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Effect of spray drying on the properties of camelina gum isolated from camelina seeds

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ABSTRACT

Camelina gum from camelina seed was solubilized with water and isolated using spray drying method. Ethanol precipitation method was used as control. The effect of drying temperature (140–180 °C) on the properties of camelina gum was studied. The camelina gum yield from spray drying isolation is up to 1.89% of camelina seed, which is significantly less than that from ethanol isolation method (2.04%). In addition, the camelina gum isolated from spray drying has lower polysaccharides (62 vs 72%) and higher protein (7 vs 6%). In general, the viscosity of camelina gum isolated from spray drying is less than that isolated from ethanol precipitation method. Results showed that relative high temperature (165 °C) had a positive effect on the gum yield, purity, and viscosity. High drying temperature (180 °C) led to decreased gum yield and viscosity due to chemical structure decomposition of gum. Camelina gum solution exhibited superior stability in both acidic and weakly basic ranges. At pH 12, its viscosity was greatly increased, attributed to the promotion of crosslinking between polysaccharides and protein in camelina gum. The results of the additive effects on the rheological properties of camelina gum revealed that camelina gum has a good compatibility with those additives studied, indicating that camelina has a great potential for being used as a stabilizer.

1. Introduction

Camelina falls into the Brassicacease family and oilseed crop in North America, has been native to Europe and Asia, and its growing history can be traced back to 600 BCE in Germany (Li et al., 2016). Camelina was recognized as “Gold of pleasure” and spread to the United States because it has the merits of a short growth cycle (85–100 days) and strong cold tolerance (Budin et al., 1995). Camelina’s chemical composition varies with species, growing location, and environment, and it roughly contains 30–49% oil, 23–30% protein, 10% carbohydrates and 6.6% ash (Berti et al., 2016; Budin et al., 1995). Camelina oil is known for rich alpha-linolenic acid (ALA) and omega-3 fatty acid, which greatly promotes camelina research value for food and non-food usage such as biodiesel and biolubricant (Budin et al., 1995; Li et al., 2014).

Camelina gum extracted from camelina seed has shown excellent viscoelastic property and has the potential for use as a thickener, suspending agent, film, and stabilizer (Li et al., 2016; Qi et al., 2016). Previous studies indicated that around 2% of camelina gum was extractable from camelina seeds, and camelina gum is the heterogeneous material that consists of polysaccharide (70%) and protein (12.3%) (Li et al., 2016; Li et al., 2010). The extracted polysaccharide from camelina seed has been composed of galactose (58.1%), glucose (25.0%), rhamonse (11.6%), and xylose (5.2%) (Li et al., 2016). Camelina gum extracted from seeds showed superior viscosity and elastic modulus compared with commercial gums of k-carrageenan and hydroxyethlcellulose (HEC) which are widely used in food and pharmaceutical industries as thickeners or stabilizers (Li et al., 2016). Similar to other gums, camelina gum showed the potential of being used as emulsifier, stabilizer, gelling agent and adhesive in food and non-food manufacturing (Glicksman, 1982; Cui, 2005). Based on previous studies, to isolate camelina gum, the gum needs to be solubilized with water and separated from the insoluble part with a process called filtration or centrifugation, then, the gum is precipitated with organic solvent such as ethanol, followed by a freeze drying process (Li et al., 2010; Li et al., 2016).

The drying method is paramount in gum production and plays an important role to the properties of gum (York, 1983). Freeze dry, spray dry, vacuum dry, and air dry methods are the major drying methods that have been widely used in gum isolation. Previous studies have revealed that spray drying could be used to harvest pure, white, uniform gum powder with improved product properties and eliminated

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harmful bacteria (Glicksman, 1982; Aponte et al., 2016). In addition, spray drying is more cost-effective; it can be 30–50 times less expensive than freeze drying for industrial scale production of pharmaceutical and food additives (Oomah and Mazza, 2001; Silva et al., 2011).

The previously described method for camelina gum isolation requires a mass of organic solvent and energy consumption. There is no study regarding isolating camelina gum with the spray drying method that has been reported so far. The objective of this study was to investigate the effect of spray drying technology at varied temperatures on the yield, purity, and rheological properties of camelina gum. Additives such as sugar and salts have shown great influence on the viscoelastic properties on gums (Maurer et al., 2012; Higiro et al., 2007). Therefore, the effect of different additives on the properties of camelina gum was evaluated. The morphological properties of gum were also characterized.

2. Materials and methods

2.1. Materials

Camelina seed was supplied by Montata Gluten Free Processors LLC and was manually cleaned with 48 mesh sieve (W. S. Tyler Company, Belgrade, MT, USA) before use. Phenol 5.0% (w/v), sucrose, glucose, NaCl and HCl were purchased from Fisher Scientific Co., (Fair Lawn, NJ, USA). All the reported experimental data were obtained in triplicate with the mean values.

2.2. Camelina gum isolation

Cleaned camelina seeds were mixed with distilled water at a solid/liquid ratio of 1:20 (w/w) with a Hobart D300 Stand Mixer (Hobart Co, OH, USA), and the mixture was stirred for 2 h at room temperature. The mixed solution was then passed through a sifter with 40-mesh screen (420 μm) to separate the extracts solution and seeds residues. Centrifugation of the camelina gum suspension at 7000 rpm for 10 min was performed to remove the precipitate (Sorvall RC 6+ Centrifuge, Thermo Scientific Asheville, NC, USA). The supernatant was filtered with 4-layers cheese cloth first to remove impurities and then dried in a spray dryer (LPG-5 Centrifugal spray dryer, Jiangsu, China) operated at an inlet temperature of 140 °C to 180 °C and outlet temperature of 100 °C. The sample feeding rate was controlled at 4–5 kg/hour by adjusting the pump speed. The collected gum powder was then ground using an Udy cyclone sample mill with 0.25 mm screen (Udy, Ft. Collins, CO, USA), packed and stored at room temperature for further analysis.

The control sample was extracted using ethanol precipitation and freeze drying method for comparison. The camelina seeds mixed with distilled water at the solid/liquid ratio of 1:20 (w/w) were stirred for 2 h at room temperature. The extracted solution was filtered with 40 mesh sieve followed by centrifugation at 7000 rpm for 10 min. The supernatant was treated with absolute ethyl alcohol to precipitate camelina gum out of solution. The freeze dryer VIRTIS 360.66 (The Virtis Company, INC, Gardiner, NY, USA) was then applied to remove moisture in camelina gum suspensions. The precipitate was collected and ground using an Udy cyclone sample mill with 0.25 mm screen (Udy, Ft. Collins, CO, USA).

2.3. Chemical analysis

The total sugar content of camelina gum was measured by the phenol-sulphuric acid method (Dubois et al., 1956). First, Standard curve ($y = 64.993x + 0.0049$ with R^2 of 0.997) was established using glucose. Next, 20 mg of the sample was placed in a 500 mL volumetric flask, and diluted with distilled water to the full-scale volume. One mL of the dissolved sample solution was transferred to a fitted test tube, then mixed with 1 mL of the pre-configured phenol (5%, w/w) solution and 5 mL of sulfuric acid, and the mixture was shaken using Maxi Mix II mixer (Barnstead International, Dubuque, IA, USA). After the mixture was cooled at room temperature for 20 min, the sample was measured at 490 nm by using Colorimetric (BioMate™ 3 Spectrophotometer, Thermo Electron Co. Madison, WI, USA). Nitrogen (N) was measured via PerkinElmer 2400 Series II CHNS/O Elemental Analyzer (Shelton, CT, USA). Protein content was calculated with the formula of N% times a coefficient of 6.25 (Li et al., 2014, 2016).

2.4. Rheological properties

The Bohlin Model CVOR 150 rheometer (Bohlin Instruments, Southborough, MA, USA) was used to measure apparent viscosity, elastic modulus (G') and viscous modulus (G") as a function of shear rate. The cone and plate spindle with 40 mm diameter head and an angle of 4 ° were used for all rheological measurements with 20 μL silicone oil which has low viscosity compared to sample to ensure that the solution will not volatilize.

2.4.1. Apparent viscosity measurement

To prepare the gum solutions, spray-dried camelina gum was mixed with distilled water to reach the desired concentration gradient (0.1%, 0.5%, 1.0%, and 2.0%), and the solutions were then stirred with a Fisherbrand Magnetic Stirrers magnetic stirrer (Fair Lawn, NJ, USA) for 2 h until the solutions became homogenous. The apparent viscosity test with exponentially increasing in shear rate between 0.001-10 (1/s) was measured at 25 °C. Effect of temperature on the viscosity was determined by measuring the change in camelina gum viscosity by continuously change the temperature from 4 to 85 °C at a heating rate of 10 °C/min. Single shear rate at 0.1 (1/s) was selected for test. To investigate the effect of pH, the pH of camelina gum solution (2%) was adjusted by HCl and NaOH. Apparent viscosity was measured at the pH range from pH 1.0–12.0 with 1.0 pH unit gradient (only results at pH 3.0, 7.0, 10.0, and 12.0 were reported) at room temperature at a shear rate range of 0.001-10.0 (1/s). Effects of salts and sugar additive (0.5-10%) on apparent viscosity of camelina gum were studied at a shear rate range of 0.001-10 (1/s).

2.4.2. Frequency sweep

Oscillatory evaluations were performed using a Bohlin CVOR 150 rheometer (Malvern Instruments, Southborough, MA, USA). A thin layer of silicone oil with low viscosity was used to prevent water evaporation during measurement. Frequency sweeps tests were performed from 0.01 Hz to 10 Hz at 25 °C with 0.05% strain in order to be in the linear viscoelastic region. Three replicates were measured for each sample. The elastic modulus (G') and viscous modulus (G") were continuously registered. The viscoelastic properties of camelina gum at different pH were also exhibited including pH 3, 7, 10, and 12. The viscoelastic properties of the interaction of camelina gum with other substances by adding NaCl, $CaCl_2$, sucrose, and ethanol were also studied.

2.5. Scanning electron microscopy

For scanning electron microscopy (SEM) image, the thin layers of the camelina gum isolated at different drying conditions were prepared, and the gum was coated with an alloy of 60% gold and 40% palladium with a sputter coater (Desk II Sputter/Etch Unit, Moorestown, NJ, USA). The coated samples were viewed and photographed using a Hitachi S-3500N SEM (Hitachi Science system, Ibaraki, Japan) at an accelerating voltage of 5 kV, 10 kV, and 20 kV using magnification from 3 kV, and 18 kV to obtain the morphological properties.

2.6. Transmission electron microscopy

Transmission electron microscopy (TEM) measurement of camelina