



Effects of drying methods on rheological properties of flaxseed gum

Yong Wang^{a,1}, Li-Jun Wang^{b,1}, Dong Li^{a,*}, Jun Xue^c, Zhi-Huai Mao^a

^a College of Engineering, China Agricultural University, P.O. Box 50, 17 Qinghua Donglu, Beijing 100083, China

^b College of Food Science and Nutritional Engineering, China Agricultural University, P.O. Box 50, 17 Qinghua Donglu, Beijing 100083, China

^c Guelph Food Research Center, Agriculture and Agri-Food Canada, Guelph, Ont., Canada

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ABSTRACT

Flaxseed gum solutions were extracted and dried by different methods: ethanol precipitation, freeze drying, 105 °C oven drying, 80 °C oven drying, spray drying, and vacuum drying. The effects of these drying methods on the rheological properties of flaxseed gum were investigated in present study. Ethanol precipitation increased the apparent viscosity of flaxseed gum solution, while all the other methods decreased the apparent viscosity. Most of the drying methods slightly increased the activation energy, except ethanol precipitation. In frequency sweep test, all the drying methods reduced the G' and G'' values. In creep–recovery tests, the data were modeled by Berger's model. The E_2 and η_1 values were reduced by all of the drying methods in this study. Some relationships were found between the parameters in the Power Law model of the frequency sweep test and the parameters in Berger's model.

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1. Introduction

Flaxseed has the natural properties of fiber, lignans, and omega-3 fatty acids that provide preventative and restorative abilities to human diet (Zhang et al., 2007). Flaxseed polysaccharide, commonly referred to as gum, is of special interest because it has been proven that the rarely occurring hexose, L-galactose, is a constituent sugar (Easterby & Jones, 1950). Flaxseed gum has good water-holding capacities. The water binding ability and the rheological properties of flaxseed gum are similar to those of guar gum (Fedeniuk & Biliaderis, 1994). Flaxseed gum also shows weak gel properties, thus it can be used to replace most of the non-gelling gums for food and non-food applications (Chen, Xu, & Wang, 2006; Cui & Mazza, 1996). Moreover, flaxseed gum is a potential functional food as a water-soluble dietary fiber, which appears to play a role in reducing diabetes and heart disease risk (Cunnane et al., 1993; Oomah & Mazza, 1998; Stavro, Marchie, Kendall, Vuksan, & Jenkins, 2003).

The influences of concentration, pH, temperature, electrolyte conditions, extraction conditions, and mixing with starch on the rheological properties of flaxseed were investigated extensively using both small amplitude oscillatory and continuous shear measurements (Cui, Mazza, & Biliaderis, 1994a; Cui, Mazza, Oomah, & Biliaderis, 1994b; Mazza & Biliaderis, 1989; Oomah, Kenaschuk, Cui, & Mazza, 1995; Wang et al., 2008). Flaxseed gum has been extracted and dried by many methods. Oomah and Mazza (2001)

optimized the parameters in spray drying of flaxseed gum, according to its rheological properties, gum yield, color, and cyanogenic glycoside content. Freeze drying has also been adopted for the preparation of flaxseed gum (Mazza & Biliaderis, 1989; Rebolé et al., 2002). And some researchers have used ethanol to precipitate the flaxseed gum from the water-extracted gum solution (Batty, 1993; Cui et al., 1994a; Fedeniuk & Biliaderis, 1994).

Drying process is of vital importance in the production of flaxseed gum, since flaxseed gum is often used as powder in food industry. And the rheological properties are among the most important functional properties of gums. However, there has been little published research on the effects of different drying methods on the rheological properties of flaxseed gum. Therefore, the main objective of this study was to investigate the influences of different drying methods on the rheological properties of flaxseed gum. The drying methods were selected based on the literature and preliminary experiments as follows: ethanol precipitation, freeze drying, 105 °C oven drying, 80 °C oven drying, spray drying, and vacuum drying. Considering the properties of flaxseed gum, the rheological tests included apparent viscosity, activation energy, frequency sweep, creep–recovery, and gelling temperature. This study should provide useful information for the processing of flaxseed gum, both for research and for potential industrial applications.

2. Materials and methods

2.1. Materials

Flaxseed was purchased from the Hebei province of China, with moisture content of 6.50%.

* Corresponding author. Tel./fax: +86 10 62737351.

E-mail address: dongli@cau.edu.cn (D. Li).

¹ These authors contributed equally to this work.

Nomenclature

E_a	activation energy, kJ/mol	R^2	correlation coefficient (dimensionless)
E_1	instantaneous elastic modulus, Pa	T	absolute temperature, K
E_2	retarded elastic modulus, Pa	t_2	relaxation time, s
G'	storage modulus, Pa	$\dot{\gamma}$	shear rate, s^{-1}
G''	loss modulus, Pa	δ	loss angle, $^\circ$
K	consistency index, $Pa\ s^n$	η_a	apparent viscosity, Pa s
K'	index, $Pa\ s^n$	η_∞	frequency factor (dimensionless)
K''	index, $Pa\ s^n$	η_1	coefficient of viscosity associated with viscosity flow, Pa s
n	flow behavior index (dimensionless)	σ	constantly applied compressive stress, Pa
n'	frequency exponent (dimensionless)	τ	shear stress, Pa
n''	frequency exponent (dimensionless)	ω	angular frequency, rad/s
R	gas constant, J/mol K		

2.2. Flaxseed gum extraction

Flaxseed (100 g) was washed in water for 1 min to remove the surface dust, and then mixed with 900 mL deionized water. The flaxseed and water were then stirred for 5 h at a speed of 300 r/min, in a 60 °C water bath, according to the method of Cui (2001). Since the focus of this study was to investigate the influence of drying methods on flaxseed gum, all parameters influencing the gum extraction were fixed at constant value. The extracted flaxseed gum solution was filtered through 40-mesh screen. And the extracted flaxseed gum solution was adjusted to 1% concentration by adding proper amount of deionized water (pH 6.4–7.0).

The protein content of flaxseed gum extracted by the above method was $14.4 \pm 0.2\%$, determined by Kjeldahl method, using a FOSS Kjeltac 2300 analyzer (FOSS Co., Höganäs Sweden). The nitrogen data were converted into protein values employing a conversion factor of 6.25. The fat content in flaxseed gum was $0.59 \pm 0.13\%$, determined by Soxhlet standard extraction using Universal Extraction System (B-811, BÜCHI Labortechnik AG, Switzerland). And the ash content was $3.35 \pm 0.12\%$. Analysis was performed in triplicate.

2.3. Drying methods

Extracted flaxseed gum solution (1%) was spray dried in a GPW120-II spray drier (Shandong Tianli Drying Equipment Inc., Jinan, China). The inlet temperature was set at 200 °C, and outlet temperature was set at 105 °C. The feed rate was 4 mL/min. The dried flaxseed gum was collected in the materials box at the bottom of the cyclone.

Extracted flaxseed gum solution (1%) was vacuum dried in a DZ-3 vacuum drier (Tianjin Taisite Instrument Co., Tianjin, China). The temperature was set at 60 °C for 24 h. And the vacuum pressure was less than 60 Pa.

Extracted flaxseed gum solution (1%) was dried in a hot air oven (model 101-3, Shanghai Luda Experimental Instrument Co., Shanghai, China) at 105 °C for 8 h and at 80 °C for 24 h, respectively.

Extracted flaxseed gum solution (1%) was precipitated with two volumes of 95% ethanol, collected by centrifugation at 3000 r/min for 10 min using an LG10-2.4 A machine (Beijing Medical Centrifuge Corporation, Beijing, China), according to the method of Cui et al. (1994a) with some modification on drying method. The precipitated flaxseed gum was then dried in a hot air oven at 80 °C for 8 h.

Extracted flaxseed gum solution (1%) was freeze dried in a LGJ-18S freeze dryer (Beijing Songyuan Huaxing Technology Develop Co., Beijing, China) for 24 h. The freeze dryer was equipped with a temperature controller, which controlled the temperature to increase steadily.

2.4. Solution preparation

For rheological tests, flaxseed gums dried by different methods were dissolved in deionized water (pH 6.4–7.0) using a magnetic stirrer for 30 min at 25 °C, to make the 1% (dry base, w/w) flaxseed gum solutions. The untreated flaxseed gum was used as the control sample to indicate the effects of different drying methods on the rheological properties of flaxseed gums during testing.

2.5. Rheological tests

Rheological measurements were performed using AR2000ex rheometer (TA Instruments Ltd., Crawley, UK). The temperature was controlled by a water bath connected to the Peltier system in the bottom plate. A thin layer of silicone oil was applied on the surface of the samples in order to prevent evaporation. The linear viscoelastic region was determined for each sample through strain sweeps at 1 Hz (data not shown). Viscoelastic properties (storage modulus G' , loss modulus G'' , and δ) of flaxseed gum solutions were determined within the linear viscoelastic region. An equilibration of 2 min was performed before each measurement.

2.5.1. Continuous shear measurements

The continuous shear tests were performed at 25 °C over the shear rate range of 0.1–100 s^{-1} to measure the apparent viscosity. A steel cone geometry (60 mm diameter, 59 μ m gap) was chosen for the continuous shear measurements.

2.5.2. Activation energy measurements

The apparent viscosity was determined over the temperature range from 10 °C to 50 °C, in order to determine the activation energy by the Arrhenius relationship, at a constant shear rate of 10 s^{-1} . A steel cone geometry (60 mm diameter, 59 μ m gap) was chosen for the activation energy measurements.

2.5.3. Frequency sweep measurements

The frequency sweep tests were performed at 25 °C over the angular frequency range of 0.01–10 rad/s. The strain amplitude for the frequency sweep measurements was selected as 1% according to the strain sweep results (data not shown) in order to be in the linear viscoelastic region for all samples. An aluminum parallel plate geometry (40 mm diameter, 1 mm gap) was chosen for the frequency sweep measurements.

2.5.4. Creep–recovery measurements

Creep–recovery experiments were carried out using shear stress of 7.958 mPa at 25 °C. The variation in shear strain in response to the applied stress was measured over a period of 2 min; the stress

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