Video Article

Preparation and Testing of Plant Seed Meal-based Wood Adhesives

Zhongqi He[1], Dorselyn C. Chapital[1]

[1]Southern Regional Research Center, Agricultural Research Service, United States Department of Agriculture

Correspondence to: Zhongqi He at Zhongqi.He@ars.usda.gov

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Abstract

Recently, the interest in plant seed meal-based products as wood adhesives has steadily increased, as these plant raw materials are considered renewable and environment-friendly. These natural products may serve as alternatives to petroleum-based adhesives to ease environmental and sustainability concerns. This work demonstrates the preparation and testing of the plant seed-based wood adhesives using cottonseed and soy meal as raw materials. In addition to untreated meals, water washed meals and protein isolates are prepared and tested. Adhesive slurries are prepared by mixing a freeze-dried meal product with deionized water (3:25 w/w) for 2 hr. Each adhesive preparation is applied to one end of 2 wood veneer strips using a brush. The tacky adhesive coated areas of the wood veneer strips are lapped and glued by hot-pressing. Adhesive strength is reported as the shear strength of the bonded wood specimen at break. Water resistance of the adhesives is measured by the change in shear strength of the bonded wood specimens at break after water soaking. This protocol allows one to assess plant seed-based agricultural products as suitable candidates for substitution of synthetic-based wood adhesives. Adjustments to the adhesive formulation with or without additives and bonding conditions could optimize their adhesive properties for various practical applications.

Video Link

The video component of this article can be found at https://www.jove.com/video/52557/

Introduction

Adhesive bonding of wood plays an increasing role in the forest product industry and is a key factor for efficiently utilizing timber resources[1]. Interest in the use of natural product-based adhesives for wood increased steadily from the 1930s to reach a peak around 1960[2]. After this period, the price of petroleum-based adhesives became so low that they displaced protein adhesives from several traditional markets. In the past two decades, this trend has reversed with renewed interest in the use of materials that are renewable, biodegradable, and more environmentally acceptable. These natural resources include, but are not limited to, soy protein[3-5], cottonseed protein[6], rice bran[7], wheat gluten[8], distillers grain protein[9], canola protein and oil[10-12], lignin from sorghum and sugar cane bagasse[13,14], and polysaccharides derived from shrimp shells[15].

Whereas seed protein isolates have been widely evaluated as potential wood adhesives, the isolation procedure involves corrosive alkaline and acidic reagents and it makes isolate-based adhesives relatively expensive and less environment-friendly[16]. Thus, some defatted seed meals (flours) with or without treatment have also been tested for the adhesive purpose, even though the adhesive properties of these meals do not perform as well as protein isolates[17-19]. We have sequentially fractionated cottonseed meal (CM) into different fractions, and examined their adhesive strength in bonding wood veneers[20,21]. The water-insoluble solid fraction (hereinafter washed cottonseed meal-WCM) could be used as wood adhesives, comparable to cottonseed protein isolate (CSPI), and would be less costly to prepare than CSPI.

Adhesive strength and water resistance are two critical parameters in evaluating the performance of a potential adhesive material. Here, the adhesive strength is reported as the shear strength at break of the lap bond of each wood specimen. Water resistance of the adhesive is measured by the change in lap shear strength of the bonded wood specimen at break due to water soaking. Using defatted cottonseed and soy meals as raw materials, this protocol provides a simple and straightforward way to prepare and test plant seed-based products as wood adhesives. This protocol would be helpful in facilitating the effort in seeking more economic and environment-friendly formulations of natural product-based wood adhesives.

Protocol

1. Cottonseed and Soy Meal-based Products (Figure 1)

1. Obtain the raw materials, defatted cottonseed and soy meals, from commercially available sources.
2. Obtain the working meal by grinding the solid defatted meal in a cyclone sample mill to pass a 0.5 mm steel screen[16].
3. Prepare water washed meals from the working meals after water extraction (25 g meal:200 ml water) to separate water soluble components in the meals[21].

4. Prepare protein isolates from the working meals by alkali extraction and acid precipitation[16].

2. Preparation of Wood Veneer Strips

1. Cut wood veneers (1.59 mm thick) obtained from a commercially available source into strips 25.4 mm wide by 88.9 mm long.
2. Pencil mark a line across the wood grain at 25.4 mm (1.0") length from one end of each strip. Label these strips appropriately with testing treatments or numbers. 5 - 10 wood pairs are prepared for each testing variable.

3. Preparation of Adhesive Slurries

1. Calculate the amount of water washed meal needed per the wood specimens for testing, by application rate (*e.g.,* 4 mg dry content cm^{-2}) x total bonding area (*e.g.,* 581 cm^2 of 90 wood strips with 2.54 x 2.54 cm bonding area each) plus about 30% extra for enoughness (*i.e.,* 4 x 581 x 130% 3 g of water washed meal for the example).
2. Mix water washed meal with deionized water (3:25 w/w), and stir with a magnetic stir bar for 2 hr in a beaker sealed with Parafilm.

4. Preparation of Bonded Wood Specimens

1. Brush adhesive slurry onto one end of 2 wood veneer strips covering 25.4 mm (1.0") length. Air-dry for 10 - 15 min or until tacky.
2. Brush a second layer of adhesive slurry on top of the first layer and air-dry again. The amount of dry adhesive preparation applied is about 4.5 mg dry solid per cm^2 of bonding are of each wood strip.
3. Overlap the tacky adhesive coated area (25.4 x 25.4 mm or 1.0" x 1.0") of 2 wood veneer strips. Hot-press using a Benchtop Heated Press at 100 °C for 20 min at a pressure of 400 psi (2.8 MPa). Note the pressure is the force applied by the press divided by the overlapped area of the wood samples. These bonding parameters may be changed as needed for each testing variable.
4. Cool and condition the bonded wood specimens for 48 hr in a conditioning room or an incubator with humidity control (temperature of 22 - 23 °C and relative humidity of 50 - 60%; **Figure 2**).

5. Water Resistance Experiments

1. Immerse the bonded wood specimens, after initial conditioning, in tap water for 48 hr in a plastic tray at RT (22 - 23 °C). The wet specimens after soaking are tested immediately for the shear strength at break and reported as wet strength. Excess water on the veneer surface may be removed by gently patting with paper tissue prior to measurements.
2. Immerse another set of bonded wood specimens, after initial conditioning, in a water bath at 63 ºC for 4 hr, then dry at room conditions (temperature of 22 - 23 °C and relative humidity of 50 - 60%) O/N (18 - 20 hr). Repeat the immersion-drying cycle once with a 48 hr drying time. The dried specimens are then tested for the shear strength at break and reported as soaked adhesive strength.

6. Lap Shear Strength Measurements

1. Fit a bonded wood specimen into the 32 x 40 mm fishscale gridded wedge grips on a Materials Tester with a gripping pressure of 7 MPa, and set the crosshead speed at 1 mm min^{-1}.
2. Measure and record the shear strength at break for each bonded wood specimen. The results of multiple measurements are averaged for each adhesive formulation and test variable.

Representative Results

Each adhesive formulation's performance is determined by the shear strength of the bonded wood specimen at break and the values vary depending on the dimensions of the wood veneer used. For example, in **Table 1**, the dry and soaked adhesive strength values of the bonded specimens are lower when thinner and narrower maple strips are used (see Cottonseed-1), as opposed to the thicker and wider strips of Cottonseed-2 recommended in the protocol, using the same cottonseed-based adhesive formulation. Also observed were more wood failure specimens during adhesive shear strength measurements of thin and narrow wood veneers. Specifically, 3 of the defatted meal, 4 of the washed meal, and all 10 of the protein isolate failed in the wood grain rather than at the adhesive joint in dry bonded specimens, and when using the same three adhesive formulations, respectively, 0, 6 and 9 of the soaked specimens failed in the wood grain. This indicates that the adhesive is stronger than the thin wood strips[21]. A general observation seems applicable to both raw materials used. That is, the adhesive performance of water washed cottonseed meal is comparable to that of cottonseed protein isolate. On the other hand, for soy products, both dry and soaked strengths of the water washed meal are similar to those of defatted meal than to those of protein isolate, which may reflect the difference in chemical composition of cottonseed meal and soy meal.

Table 2 compared the shear strength of dry, wet, and soaked specimens bonded at 100 °C using water washed cottonseed meal and four wood types. The shear strength is in the order: dry > soaked > wet for all four types of wood, indicating the same trend that water weakens the bond strength of these wood specimens, and part of the adhesive bond strength is recovered after drying. The dry shear strength of poplar, Douglas fir, and white oak are basically same, but the dry strength is lower with walnut. The small difference makes the impact of wood type on the dry adhesive strength only significant at $P = 0.1$. The impact of the wood type is more statistically significant on the wet and soaked shear strength data with $P < 0.001$. In actuality, the order of the wet and soaked strength of the bonded specimens for the 4 woods is not same as that of the dry strength. We attribute this observation to the difference in the degree of expansion (swelling) of each type of wood during soaking; the expansion rate of the wood veneer may become incompatible with the adhesive and could exert certain stresses to lower the bond joint's adhesive strength.

Sun and Bian[22] proposed that wood types with higher linear or bulk volume expansion would have higher shrinkage stress during drying, which partially explains the higher delamination rates of maple and poplar pairs than walnut and pine during their water-soaking tests.

Figure 1. Seed meal based materials. Top - cottonseed, bottom - soy. From left to right: defatted meal, working meal, water washed meal and protein isolate. Please click here to view a larger version of this figure.

Figure 2. Bonded wood specimens set aside for conditioning (temperature of 22 - 23 °C and relative humidity of 50 - 60%). Left 5, poplar; Right 5, walnut. The bonded area (25.4 x 25.4 mm or 1.0" x 1.0") is shown between the red lines at the most left pair. Please click here to view a larger version of this figure.

Adhesive	Dry strength	Soaked strength
Cottonseed-1 [‡]:		
Defatted meal	1.49 ± 0.14 a	1.37 ± 0.17 a
Water washed meal	1.55 ± 0.11 a	1.55 ± 0.15 b
Protein isolate	1.53 ± 0.18 a	1.53 ± 0.14 b
Cottonseed-2 [$]:		
Defatted meal	ND [#]	ND [#]
Water washed meal	3.26 ± 0.50 a	2.38 ± 0.51 a
Protein isolate	3.69 ± 1.13 a	2.39 ± 0.61 a
Soy bean [$]:		
Defatted meal	2.40 ± 0.50 a	1.25 ± 0.19 a
Water washed meal	2.29 ± 0.39 a	1.60 ± 0.37 a
Protein isolate	3.51 ± 0.33 b	3.76 ± 0.90 b

[‡] Adhesives were applied to thinner and narrower wood strips (0.99 mm thick x 12.7 mm wide x 25.4 mm long).

[$] Adhesives were applied to thicker and wider wood strips as described in the protocol (1.59 mm thick x 25.4 mm wide x 25.4 mm long).

[#] Not determined.

Table 1. Shear strength (MPa) of dry and soaked maple wood strips bonded at 100 °C with defatted meal, water washed meal, and protein isolate of cottonseed and soy. Data are presented in the format of average ± standard deviation (n = 4, 5, 7, or 10). Different letters in the same seed series represent the significant difference in dry or soaked strength at *P* = 0.05. The data analysis package in Microsoft Excel 2007 was used for statistical analysis.

Wood	Dry strength	Wet strength
Poplar	4.52 ± 0.54	1.73 ± 0.20

Douglas fir	4.30 ± 0.96	2.24 ± 0.14
Walnut	3.59 ± 0.23	1.78 ± 0.10
White oak	4.33 ± 0.32	1.66 ± 0.25
Significance level (P>F)	0.1	<0.001

Table 2. Shear strength (MPa) of dry, wet, and soaked poplar, Douglas fir, walnut, and White oak wood strips bonded at 100 °C with water washed cottonseed meal. Data are presented in the format of average ± standard deviation (n = 5). The data analysis package in Microsoft Excel 2007 was used for statistical analysis.

Discussion

This paper presents a basic procedure to prepare and test plant seed-based products as wood adhesives. The adhesive slurries exampled in this protocol are simply the defatted seed meal product and water. Various adhesive formulations can be reached by addition of testing reagents (such as sodium dodecyl sulfate, sodium bisulfite or tung oil)[5,6,23] and/or changes in mixing conditions (such as pH, ratio of solid and water)[3,24,25]. Adjustment of the adhesive formulation is also needed if the rheological properties of the adhesive slurry are not suitable for appropriate application to the wood strips.

The solid-surface test materials, wood veneers, are natural products so that one can expect high variation of wood textures and surface roughness. For this reason, testing replicates from 3 - 10 have been reported in literature. Due to these variation and other known and unknown factors, it is not uncommon to see large standard deviations (>10%) observed in the shear strength measurements, as in **Table 1** and the literature[6-8,12,25], and this may undermine some statistical analysis at $P \leq 0.05$. Thus, some papers simply present the data with standard deviations, then compare and discuss them without statistical significance analysis (*e.g.,* [7,8,12,26]). This approach still makes some sense by showing general trends of influence from test variables.

It should be noted that the measurement of shear strength is also sensitive to the sample dimensions and the numerical results cannot be compared between different geometries. The higher values of shear strength of Cottonseed-2 than Cottonseed-1 in **Table 1** are apparently due to thicker and wider wood specimens used for Cottonseed-2. It is reported that the strength of a lap-shear joint can vary with the total length of the specimen even for a fixed overlap length[27]. Thus, the comparison can only be made between samples in the same set of tests, not between different test geometries, such as between Cottonseed-2 and soybean (**Table 1**). More information on the effects of geometry and material properties on the fracture of single lap-shear joints can be found in Kafkalidis and Thouless[27].

The shear strength was tested in reference to the American Society For Testing and Materials (ASTM) Standard Method D-906[22]. This protocol presents two common methods used for evaluation of the water resistance: (1) wet strength- the shear strength of the bonded specimens measured immediately after soaking in tap water at 23 °C for 48 hr, which was based on ASTM Standard D1151-00 [11]; and (2) soaked strength- the shear strength of the bonded specimens measured after soaking - drying cycles, which was similar to the Chinese National Standard for Plywood (GB/T 17657-1999 ASTM Standard D1151-00 [11]. Some papers report wet strength only[5], or soaked strength only[6], or both[11]. It is also worth pointing out that the soaked strength in this protocol is measured after two cycles of soaking at 63 °C for 4 hr and drying at RT O/N (18 - 20 hr)[6]. Some researchers measure soaked strength after one longer soaking and drying process at RT (*i.e.,* 48 hr soaking and 2- to 7-days drying both at 23 °C)[11,25]. In our opinion, one can choose either method based on their experimental time availability and their project goals.

In this work, we tested the adhesive strength with the single two-layer joint specimens. Although this approach is most widely used (*e.g.,* [4,6,9,11]), more complicated or multiple overlapped wood specimens have also be used in the adhesive tests (*e.g.,* two 2-layer joints with three wood strips[7,22], and three 3-layer joints with three wood strips)[29].

Disclosures

The authors have nothing to disclose.

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