Decorative Materials from Rice Straw and Cornstarch Adhesives

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Abstract

The main goal of this work was to use rice straw fiber as a filler in the production of environmentally sound decorative materials using cornstarch adhesives as matrix. Treatments of rice straw with NaOH, oxalic acid, and hot water were undertaken to evaluate the effect of such treatments on the characteristics of rice straw and the performance of produced materials. The influence of temperature and shear rate on apparent viscosity of cornstarch adhesives was also investigated. Results showed that all treatments were efficient in partially modifying rice straw surface properties, as evidenced by Fourier transform infrared spectroscopy, and improving wettability of rice straw. Scanning electron microscopy suggested that NaOH and hot-water treatments resulted in a more significantly changed rice straw substrate than oxalic acid treatments. Apparent viscosity of the cornstarch adhesive decreased with increasing shear rate and increased and then decreased with increasing temperature, reaching a peak value at 10°C. The dependence of physical–mechanical properties of obtained materials on treatments performed on rice straw was also studied. Hot-water–treated straw materials displayed the best set of final mechanical properties. The materials exhibited poor waterproof performance but considerable moisture resistance and environmental friendliness, and such materials could be used for indoor decorative materials.

As the global demand for petroleum-based plastics continues to rise, environmental concerns and elevating crude oil prices have triggered a search for replacements for these nonbiodegradable plastics. The use of biorenewable resources for the production of biopolymers has gained a large amount of interest over the past decade because of their low cost and ready availability (Andjelkovic et al. 2005, Li and Larock 2005). Among them, agricultural residues are emerging as a source of raw materials that provide a renewable and environmentally friendly alternative to biomass resources, thus easing the high demand for woody materials (Sampathrajan et al. 1992). Besides their abundance and renewability, agricultural residues have advantages for the world economy, environment, and technology (Çöpür et al. 2007). Straw and starch are both rich in resources, and their composites represent low density and biodegradable characteristics that other composites cannot match (Reddy and Yang 2007, Zhou and Zhang 2007). Using straw and other agricultural residues for preparation of composite materials has become a focus of the material field. Such composite materials have lower water absorption, better acoustic insulation properties, and higher internal bond strength, flexibility, and flexural strength. However, discarded tires, formaldehyde-based resins, and polymers are the main matrix (Bledzki et al. 2010, Kuang et al. 2010), while crushed biologically based materials (e.g., straw powder, rice husk powder, husk powder, or extracted straw fiber) are the main reinforce-

ments for preparation of these composites (Yang et al. 2004, Alemdar and Sain 2008, Halvarsson et al. 2008, Jiang et al. 2009, Pan et al. 2010). Smashing agricultural residues and extracting fiber from straw does waste a lot of energy, and the matrix of such composites is difficult to degrade, and some of these residues will release formaldehyde.

Formaldehyde-based adhesives such as urea (UF), melamine (MF), and phenolic (PF) resins dominate the current wood adhesive market. Despite the well-known advantages of such resins, formaldehyde emissions and their nonrenewable nature have become a matter of increasing concern. Therefore, environmentally friendly adhesives from renewable resources and free from formaldehyde have been developed to replace the UF, MF, and PF binders. Biologically based adhesives (i.e., soybean protein or starch-based adhesives) can be used as environmentally friendly adhesive substitutes for the traditional synthetic

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Forest Prod. J. 62(2):150-156.

adhesives in composite or particleboard manufacturing (Khosravi et al. 2010, 2011; Kong et al. 2011; Liu et al. 2011). However, the bonding capacity and stability of starch-based adhesives are usually too low for practical use. Therefore, some measures were undertaken to improve performance of starch-based adhesives (Tan et al. 2011; Wang et al. 2011, 2012).

China is the largest rice producer in the world. Rice straw has a hard surface and a low-packing density. Current landfill practices and the incineration of rice straw tend to destroy the local environment. Currently, rice straw is mostly used to make carbon energy and is seldom used to make composites. Because energy savings and environmental protection are in such urgent need, this work focused on preparing cornstarch-based decorative materials using rice straw as a natural fiber filler by a compression molding process. Treatments of rice straw with NaOH, oxalic acid, and hot water were undertaken to evaluate the effect of such treatments on the characteristics of rice straw. Surface chemistry and morphology of the rice straw were studied by Fourier transform infrared spectroscopy (FTIR) and scanning electron microscopy (SEM), respectively. The influence of temperature and shear rate on apparent viscosity of cornstarch adhesive was investigated. The effect of treatments on physical-mechanical properties, modulus of rupture (MOR), modulus of elasticity (MOE), flexural strength (FS), impact strength (IS), tensile strength (TS), internal bond (IB), water absorption (WA), moisture absorption (MA), and 2-hour thickness swelling (2 hTS) of the resulting materials, was also investigated.

Experimental

Materials

Rice straw was from Luhe, Nanjing (China), moisture content was 8.09 percent. After smashing, RS was 1 to 30 mm in length. Cornstarch was food grade and provided by Jincheng Food Co., Ltd. (Shandong, China).

Methods

Rice straw treatments.—Rice straw was extensively washed with distilled water in order to remove impurities (mainly dust). This operation was performed several times at room temperature and under vigorous stirring. After successive washings, rice straw was dried in an aircirculated oven at 103°C. This material was stored in hermetic plastic containers in order to prevent microbial attack (i.e., fungi) before using it in the following treatments. Washed rice straw without any further treatments was used as a control and was labeled CRS.

Some components of cellulose fibers represent a hydrophobic blockage for fiber wetting, and they had to be efficiently removed (Leiva et al. 2007, Ndazi et al. 2007, Wang et al. 2007). Rice straw is rich in silica and waxes, deteriorating the properties and making it unsuitable for textile applications (Ndazi et al. 2007, Salam et al. 2007, Wójciak et al. 2007). In order to improve rice straw's wettability and performance, different treatments were applied. CRS was soaked in 2 percent NaOH solution for 2 hours at room temperature with occasional shaking, followed by washing with distilled water several times to leach out the absorbed NaOH until neutral pH (7 ± 0.5) was reached, and subsequently was oven dried. The NaOH-treated CRS was labeled NRS.

CRS was soaked in 2 percent oxalic acid solution for 2 hours at room temperature with occasional shaking. Afterward, the oxalic acid-treated rice straw was washed with abundant distilled water to leach out the absorbed oxalic acid until neutral pH was reached, and subsequently was oven dried. The oxalic acid-treated CRS was labeled ORS.

CRS was soaked in hot water for 2 hours at 100°C followed by oven drying. The hot-water-treated CRS was labeled HRS. Table 1 shows the three treatments of rice straw.

Preparation of cornstarch adhesives.—Cornstarch adhesives were prepared by the dispersion of the cornstarch powder into distilled water at a cornstarch-to-water ratio of 1:10 while stirring at room temperature for 2 hours with 1.2 percent by weight sodium thiosulfate $(Na_2S_2O_3)$ on cornstarch to prevent decomposition and aging. The resulting adhesives were then ready to be mixed with rice straw.

Preparation of rice straw/starch decorative materials.— The materials were prepared using a hot compression molding process. Treated and untreated CRS were blended with the laboratory-made cornstarch-based adhesives (10 wt% solids) in an orbital paddle mixer for 10 minutes at room temperature (rice straw loading contents of 55 wt%). The equilibrated mixtures were subsequently hot pressed into materials in a 10 by 10-cm steel mould equipped with stops to achieve the same thickness (0.45 cm). The press time, pressure, and temperature were 30 minutes, 4 MPa, and 120°C, respectively. Figure 1 shows the flowchart of the prepared materials.

FTIR of rice straw.—A Nicolet iS-10 FTIR (Thermo Fisher Scientific, USA) was used to obtain spectra for the treated and untreated rice straw. FTIR spectra were recorded in a range from 4,000 to 400 cm⁻¹ at a resolution of 4 cm⁻¹ with 32 scans.

SEM of rice straw.—The effect of treatments on the outer surface of rice straw was analyzed by SEM, using a JSM 6300 microscope (Japan) at an acceleration voltage of 20 kV. Microphotographs were taken on gold-coated surface of the specimens.

Contact angle of rice straw surface.—The contact angle was measured to check the water repellent property of rice straw, using a jc2000D contact angle instrument (Jinchen, China). The temperature during measurements was 23° C \pm 1°C and the relative humidity was 55 ± 3 percent.

Apparent viscosity of cornstarch adhesives.—The viscosity of the corn starch–based adhesives was measured with a Yueping NDJ-5S viscosimeter (Shanghai, China). The adhesives were prepared as described above and were transferred into the sample holder of the viscometer in an electric-heated thermostatic water bath. The viscometer rotor was immersed in the adhesives. The electric-heated thermostatic water bath controlled the temperature, and the

Table 1.—Treatments of rice straw.

	Treatments			
	NaOH	Oxalic acid	Hot water	Untreated
Conditions	2% concentration solution, 2 h room temperature		2 h, 100°C	
Label	NRS	ORS	HRS	CRS



Figure 1.—Flowchart of composites prepared.

control panel on the viscometer controlled the rotor speed, thus controlling shear rate. The viscosity values were averaged over three measurements.

Physical-mechanical properties of decorative materials.—FS, MOE, IB, TS, and MOR of the obtained materials were determined in a TMS-Pro test machine (Food Technology Corp., USA). IS was determined in a Charpy impact test machine, Jinjian XJJ-5 (Chengde, China). All samples were conditioned at 65 percent relative humidity at 20°C and for 7 days before testing. MA, WA, and 2 hTS were also measured. The weight and thickness were measured immediately after soaking. The final values were the average of five measurements.

Results and Discussion

Effect of treatments on FTIR of rice straw

Rice straw had the same basic components as wood but in different proportions, depending on the rice variety. Therefore, it would be expected that rice straw should behave similarly to wood in composites production. Rice straw had lower cellulose and lignin content than wood, but higher amounts of silica, which may reduce the interactions with adhesives such as cornstarch-based adhesives (Leiva et al. 2007, Ndazi et al. 2007). Treatments on rice straw were undertaken in order to upgrade rice straw wettability and surface properties. The effectiveness of each treatment was evaluated by examining their influence on rice straw's relative FTIR, surface morphology, and contact angle, as well as on the performance of the obtained composites.

Figure 2 shows the FTIR spectrum of CRS, HRS, NRS, and ORS. Observation of the absorption bands showed that the changes between treated and untreated rice straw were mainly due to the formation of oxygen-containing functional groups (in the form of carboxylic, ester, and phenolic

groups), which were associated with the 1,800 to 700 cm⁻¹ range. The band centered at 1,730 cm⁻¹ was usually ascribed to the stretching vibration of C=O in ketones, aldehydes, ester, and carboxyl groups (Kaushik et al. 2010). The band around 1,620 cm⁻¹ was attributed to COO asymmetric stretching. The broad adsorption band in the 1,300 to 1,000 cm⁻¹ range could be assigned to various C bonds (Si-C, C-O, C-O-C, and C-N), such as those in ethers, phenols, and hydroxyl groups (Mascarenhas et al. 2000). These results indicated that all treatments gave rise to a large increase in carboxylic and ester group C=O bonds.

The changes in the amount of hemicelluloses were seen by changes in the absorbance band approximately at 1,640 cm⁻¹, which was associated with adsorbed water after treatments (Sun et al. 2000). The treated rice straw had most likely absorbed moisture during storage because it was not kept in a moisture-free environment. This band showed a decrease after NaOH and hot-water treatments. The absorption band at approximately 1,030 cm⁻¹ was characteristic for C–O stretching of an alcohol both in cellulose and hemicelluloses (Revol 1982); the spectra showed an unexpected decrease in absorption after NaOH and hotwater treatments.

Aromatic rings, characteristic of lignin, showed an increased absorption at the following bands (Stewart et al. 1995): Aromatic skeletal vibrations (C–C stretch) assigned at 1,600 cm⁻¹ and 1,510 cm⁻¹ together with C–H out-ofplane bending vibrations assigned at 835 cm⁻¹, aliphatic C–H stretching assigned at 1,370 cm⁻¹, and an absorption band typical of guaiacyl rings assigned at 1,320 cm⁻¹. Overall, the absorption bands typical for lignin compounds generally increased after NaOH and hot-water treatments, which must be related to the solubilization of polysaccharides during treatments.



Figure 2.—Fourier transform infrared spectroscopy of rice straw.

Effect of treatments on surface morphology of rice straw

The morphological characteristics of the rice straw were altered by treatments. Before treatments, alternate smooth and irregular surfaces can be observed on the surface of rice straw (Fig. 3a). It is suggested that smooth surfaces correspond to small mastoids embedded in the subepidermal sclerenchyma. Irregular surfaces had two types of excrescences and presented trichomes. Hot-water treatments and NaOH treatments dramatically decreased excrescences and the wax–silica layer (Fig. 3b). NaOH treatments decreased the quantity of excrescences and silica and maintained the overall cell structure while eliminating hemicellulose and some amorphous substances in the rice straw (Fig. 3c). After oxalic acid treatments, fine cracks were produced on the surface of the rice straw, which suggested weakening of the rice straw structure (Fig. 3d).

Effect of treatments on wettability of rice straw surface

The final properties of composites depended on fiber properties and matrix properties as well as the adhesion between the reinforcing fiber and the matrix. Wetting the fiber was an integrated step in the adhesion process and played an important role. Figure 4 shows the contact angles of rice straw before and after treatments. Smaller contact angles indicated stronger infiltration, which indicated greater force with starch adhesives and higher interface bonding strength of the composites. Untreated rice straw had larger surface contact angles, and hot-water treatments, NaOH treatments, and acid treatments could reduce the contact angle on the rice straw surface at different levels. Hot-water treatments and NaOH treatments greatly effected rice straw wettability. Although the contact angles decreased with contact time (water was gradually spreading and permeating throughout the straw surface), the contact angle on the untreated rice straw surface was still 24° , 34° , and 7° higher than that on the hot-water-treated, NaOH-treated, and oxalic acid-treated rice straw surface, respectively.

Effect of temperature and shear rate on apparent viscosity of cornstarch adhesives

The gluing ability of cornstarch adhesives depended on their capacity to disperse and permeate in surface of adherends. Dispersion and permeation increased the contact area and adhesion onto surfaces, allowing the glue to form mechanical entanglements within the pore structure during the curing process, which in turn increased their bonding strength. Figure 5 shows the curve of effect of temperature and shear rate (decided by viscometer rotor speed) on the apparent viscosity of cornstarch adhesives. The apparent viscosity of cornstarch adhesives reached a peak value under different speeds at 10°C and then decreased with increasing temperature. This was due to higher temperatures (>10°C), which reduced the friction between the starch molecules. The apparent viscosity of cornstarch adhesives decreased with increasing speed at the same temperature, indicating the presence of a shear thinning phenomenon of cornstarch adhesives.

Effect of treatments on mechanical properties of decorative materials

Figure 6 shows the mechanical property results of obtained materials. The effect of rice straw treatments on mechanical properties would be the result of the competition



Figure 3.—Scanning electron microscopy images of rice straw (a) control rice straw, (b) hot-water-treated rice straw, (c) NaOH-treated rice straw, and (d) oxalic acid-treated rice straw.



Figure 4.—Contact angles of rice straw. HRS = hot-water-treated rice straw; NRS = NaOH-treated rice straw; ORS = oxalic acidtreated rice straw; CRS = control rice straw.



Figure 5.—Effect of temperature and rotor speed on apparent viscosity of cornstarch adhesives.

between the increased adhesion due to the more exposed polar groups on the rice straw's surface and the damage (cracks, etc.) caused by the chemical agents used. We calculated the average values of MOR, MOE, FS, IS, TS, and IB of materials produced with CRS, NRS, ORS, and HRS and bonded with cornstarch adhesives. FS, MOE, IS,

TS, and MOR values showed that hot-water treatments on RS induced better performance on the obtained materials. NaOH treatments on rice straw improved IB and MOE values but reduced FS, IS, TS, and MOR values of the materials. In contrast, oxalic acid treatments on rice straw reduced IB, FS, MOE, and IS of the materials significantly, and only TS and MOR values were more than those of untreated rice straw materials. This could be attributed to the higher content of silica, which existed in the form of a nonpolar surface structure, resisting the adsorption of rice straw with cornstarch adhesives. The waxy layer had negative impacts on the mechanical properties of rice straw materials. For example, the smooth layer of wax on the surface of rice straw created less friction between rice straw and adhesives; cornstarch adhesive had difficulty penetrating the surface of the rice straw, and thus the gluing process did not form good mechanical entanglements.

Hot-water treatments on rice straw partially removed the silica and wax on the surface, which reduced the contact angle on the straw surface previously mentioned in Figures 3 and 4 and increased infiltration with starch adhesives, creating higher interfacial bonding strength between rice straw and adhesives. Rice straw also maintained a better



Figure 6.—Effect of treatments on mechanical properties of composites. HRS = hot-water-treated rice straw; NRS = NaOH-treated rice straw; ORS = oxalic acid-treated rice straw; CRS = control rice straw; IB = internal bond; FS = flexural strength; MOE = modulus of elasticity; IS = impact strength; TS = thickness swelling; MOR = modulus of rupture.



Figure 7.—(a) Moisture content (MC) and 2-hour thickness swelling (2hTC) and (b) equilibrium moisture absorption (MA) of composites. HRS = hot-water-treated rice straw; NRS = NaOH-treated rice straw; ORS = oxalic acid-treated rice straw; CRS = control rice straw.

structure, and the materials obtained from HRS had higher mechanical properties. NaOH treatments on rice straw led to greater physical and chemical changes and reduced the stiffness of rice straw, improving the IB values. On the other hand, IB proved to be more sensitive to NaOH treatments on rice straw. It is noteworthy that NaOH-treated rice straw produced an increment in the average IB values. These findings were mainly attributed to the exposed hydroxyl groups from the lignocellulosic substrate. NaOH can dismantle the ultrastructure of rice straw, which in turn unfolded and exposed many hydroxyl groups. This may allow for improved bonding with polar groups (hydroxyl and carboxyl side-chain groups) from the cornstarch adhesives. In addition, the unfolded fibers had increased contact area with cornstarch adhesives, which can also contribute to the increasing internal bond strength. Oxalic acid treatments on rice straw slightly improved the wettability of the rice straw, but because of cellulose degradation and structural changes, the mechanical strength of the rice straw deteriorated (Sahin and Young 2008), giving NRS lower IB, FS, MOE, and IS values.

Effect of treatments on physical properties of decorative materials

RS-based materials can be used for indoor furniture and interior decoration. However, owing to their high hydroxyl content, such materials are susceptible to high humidity environments, leading to dimensional instability. Therefore, the WA and MA properties of rice straw–based materials are important research topics to address.

The moisture content (MC), 2 hTS, and equilibrium moisture absorption (EMA) for all materials are given in Figure 7. From Figure 7a, it can be concluded that the MC of NRS-based materials had a peak value of 5.6 percent and the MC of ORS-based materials had a minimum value of 4.3 percent. The 2 hTS of NRS-based materials had a minimum value of 182.5 percent. This was mainly due to the greater hydrolysis induced by NaOH treatments on rice straw, which contributed to the higher interfacial bonding strength between rice straw and adhesives. Although there were dramatic values for the 2 hTS, this can be attributed to the substantial amounts of hydrophilic components in the rice straw and cornstarch. Starch molecules possessed many hydroxyl groups, whose hydrogen bonding with water molecules is much greater than their binding force to the substrate. Desorption of water molecules severely decreased the bonding strength of starch adhesives under wet conditions. Poor water resistance of starch adhesives led to severe swelling of the materials, and 2 hTS values increased up to 179.1 to 208.1 percent.

From Figure 7b, EMA values of the materials increased from 7.1 to 7.9 percent. NRS-based materials had a maximum value of 7.9 percent, and CRS-based materials had a minimum value of 7.1 percent. This could account for the better wettability of the NRS and better water resistance of CRS that had silica and wax, which served as a waterresistant layer for rice straw. However, compared with rice straw powder/polypropylene composites, whose EMA value was 6.2 percent (Dongjun 2010; rice straw powder loading contents of 80 wt%), rice straw/cornstarch adhesive materials had slightly lower moisture resistance.

Conclusions

In this work, biologically based decorative materials were prepared using rice straw as a natural fiber bonded with cornstarch-based adhesives to give a zero formaldehyde emissions product. Treatments on rice straw were undertaken to improve wettability of rice straw fiber and improve the adhesion with adhesives such as those derived from cornstarch. All the treatments modified the rice straw surface properties, as evidenced by FTIR and improved rice straw wettability. SEM suggested that NaOH and hotwater treatments resulted in a more dramatically altered rice straw substrate than with the oxalic acid treatments. Apparent viscosity of cornstarch adhesives decreased with shear rate increasing, and increased and then decreased with increasing temperature, reaching a maximum at 10°C. Hotwater-treated rice straw materials displayed the best set of final mechanical properties. The materials exhibited dramatic 2 hTS properties and poor water resistance owing to the significant hydrophilic nature of both the rice straw and cornstarch adhesive. The biologically based decorative materials described in this work should thus be good candidates for indoor applications for which the requirements for water resistance are not stringent.

Acknowledgment

The financial support of the basic research projects of National University special fund (KYZ200921) is gratefully acknowledged.

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