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Short communication

Coating applications to natural fiber composites to improve their physical, surface and water absorption characters *

Zengshe Liu^{a,*}, Brent H. Tisserat^b

^a USDA, ARS, National Center for Agricultural Utilization Research, Bio-Oils Research Unit, 1815 N. University St, Peoria, IL, 61604, United States ^b USDA, ARS, National Center for Agricultural Utilization Research, Functional Food Research Unit, 1815 N. University St, Peoria, IL, 61604, United States

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ABSTRACT

Natural (organic) fibers are used in reinforced composites and natural fiber composites (NFCs). These fibers have advantages over synthetic fiber composites such as high mechanical properties, lower densities and biodegradability. However, one major disadvantage of NFCs is their hydrophilicity. In this study, we report a method to prepare dried distillers grains with solubles (DDGS)-flax composites which can be subsequently coated with acrylated epoxidized soybean oil (AESO) and polymerized by UV light with Irgacure^{*}819 initiator. The physical, mechanical and water resistance properties of coated DDGS-flax composites were compared to the untreated samples. Results showed that water resistance property was improved by AESO coating. The surface roughness, color and mechanical properties of coated DDGS-flax composites only slightly differed from untreated composites.

1. Introduction

Natural (organic) fibers are employed in natural fiber composites (NFCs). NFCs compete with synthetic composites which employ inorganic fibers (e.g., glass fibers) (Faruk et al., 2012; Pickering et al., 2016; Liu and Dai, 2007; Marshall et al., 2007; Motsanos, 2009; Yu et al., 2009). Natural fibers have advantages over glass fibers because they cost less, are biodegradable and weigh less (George et al., 1999; Faruk et al., 2012; Pickering et al., 2016; Liu and Dai, 2007; Marshall et al., 2007; Motsanos, 2009; Yu et al., 2009). NFCs usually consist of a biological or natural fiber portion (e.g., ramie, flax, hemp, jute, cotton) which provides the reinforcement fraction and a thermoplastic matrix portion (e.g., polyethylene (PE) or polypropylene (PP)). The use of natural fibers in the composites shows the advantages due to their relatively high strength, stiffness and low density. They are also renewable, biodegradable, less abrasive to tooling and less irritating to the skin and respiratory systems of people processing them. The properties of natural fibers vary considerably. Chemical composition and structure of the fiber greatly influence the mechanical properties of natural fibers. Generally the high performance of the biological fiber is related to high cellulose content with cellulose microfibrils aligned in the fiber direction which tends to occur in the bast fibers (e.g., flax, hemp, kenaf, jute, ramie) (Pickering et al., 2016). The matrix portion in the natural fiber composite, aside from being the binding agent, also provides a barrier against adverse environments, protects the surface fibers from mechanical abrasion and transfers the load to the fibers (Pickering et al., 2016). Most matrix materials in NFCs are synthetics derived from petroleum products such as thermoplastics (*e.g.*, PP, PE, polyolefin, polyvinyl chloride) or thermosets (polyesters, epoxy resins, phenol formaldehyde) (Faruk et al., 2012; Pickering et al., 2016). Interfacial bonding between the fiber and matrix is responsible for the strength or mechanical properties of the composite (Pickering et al., 2016).

Non-woven mats are employed in NFCs because of their low cost and ease of manufacture (Realhemp, 2016). However, they have limited geometry (i.e., random orientation) and packing arrangements (Ratim et al., 2012). Efforts to utilize non-woven mats often consist of permeating a mat with a plastic material usually through heated compression molding (Englund et al., 2004; George et al., 1999). In this study, we substituted a biological matrix for the traditional synthetic matrix to create an entirely bio-based NFC. The biobased matrix employed was generated from dried distiller's grains with solubles (DDGS). DDGS were applied to flax mats via a slurry (a watery mixture of insoluble matter) by immersion under vacuum. Following this treatment, mats were hot pressed to create the final NFC.

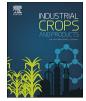
Even NFCs created with petroleum resins swell because of water uptake that leads to microcracking of the composites and degradation

* Corresponding author.

E-mail address: kevin.liu@ars.usda.gov (Z. Liu).

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of mechanical properties. This situation ultimately causes poor dimensional stability. These problems have been addressed by researchers (De Bruijin, 2000; Karmaker and Youngqist, 1996). Similarly, the NFC mats created with DDGS-flax ingredients are even more hydrophilic in nature. To address this situation, we sought to develop a bio-based, acrylated epoxdized soybean oil (AESO) coating that provides hydrophobic properties to the DDGS-flax mats. Coated DDGS-flax mats were exposed to UV light and polymerization with an Irgacure *819 photoinitiator. The mechanical and water resistance properties of coated DDGS-flax composites were investigated and reported herein.

2. Materials and methods

2.1. Materials

AESO was purchased from Sigma-Aldrich (Milwaukee, WI). Irgacure^{*}819 was obtained from BASF Company (Vandalia, IL). Absolute 200 proof ethanol (Decon Laboratories, Inc., King of Prussia, PA) was used without further purification. Inhibitor remover column for removing hydroquinone and monomethyl ether hydroquinone was purchased from Sigma-Aldrich (Milwaukee, WI).

2.2. AESO solution preparation and photo-polymerization

AESO with 4000 ppm monomethyl ether hydroquinone inhibitor was removed by the column before use. 10 g AESO in 100 ml ethanol was stirred magnetically until AESO dissolved in the ethanol solution and 0.05 g Irgacure *819 was added. The solution was stored for coating application. Photo-polymerization was accomplished using unfiltered radiation from a 450 W Hanovia mercury lamp (55.7 W or 7.7 × 1019 photons/s, at λ < 325 nm) (Ace Glass Inc., Vineland, NJ). The lamp was placed in a water-cooled (18 °C) immersion jacket, and the coated samples to be irradiated were placed in the chamber.

2.3. Mat preparations

DDGS obtained from an ethanol plant located in Central Illinois (Archers Daniel Midland Co., Decatur, IL) was ground in a Wiley mill (Model 4, Thomas Scientific, Swedesboro, NJ) through a 2 mm screen mesh. Ground DDGS were defatted using hexane in a Soxhlet extractor. Following solvent extraction, DDGS were finely ground in a ball-mill (Model 801 CVM, U.S. Stoneware, East Palestine, OH) using milling jars and grinding pellets. DDGS were subsequently screened through a #80 screen producing \geq 177 µm particles. Defatted DDGS flour contained \sim 27% protein.

A slurry was prepared consisting of 15 g of DDGS and 200 ml of distilled water stirred for 30 min. Flax (Linum usitatissimum L. family (450 g m^{-2}) Linaceae.) non-woven mats were cut into 17.8 cm L \times 7.6 cm W pieces (HempFlax BV, Oude Pekela, Netherlands). Mats were submerged using plastic sheets containing predrilled holes into the slurry in an aluminum tray within a Pyrex vacuum chamber (24 cm L \times 19 cm W \times 7 cm H \times 2600 mm³ cap.) (Best Value Vacs, Naperville, IL) fitted with an aluminum drip pan $(21 \text{ cm L} \times 13 \text{ cm W} \times 3 \text{ cm H} \times 600 \text{ mm}^3 \text{ cap.})$ (Webber-Stephne, Palatine, IL). Mats were subjected to a vacuum at -29 in Hg (-9.8 bar) for 30 min. Mats were removed from the slurry and the excess solution was allowed to drain away for 10 min.

Mats were transferred into an aluminum mold consisting of a frame (20.3 cm L \times 10.2 cm W \times 1.9 cm H, O.D.); cavity (17.8 cm L \times 7.6 cm W \times 1.9 cm H, I.D.) and attached to a base plate (15.2 cm W \times 22.2 cm L \times 1.9 cm H via 6 pre-drilled screws). An aluminum plunger (17.8 cm L \times 7.6 cm W \times 1.9 cm H) was employed to press the mat. Molds containing the mats were placed in a 185 °C preheated Carver press. Molds were initially pressed to 5 mton and held for 1 min, and pressure was released to aid in the removal of trapped air in the mats. Molds were further pressed 7.5 mton and held for 1 min and

pressure released. Subsequently, the mold was pressed to 10 mton and held for an additional 2.5 min and pressure released. The total heating/ compression time was 4.5 min. Molds were removed from the press and allowed to cool to room temperature prior to opening.

2.4. Spectrophotometric properties

Color measurements were made on the tensile bars using the Commission International de l'Eclairage (CIE) Lab (Vienna, Austria) parameters (L*, a*, b*) with a Chroma Meter CR-400 spectrophoto-colorimeter (Konica Minolta, Ramsey, NJ). The colorimeter was calibrated with a white tile. Color values obtained were lightness or brightness (L*, ranging from 0 (black) to 100 (white)); redness (a*, ranging from –100 (green) to +100 (red)); yellowness (b*, ranging from –100 (blue) to +100 (yellow); Chromaticity (C*_{ab}, quality of color); and Hue angle (H*_{ab}, the real color). Values C*_{ab} and H*_{ab} values were obtained from the formulas: $\sqrt{(a^{*2} + b^{*2})}$ and arctan (b*/ a*), respectively.

2.5. Measuring surface properties

The surface properties of the composite panels were determined with a portable Surface Roughness Measuring Tester (Model SJ-210, Mitutoyo Corp., Kanagawa, Japan) equipped with a stylus profile detector. Three roughness parameters: average roughness (R_a), mean peak-to-valley height (R_x) and maximum roughness (or maximum peak-to-valley height) (R_y) were characterized by ISO 4287:1997 and were used to evaluate the surface properties of the composite panels. Each panel was subjected to five surface roughness readings. Specifications for the tester were set at: speed, 0.5 mm/s; pin diameter, 10 µm; and pin angle, 90°. The length of tracing line (L_t) was 12.5 mm and cut-off (λ_x) was 2.5 µm. Measuring force of the scanning arm was 4 mN (0.4 gf). Measurements were conducted at 25 ± 2 °C and the detector was calibrated prior to testing.

2.6. Mechanical property measurements

Specimen bars were obtained by cutting the dried processed mats using a clipper press mounting a mold that cut specimen samples of 6.4 cm L \times 1.27 cm W $\times \sim$ 0.2 cm thickness. These specimen samples were used for both the mechanical test (ASTM D638) and flexural test (ASTM-D79). Samples were conditioned for approximately 240 h at standard room temperature and humidity (23 °C and 50% RH) prior to any test evaluations. Molded specimens were given a modified ASTM D638 test to obtain the Young's modulus (YM), tensile strength (TS) and elongation at break (%El) using a universal testing machine, Instron Model 1122 (Instron Corporation, Norwood, MA). The speed of testing was 5 mm/min.

2.7. Statistical analysis

Five replicates were conducted for each test administered and their average and standard errors reported. Significant differences among the mats were determined according to ANOVA statistical analysis (P < 0.05). The Duncan multiple range test was employed to determine significant differences between the average values of the groups.

2.8. Water absorption

The specimen dimensions for water uptake experiments were 10 mm L \times 10 mm W \times 1 mm H. The composite samples were thin enough so that the molecular diffusion was considered to be one dimensional. A minimum of three samples were tested for each material.

Samples were first dried overnight at 70 $^\circ C$ and then cooled to room temperature and immersed in distilled water at 25 $^\circ$ C. The samples were

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