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Dynamic rheological properties of *Lepidium perfoliatum* seed gum: Effect of concentration, temperature and heating/cooling rate

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ABSTRACT

Dynamic rheological properties of *Lepidium perfoliatum* seed gum (LPSG) in the linear viscoelastic region were investigated as a function of concentration (1.5, 2, 2.5 and 3% w/v), temperature (5–85 °C) and heating–cooling rate (1, 5 and 10 °C/min). It was clearly observed that the gum dispersions exhibited viscoelastic properties in a given temperatures. The storage modulus (G') was always higher than the loss modulus (G'') in all concentrations and temperatures. Mechanical spectra of LPSG were classified as weak gels based on the frequency sweep, complex viscosity (η^*) and tan δ results. Moreover, G' and G'' changes were found to be dependent on concentration, temperature and heating–cooling rate. At 5 °C, the storage modulus increases with increase in gum concentration. Similar results were observed for 85 °C, except for sample containing 2.5% LPSG. Gum solutions measured at 85 °C had higher storage modulus compared to those evaluate at 5 °C. The effect of temperature on LPSG was investigated during heating and cooling. At higher gum concentrations (2.5–3%), as the temperature increased from 50 to 85 °C, the storage modulus started to increase. While for low LPSG concentrations, increase in temperature had no significant effect on the storage modulus. The storage modulus of 3% LPSG increased drastically by cooling the samples from 85 to 5 °C. This increase was much higher for samples with 1 °C/min cooling rate.

1. Introduction

Lepidium, of the Cruciferae family, is a genus of 230 species and is distributed throughout the world. *Lepidium perfoliatum* plant is native to some of middle-east's countries such as Egypt, Arabia, Iraq, Iran and Pakistan (Amin, 2005). The seeds have been used for hundreds of years in traditional Iranian medicinal prescriptions. *Lepidium perfoliatum* seed gum (LPSG) occurs mainly at the outermost layer of hull. This hull is able to release mucilaginous material easily when soaked in water. Therefore, aqueous extraction is one of the most common techniques applied for the extraction of the seed mucilaginous material (Koocheki, Taherian, Razavi, & Bostan, 2009). Our previous researches showed that LPSG can be used as a new source of food hydrocolloid. This gum has a great potential to act as a thickening and stabilizing agent in food systems (Koocheki, Taherian, & Bostan, 2013; Soleimanpour, Koocheki, & Rassoul Kadkhodaee, 2013a, 2013b). Steady shear flow properties showed that LPSG has high viscosity, yield stress and strong shear thinning characteristics (Koocheki et al., 2013). This gum is able to bind and immobilize a large amount of water thus increasing viscosity, modifying texture and stabilizing product consistency.

Dynamic rheological properties can be used along with steady shear rheological properties to provide insight on the structure of the sample (Clark & Ross-Murphy, 1987). Small amplitude oscillatory shear analyzes are a type of dynamic rheological test in which stress and strain are varied harmonically with time in the linear viscoelastic region (LVR). Expanding the database on the viscoelastic properties of gums solutions/dispersions is critical to the food processor for adjusting processing parameters, monitoring consistency as well as predicting the stability of fluid food systems and the final textural attributes of formulated foods. Therefore, dynamic rheology is one of the methods most extensively used to assess the viscoelastic behavior of polysaccharide solutions/dispersions or gels. The viscoelastic properties of various gums such as xanthan, guar (Mills & Kokini, 1984), pectin (Gigli, Garnier, & Piazza, 2009), *Opuntia ficus* indica mucilage (Medina-Torres, Brito-De La Fuente, Torrestiana-Sanchez, & Katthain, 2000), basil seed gum (Rafe & Razavi, 2013) and gum tragacanth (Balaghi, Mohammadifar, Zargaraan, Ahmadi Gavlighi, & Mohammadi, 2011) have been reported by other researchers.

Because of difference in gum structure and extrinsic conditions within the fluid food system, the rheological behavior is quite

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different from one gum solution to another. Due to the diversity of gums and their modified derivatives, food companies may have a difficult time making decisions regarding the choice of gums for addition to their fluid food formulations. Thus, understanding the rheological properties of LPSG is essential for evaluating its potential applications and use as food thickeners or stabilizers.

Although the physicochemical properties and steady-state rheological evaluation of LPSG have been recently established (Koocheki et al., 2013), there is no published information about the viscoelastic behaviour of this gum. Therefore, the main aim of the present work was to determine the dynamic rheological properties of LPSG and its dependency to concentration and temperature. The impact of successive heating–cooling cycles on the viscoelasticity was also determined.

2. Materials and methods

2.1. Material

L. perfoliatum seeds were purchased locally from the medical plant market, Mashhad, Iran. The seeds were cleaned manually to remove all foreign matter such as dust, dirt, stones, chaff, immature and broken seeds. *L. perfoliatum* seed gum was extracted according to the method described previously (Koocheki et al., 2009). Gum extracted under this condition contains 88.23% total sugar, 4.6% protein, 6.0% moisture, 0.18% ash and no fat (Koocheki et al., 2013).

2.2. Gum samples preparation

After preparation of dried gum, aqueous dispersions of *L. perfoliatum* seed gum at different concentration (1.5, 2, 2.5 and 3% w/v) were prepared in deionized water containing 0.02% sodium azide as a microbial preservative while stirring for 30 min. 10 ml of dispersions were stirred by roller shaker for 24 h at room temperature; then they were left at 4 °C overnight (to ensure a complete hydration) prior to use in oscillation tests.

2.3. Small dynamic oscillatory measurements

Dynamic rheological measurements were conducted with a Physica MCR301 controlled stress/strain rheometer (AnTon paar GmbH, Germany) equipped with the parallel plate system (50 mm diameter, and 1.000 mm gap). Each sample was transferred onto the rheometer plate at the desired temperature and excess material was wiped off with a spatula. The temperature of the bottom plate was controlled with a Peltier system (Viscotherm VT2, Phar Physica) for the fast and precise temperature control. In order to relax the samples before the measurements, all samples were allowed to rest at the initial temperatures for 1 min. The rim of the sample was coated with a thin layer of silicon oil to prevent evaporation during measurements at high temperatures. The viscoelastic parameters were determined for the LPSG at four levels of concentration.

Physica Rheometer Data Analysis software (Rheoplus/32, version V3.40) was employed to calculate the storage modulus (G′), loss modulus (G''), loss tangent (tan δ) and complex viscosity (η^*) and analyze the rheological results. At least duplicate of each measurement were made.

Before making detailed dynamic measurements to probe the sample's microstructure, the linear viscoelastic region (LVR) must first be defined. The linear viscoelastic region (LVR) for LPSG samples was determined by performing an amplitude sweep measurements (0.01–100%) at constant frequency (1 Hz) and two temperatures of 5 and 85 °C.

Frequency sweep tests at a constant strain in the LVE region were carried out to determine the viscoelastic nature of LPSG. In this test a strain of 0.02 was applied in order to disturb as less as possible the network formation. The mechanical spectra were characterized by values of G', G'' (Pa) as a function of frequency in the range of 0.01–10 Hz and two temperatures (5 °C and 85 °C). The storage modulus can be used as a measure of the elastic component of the sample and similarly, the loss modulus – the viscous component of the sample.

The temperature sweep measurements were performed at the constant strain of 0.2%, which was well within the linear viscoelastic region, while the frequency was fixed at 1.0 Hz. To investigate the effect of temperature on the rheological properties of LPSG, a program was set up. Briefly, the program was included: (1) a linear temperature increase from 5 °C to 85 °C (heating), using three rates of 1, 5 and 10 °C/min, (2) holding at 85 °C for 20 min, (3) a cooling step with a linear temperature decrease to 5 °C (cooling) at the same speeds and (4) holding at 5 °C for 20 min.

2.4. Statistical analysis

The experiments were organized as a randomized full factorial design with hydrocolloids (four levels) and temperature (two levels) as the main effects. The whole design was replicated twice. The results were analyzed using a general linear model (GLM) procedure of the MINITAB ver. 14.2. The level of significance was preset at $P < 0.05$.

3. Result and discussion

3.1. Strain sweep measurements

With increasing stain, two different regions namely linear viscoelastic region where G' and G'' were almost constant, and nonlinear region in which G' and G'' started to decrease were distinguished. In the strain sweep tests, G' remained constant until the strain reached a critical point at which G' started to decrease sharply, as demonstrated in Fig. 1. The strain at which G' decreased sharply is defined as the critical strain. Therefore, critical strain reflects the deformability of the gum.

Strong gum solutions maintain longer at linear state compared to weak gum solutions (Steffe, 1996); in other words, viscoelastic moduli can be linear in a wide strain range. The linear region for dilute solutions is less than concentrate solutions and this is less than gels. While the strain value at the limit of LVE rarely exceeds 0.1 for colloidal gels, a larger LVE region with a strain equal to or exceeds 1 is for natural biopolymer gels (Clark & Ross-Murphy, 1987).

The limiting values of strain (γ_L), tan δ and τ obtained within the LVE range are presented in Table 1. At low gum concentrations (1.5–2.5%) and temperature, the elastic modulus remained constant at strain up to about 1%. With increase in gum concentration, the strain at which the elastic modulus decreases, increased to more than 1% (Table 1). This indicates that increasing gum concentration increased the strength of the system and got more rigid. Increase in temperature from 5 to 85 °C, also caused γ_L to increase. The limiting value of strain (γ_L) was high for 3% LPSG at 85 °C, implying a higher stability of the viscoelastic material under the γ-amplitude. Therefore, the strain of 0.02% for frequency sweep and 0.2% for temperature sweep tests were well within the linear viscoelastic region, where the weak gel network was not damaged by the strain imposed during the measurements.

The values of G' and G'' at LVE region also increased with increase in gum concentrations (Table 1). The effect of increasing temperature was similar to that of concentration. Increasing temperature from 5 to 85 °C increased the structural strength (G' at LVE) of gum solution at constant concentration.

This type of test also determines the maximum deformation that a system can withstand without structural failure; in other