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Adhesion properties of camelina protein fractions isolated with different methods

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ABSTRACT

The objective of this research was to investigate the effects of protein extraction methods on the adhesion performance of different camelina protein fractions. Physicochemical properties were also studied, including the electrophoresis profile; rheological, thermal, and morphological properties; and crystallization. Two camelina protein fractions, globulin and glutelins, were isolated from defatted camelina meal using three different methods resulting in total of six protein fractions including globulin 0–2 and glutelin 0–2. Dry adhesion strength of camelina protein adhesives exhibited nearly 100% wood cohesive failure at the curing temperatures of 150–190 °C, except glutelin 2 and globulin 0. The overall adhesion performance of globulin fraction behaved better than glutelin fraction. The greatest wet shear strength of globulin 1 and 2 was around 3.3 MPa, curing at 190 °C. The wet shear strength of glutelin 2 was inferior to glutelin 1 due to the negative effects of NaCl. Glutelin had higher protein aggregation than globulin, as indicated by higher crystallinity, higher thermal stability, and dense protein aggregation. The compact structure of glutelins may partially contribute to their lower adhesion strength.

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1. Introduction

Camelina, a member of the mustard family and a distant relative of canola, is known in North America primarily as a weed, but ancient European agriculturists called it “gold of pleasure.” Camelina monocultures were cultivated in the Rhine River Valley as early as 600 BC. The crop was widely grown in Eastern Europe and Russia until the early 1940s, with some production lasting into the 1950s. Today, camelina is produced in Slovenia, Ukraine, China, Finland, Germany, Austria, and the United States. Camelina contains 30-38% oil, and current interest in the crop is mostly focused on applications for its oil. Camelina has high levels of polyunsaturated fatty acids (90%), of which 38% are linoleic acids (18:3, omega-3) (Putnam et al., 1993), suggesting that camelina is a good candidate for making high-quality edible oils. Camelina oil also shows great potential as a source of biodiesel; camelina-derived synthetic fuel has been used to power a variety of military and commercial aircraft (Winchester et al., 2013). Increased interest in camelina oil-based biodiesel or fuel will trigger the spread of intentional planting of camelina, leading to greater availability of byproducts such as camelina meal, which is a co-product of the camelina oil extraction process.

Camelina meal typically contains 40% crude protein, a maximum of 12% crude fiber, less than 15% residual oil, and a small portion of vitamins (Sampath, 2009). Camelina meal is used mainly as an additive for animal feed as a protein or omega-3 supplement resource (Ryhanen et al., 2007; Rokka et al., 2002). Utilization of camelina protein as a value-added product is important if camelina is to be used for food and industrial applications such as adhesives. Previous study data have indicated that the amino acid profile of camelina protein is similar to canola protein, which has displayed potential as an alternative to conventional petroleum-based adhesives (Li et al., 2014, 2011a,b). Therefore, our hypothesis was that camelina protein may have adhesion properties comparable to canola protein. Soybean has become the most promising and most often investigated bio-based adhesive source in the last few decades (Qi et al., 2012; Qi and Sun, 2011), but using soybean this way reinforces the conflict between bio-based products and human food, so exploring alternatives to soy protein-based adhesives is desirable.

According to a previous report (Li et al., 2014), three protein fractions are extractable from camelina meal: albumin, globulin, and glutelin. The objective of this research was to study the adhesion performance of camelina protein fractions isolated by different methods. Albumin fraction was not studied because it is water-

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soluble and has strong swallowing properties, resulting in poor adhesion performance. Other investigated properties were protein electrophoresis profile; rheological, thermal and morphological properties; and crystallization.

2. Materials and methods

2.1. Materials

Camelina pellets with 15% lipid (db), 32.4% crude protein (db), and 11.0% moisture content (db) were provided by Montana Gluten Free Processors (Belgrade, Montana, US). The pellets were produced with a screw oil press at approximately 80 °C. Camelina meal (CM) with particle size <0.5 mm was obtained by milling the pellets using a cyclone sample mill (Udy Corp., Fort Collins, CO, USA). CM was then defatted with hexane at a solid/liquid ratio of 1:10 (w/v) for 2 h at room temperature in three cycles. The defatted camelina meal (DCM) was placed in a fume hood in a very thin layer (~2 mm) for 24 h to evaporate residual hexane. Hexanes, Bradford Assay, hydrochloric acid (HCl), and sodium hydroxide (NaOH) were purchased from Fisher Scientific (Fair Lawn, NJ, USA). Cherry wood veneers with dimensions of 50 × 127 × 5 mm (width × length × thickness) were provided by Veneer One (Oceanside, NY, USA).

2.2. Isolation of camelina protein fractions

Protein fractions were prepared with the method described by Li et al. (Li et al., 2014). Globulin 0–2 and glutelin 0–2 were isolated from DCM with sequences 0–2 (S0–S2), respectively. Globulin 0 and Glutelin 0 were isolated by S0 with 5% NaCl solution, and NaOH solution from two batches of DCM, separately. Glutelin 1 was isolated by S1 with NaOH solution. And globulin 1 were isolated by S1 with NaCl solution from the residue after glutelin 1 was extracted. Globulin 2 was isolated by S2 with NaCl solution at pH 8. And glutelin 2 was isolated by S2 with NaOH solution from the residue after globulin 2 was extracted. Fig. 1 describes the details of S0–S2.

2.3. Chemical analysis

Moisture content was measured using the V30Compact Volumetric KF Titrator (Columbus, Ohio). Nitrogen content was measured via a PerkinElmer 2400 Series II CHNS/O Elemental Analyzer (Shelton, CT, USA). Nitrogen was converted to protein using a factor of 6.25. All tests were performed with duplications.

2.4. SDS-PAGE gel electrophoresis

SDS-PAGE was performed on a 4% stacking gel and 12% separating gel with a discontinuous buffer system, as described by Laemmli (1970). A camelina protein sample was mixed with a buffer containing 2% SDS, 25% glycerol, and 0.01% bromphenol blue. To determine the disulfide bonds in camelina protein, SDS-PAGE was carried out under both reducing (2-mercaptoethonal) and non-reducing conditions. A total of 8 μg of protein was applied to sample wells. Molecular weight standards (14.4–97.4 kDa) were run with the samples. Electrophoresis was performed at 40 mA and 150 V for 120 min. The gel was stained in 0.25% Coomassie brilliant blue R-250 and destained in a solution containing 10% acetic acid and 40% methanol.

2.5. Scanning electron microscopy (SEM)

A Hitachi S-3500 N (Hitachi Science System, Ibaraki, Japan) SEM was used to observe the microstructure of powdered, dried camelina protein isolates. Ground protein powder was affixed to an aluminum stub with two-sided adhesive tape and coated with an alloy of 60% gold and 40% palladium with a sputter coater (Desk II Sputter/Etch Unit, Moorestown, NJ, USA). SEM images of protein isolates were performed at an accelerating voltage of 5 kV.

2.6. Rheological properties

Apparent viscosities of isolated camelina proteins were performed using a Bohlin CVOR 150 rheometer (Malvern Instruments, Southborough, MA, USA) with a CP 4/40 cone and plate fixture (4° cone angle, 40-mm cone diameter). Distance between the cone and plate was set to 150 μm for all measurements. Experiments were conducted under steady shear flow at 23 °C. Shear rates ranged from 10 s to 240 s^{-1} in increments of 10 s^{-1}. All experiments were done in duplicate, and average values were reported.

2.7. Differential scanning calorimetry (DSC)

Thermal transition properties of protein samples were measured with a DSC Q200 V24.4 instrument (TA Instruments, New Castle, DE, USA) calibrated with indium and zinc before making official measurements. Samples of dry camelina proteins weighing approximately 7–10 mg were measured in a hermetic aluminum pan under a nitrogen atmosphere with a gas flow rate of 50 mL/min. All samples were heated from 25 °C to 280 °C at a heating rate of 10 °C/min in an inert environment. All experiments were performed in duplicate.

2.8. Degree of crystallinity of camelina protein

Degree of crystallinity of dry camelina protein adhesives was studied with wide-angle X-ray diffraction (WAXD). WAXD experiments were carried out at the advanced polymers beamline (X27C), National Synchrotron Light Source, Brookhaven National Laboratory (Upton, NY, USA). Details of the experimental setup at the X27C beamline have been reported elsewhere (Chu and Hsiao, 2001). The wavelength used was 0.13714 nm. A 2D MAR-CCD X-ray detector (MAR USA Inc., Norwood, NJ, USA) was used for data collection. Data were collected from diffraction angle 5.0–35.0° (2θ). Crystallinity was estimated from an integrated diffraction intensity profile as the ratio of total crystal peak diffraction intensity to total diffraction intensity. A peak-fitting process was employed with Igor Pro 6.20 (WaveMetrics Inc. Lake Oswego, OR, USA). The d-spacing between crystal lattice planes was estimated with Bragg Eq. (1):

$$2d\sin\Theta = \lambda \quad (1)$$

where d is the space between crystal lattice planes, λ is the wavelength, and 2θ is the diffraction angle.

2.9. Wood specimen preparation

Cherry wood samples were preconditioned in a controlled-environment chamber (Model 518, Electro-tech systems Inc., Glenside, PA, USA) for 7 d at 25 °C and 50% relative humidity (RH). Camelina protein adhesives isolated with different methods were brushed separately along the edges of two pieces of cherry wood with an application area of 127 mm × 20 mm until the entire area was completely covered. The adhesive amount applied on each piece was approximately 0.06 g (dry basis). The brushing and setting procedure followed the method described by Mo et al. (2004). The brushed areas of the two pieces were assembled together at room temperature for 15 min, then pressed at 2.0 MPa at 150 °C, 170 °C, or 190 °C for 10 min using a hot press (Model 3890 Auto 'M', Carver Inc. Wabash, IN, USA).