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Dilute solution, flow behavior, thixotropy and viscoelastic characterization of cress seed (*Lepidium sativum*) gum fractions

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ABSTRACT

In this study, rheological properties of cress seed gum (CSG) and its fractions (F1, F2, F3; fractionated using stepwise extraction with water) were investigated. Cress seed gum and its fractions revealed random coil conformation in dilute regimes; chain flexibility and intrinsic viscosity increased from F1 to F2 to F3. The mechanical spectra derived from strain sweep and frequency sweep measurements indicated that the gum dispersions had viscoelastic behavior; all of them were classified as weak gels and the gel network got stronger along the series of F1, F2 and F3. Arrhenius-type model was used to describe the effect of temperature; F2 and F1 showed the highest and the lowest activation energy, respectively. All gum dispersions displayed thixotropic behavior; hysteresis loop area and structural recovery increased significantly along the series of F1, F2 and F3. In general, the results indicated that CSG and the fractions exhibited significantly different rheological properties.

1. Introduction

Nowadays, there is a great interest for natural hydrocolloids with low cost and proper functionality (Behrouzian, Razavi, & Karazhiyan, 2014). Extraction optimization, some physicochemical, functional and rheological properties of cress (*Lepidium sativum*) seed gum as a new source of hydrocolloid have been recently studied (Behrouzian, Razavi, & Karazhiyan, 2013; Behrouzian et al., 2014; Hassan et al., 2015; Jafari, Mahdavi-Khazaei, & Hemmati-Kakhki, 2016; Jouki, Khazaei, Ghasemlou, & HadiNezhad, 2013; Karazhiyan, Razavi, & Phillips, 2011a; Karazhiyan et al., 2009; Karazhiyan et al., 2011b; Naji & Razavi, 2014; Naji, Razavi, & Karazhiyan, 2012b; Naji, Razavi, & Karazhiyan, 2013; Naji, Razavi, Karazhiyan, & Koocheki, 2012a; Razavi, Bostan, Niknia, & Razmkhah, 2011; Sahraiyan, Naghipour, Karimi, & Ghiafe Davoodi, 2013; Tyler, 1965). Cress seed gum (CSG) was mainly composed of a carbohydrate polymer (77%) with a molecular weight of about 540 kDa and radius of gyration 75 nm. Structural and physicochemical studies confirmed polyelectrolyte nature of CSG, which largely originates from the presence of carboxyl groups in galacturonic acid and glucuronic acid units (Karazhiyan et al., 2009, 2011b). Newtonian flow behavior at dilute concentrations below 0.1% was observed for cress seed gum. However, it showed a pronounced shear thinning behavior in steady shear measurements, and viscoelastic behavior in dynamic tests at higher concentrations. The rheological properties of cress seed gum depended on many factors such as shear rate, temperature, time, pH, biopolymer concentration, concentration and type of salts and sugars (Behrouzian et al., 2013; Karazhiyan et al., 2009, 2011b; Naji & Razavi, 2014).

Many researchers investigated fractionation of hydrocolloids by different methods and the results indicated that the fractions had different physicochemical, functional and rheological properties (Guo, Cui, Wang, & Young, 2008; Guo et al., 2011; Jian et al., 2014; Kang et al., 2011; Mohammadifar, Musavi, Kiumarsi, & Williams, 2006; Qian, Cui, Wang, Wang, & Zhou, 2011; Qian, Cui, Wu, & Goff, 2012; Román-Guerrero et al., 2009; Simas-Tosin et al., 2010; Wagner et al., 2007; Wu, Cui, Tang, Wang, & Gu, 2007; Zhang, Zhang, Cheung, & Dong, 2003). As reported earlier, cress seed gum could be successfully fractionated using stepwise extraction with water, to give three fractions with different physicochemical and functional properties: F1 (first extractant), F2 (second extractant)

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and F3 (third extractant). Ash, moisture and uronic acid contents decreased along the series of F1, F2 and F3, whereas molecular weight and the amount of carbon, hydrogen and nitrogen increased along the same series. In general, all gums contained relatively high amounts of arabinose, xylose and rhamnose. Xylose and mannose contents were almost the same in all the samples. Fucose, rhamnose, and galactose contents decreased significantly from F1 to F2 to F3, in contrast, arabinose and glucose content increased along the same series. Generally, the major identical peaks of FT-IR spectra for cress seed gum and its fractions were similar. The results of DSC and TGA exhibited that F3 had the highest thermal stability. F3 imparted the best surface activity and the trend of surface tension was F3 < F2 < F1 at low concentrations (0.01 and 0.05%). Cress seed gum and its fractions indicated good emulsifying capabilities (>97%) and stabilities (>96%). The emulsion capacity increased slightly along the series of F1, F2 and F3, whereas, emulsion stability decreased. Foaming capacity (FC) and stability (FS) increased from F1 to F2, but F3 showed the lowest FC and FS (Razmkhah, Razavi, Mohammadifar, Koocheki, & Ale, 2016).

The rheological behavior of the fractions is of special importance to adjust processing parameters, monitoring consistency as well as predicting the stability of fluid systems, and the final textural attributes of formulated products. With respect to different physicochemical properties of the fractions, various rheological characteristics are expected for them. Therefore, the objective of this paper was to investigate rheological properties of cress seed gum (whole gum) and its fractions including: intrinsic viscosity, steady and dynamic shear rheological properties, temperature dependency and thixotropy.

2. Materials and methods

2.1. Cress seed gum extraction and fractionation

Seeds of *Lepidium sativum* were procured from a local market in Tehran, Iran. Cress seed gum (whole gum) and its fractions were prepared exactly based on the procedure as described previously (Razmkhah et al., 2016). Aqueous cress seed gum was extracted from whole seeds using distilled water (water to seed ratio of 30:1, pH 7) at room temperature (21 °C). Three fractions (F1, F2 and F3) were obtained by dividing of the water volume (water to seed ratio of 30:1) and extraction time (15 min) into three parts (water to seed ratio of 10:1 and extraction time: 5 min). All the samples (CSG, F1, F2 and F3) were purified by ethanol precipitation (three volumes of 96% ethyl alcohol for 2 h at room temperature). The collected precipitate was dried at room temperature for 24 h and the dried gums were ground.

2.2. Dilute solution measurements

Gum solutions (0.0015 g ml^{-1}) were prepared by dispersing the samples (F1, F2, F3 and CSG) in deionized water and stirring (500 rpm) with a magnetic stirrer for about 2 h at ambient temperature until full dissolution. The solutions were stored at 4 °C for 48 h in order to complete the hydration. Viscosity of dilute solutions was measured at 25 °C ± 0.1 °C, using an Ubbelohde viscometer (size 1, Fisher scientific, USA), which was suspended in a thermostatic water bath under precise temperature control. The flow time in seconds was measured at least three times.

The sample viscosity (η) was converted to relative viscosity (η_{rel}) and specific viscosity (η_{sp}) using the following equations:

$$\eta_{rel} = \frac{\eta}{\eta_s} \quad (1)$$

$$\eta_{sp} = \frac{\eta - \eta_s}{\eta_s} = \eta_{rel} - 1 \quad (2)$$

Where, η_s is the viscosity of the solvent (deionized water).

The relative viscosity (η_{rel}) was kept in the range of 1.2–2.0, in order to assure good accuracy and linearity of extrapolation to zero concentration (Da Silva & Rao, 1992). The intrinsic viscosity [η] is usually estimated by double extrapolation to zero concentration of the following models:

Huggins' equation (Huggins, 1942):

$$\frac{\eta_{sp}}{C} = [\eta] + k_H[\eta]^2 C \quad (3)$$

Kraemer's equation (Kraemer, 1938):

$$\frac{\ln\eta_{rel}}{C} = [\eta] + k_K[\eta]^2 C \quad (4)$$

Where, k_H and k_K are the Huggins' and Kraemer's constants, respectively, and C is the solution concentration.

2.3. Estimation of the molecular conformation

The power-law relation was applied to estimate exponent *b* from the slope of a double logarithmic plot of η_{sp} against concentration (Eq. 5), and this parameter provides an indication of the conformation of polysaccharides (Higiro, Herald, Alavi, & Bean, 2007; Lai, Tung, & Lin, 2000).

$$\eta_{sp} = aC^b \quad (5)$$

2.4. Rheological measurements

Gum solutions (1%) were prepared by dispersing the samples in distilled water and stirring (500 rpm) with a magnetic stirrer for about 2 h at ambient temperature until full dissolution. The solutions were stored at 4 °C overnight for complete hydration. Rheological evaluations were performed with a Physica MCR 301 rheometer (Anton Paar, GmbH, Graz, Austria) using a serrated parallel plate system (PP40/S-SN16891; d = 0.5 mm) with duplication measurement. The temperature was controlled with a Peltier system (Viscotherm VT2) equipped with a fluid circulator (Anton Paar, GmbH) with the accuracy of ±0.01. All samples were left at rest for five minutes to allow structure recovery and temperature equilibration. The samples were covered with a solvent trap to prevent evaporation during the measurements. Rheoplus software version 3.21 (Anton-Paar) was used to collect and analyze the rheological data.

2.4.1. Small dynamic oscillatory measurements

2.4.1.1. Strain sweep. Strain sweep tests were performed in the amplitude range of 0.04–700% at 1 Hz and 25 °C to determine: 1) γ_L or LVE (the linear viscoelastic range); 2) G'_{LVE} (elastic modulus at critical strain); 3) τ_y or yield stress (the resistance to mechanical force that can be determined from the limiting value of LVE range in terms of shear stress); 4) τ_f or the flow point (the stress in which internal structure breaks to such an extent that it causes the material to flow ($G' = G''$); 5) tan δ or damping factor (a view of whether the samples behaved as liquids or solids and can be