractionation of water-soluble polysaccharides

Crude polysaccharides were extracted from boat-fruited sterculia seeds powder and fractionated by anion-exchange chromatography on DEAE-Cellulose column (*D* 2.6 cm × 30 cm) into a neutral polysaccharide (NPS) and an acidic one (APS), as described previously (Wu, Cui, Tang, Wang, et al., 2007).

2.3. Determination of DE value of APS sample

The pectin standards and samples were dried in a vacuum oven at 80 °C for 3 h and desiccated overnight in a vacuum jar prior to FT-IR analysis. FT-IR spectra of the samples were obtained using a Golden-gate Diamond single reflectance ATR in a FTS 7000 FT-IR spectrometer, equipped with a DTGS detector (Digilab, Randolph, MA). The spectra were recorded at the absorbance mode from 4000 to 400 cm^{-1} (mid infrared region) at a resolution of 4 cm^{-1} with 128 co-added scans (Singthong, Cui, Ningsanond, & Goff, 2004). At least triplicate spectra were recorded for each sample.

2.4. Rheological properties

2.4.1. Preparation of the samples

Aqueous polysaccharide solutions at different concentrations ranging from 1.0 to 10% (w/v) were prepared by dissolving the dry polysaccharide powder in distilled water with agitation (60 °C, 2 h) for the steady shear tests. In the viscoelastic measurements, the polysaccharide was first dispersed in 0.1 mol L^{-1} citrate buffer (pH 3.0) at room temperature, then heating to 85 °C, and finally 60% (w/w) of sucrose was added under stirring.

2.4.2. Rheological experiments

All the rheological measurements were conducted on a strain-controlled ARES Rheometer (TA Instruments, New Castle, USA) using a parallel plate (40 mm in diameter, gap 1.0 mm) for both steady shear and oscillatory tests. Steady shear viscosity was determined at various concentrations at 25 °C. The viscoelastic properties, storage modulus (G') and loss modulus (G''), were determined through small amplitude oscillatory test at frequencies from 0.1 to 10 Hz. Prior to any dynamic experiments, a strain sweep test at a constant frequency of 0.1 Hz was conducted to set the upper limit of the linear viscoelastic zone. All oscillatory tests were performed at a strain value of 2%, which was within the linear viscoelastic region. After the upper plate was lowered onto the gel, a thin layer of low viscosity mineral oil was applied to the exposed edge of samples to prevent evaporation of water during the experiments. For all rheological measurements, values reported were the mean of at least two replicates.

3. Results and discussion

3.1. Monosaccharide composition of water-soluble polysaccharides

Protein, total carbohydrate, galacturonic acid and sugar composition of NPS and APS were reported previously (Wu, Cui, Tang, Wang, et al., 2007). NPS was a neutral polysaccharide as it was only eluted out with HAc–NaAc buffer in the anion-exchange chromatography (DEAE–cellulose) and contained no uronic acid; while APS was rich in galacturonic acid (40.1%), rhamnose (11.4%), arabinose (17.5%) and galactose (15.7%), as well as a small amount of xylose (0.6%) and glucose (0.4%). The monosaccharide composition analysis indicated APS from boat-fruited sterculia seeds was a pectic polysaccharide, which was later confirmed by FT-IR spectroscopy (Fig. 1). Since APS demonstrated significant anti-inflammatory activity (Wu, Cui, Tang, Wang, et al., 2007), the focuses of the current study were on the flow behavior and viscoelastic properties of APS and the gelation properties in the presence of co-solutes.

3.2. FT-IR spectrum and DE value of APS

Carbohydrates in the IR spectrum show high absorbencies in the region 1200–950 cm^{-1} (i.e., the so-called fingerprint region), where the position and intensity of the bands are specific for each polysaccharide, allowing its possible identification (Filippov, 1992). For pectic polysaccharides, the unique spectral shape in the fingerprint region is due to the high galacturonan content and the main absorbance regions of galacturonic acid (GalA) are at 1140, 1070, and 1030 cm^{-1} (Kačuráková, Capek, Sasinková, Wellner, & Ebringerová, 2000). The FT-IR spectrum of APS was compared with commercial pectin standards (Fig. 1). The shape of spectrum of APS was similar to that of commercial pectin standards confirming that APS was a pectic polysaccharide.