SURFACE STRUCTURE AND DYNAMIC ADHESIVE WETTABILITY OF WHEAT STRAW

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ABSTRACT

The structural features of wheat straw differ from those of wood. By means of an Optical Microscope (OM) and a Scanning Electron Microscope (SEM), three kinds of tissues (epidermis, parenchyma, and vascular tissue) were observed on the cross section of wheat straw. A smooth cuticle was found on the exterior surface. The exterior surface of wheat straw treated by NaOH solution at room temperature appeared to be chemically etched. After this treatment, the wettability of the exterior surface was improved substantially. In this study, using a wetting model describing the dynamic contact angle process, a parameter (*K*) was used to quantify the adhesive spreading and penetrating during the wetting process. By applying the wetting model, the adhesive wettabilities associated with resin type (UF, PF, and PMDI), drop location on the wheat straw surface (exterior and interior), and grain direction (along and across) were compared. The results of this study showed that PMDI resin had a lower contact angle (both initial and equilibrium) and a greater spreading and penetrating constant compared to UF and PF resins on natural (untreated) wheat straw surfaces. The *K* value of the interior surface was higher than that of the exterior surface for the same resin on the untreated wheat straw. In addition, the *K* values of the three resins on the treated wheat straw surfaces were higher than those on untreated wheat straw surfaces. This indicates that the alkali treatment was an effective method for improving the wettabilty of wheat straw surfaces. The wheat straw grain direction also significantly affected the adhesive wetting process. The *K* values of adhesive wetting along the wheat straw grain direction were always greater than those across the grain direction for the same resin.

Keywords: Wheat straw, surface structure, wettability, adhesive, penetrating, spreading, OM, SEM.

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INTRODUCTION

In the production of composite panels from annual crop and plant straws, some problems exist in seasonality, storage, scattering sources, and bondability (Rowell et al. 1997). Among these factors, bondability remains a major unsolved technical problem, especially when urea-based resins are applied (John and Robert 1995; Sauter 1996). The poor properties of UF-bonded straw boards are related to many factors such as the interface characteristics of adhesive and straw, the properties of adhesives, the structure of wheat straw, and bonding technology. It is well known that bonding is a complex process involving both physical and chemical phenomena. Bonding is related to the fluidifying, flowing, wetting, curing, and deforming of adhesives. Of all these factors, wettability appears to be an important one before resin curing.

Wetting is normally used to describe what happens when a liquid comes into contact with a solid surface. Wetting also refers to the manifestations resulting from molecular interactions between liquids and solids in direct contact at the interface (Berg 1993). The three manifestations during the wetting process are formation of a contact angle, adhesive spreading, and adhesive penetration. Adhesive wettability is the ability of the adhesive to make close contact with a surface. Zisman (1962) studied the constitution effects of adhesion. Herczeg (1965), Chen (1970), Hse (1972), and other researchers studied the wettability of wood by determining the instantaneous or equilibrium contact angles at the solid/adhesive interface. Most previous studies on the thermodynamics of liquid/solid interaction of wood examined the instantaneous or equilibrium contact angles and not the liquid spreading and penetrating processes (Nguyen and Johns 1978). However, adhesive spreading and penetrating are also important indices for evaluating wood-based or nonwood-based material adhesion. Theoretical discussion on the surface dynamic behavior of polymers has been reported by Liu et al. (1995). Recently, researchers have realized the importance of measuring the contact angle change as a function of time in liquid wetting studies (Maldas and Kamdem 1998; Scheikl and Dunky 1998). Dynamic adhesive wettability of Douglas fir (*Pseudotsuga menziesii*) and southern pine (*Pinus* spp.) was studied by Shi and Gardner (2001). Wood surface properties including wettability were reported by Gardner (2002).

The structure of wheat (*Triticum aestivum L.*) straw, a type of annual herbage plant, differs from that of wood. Fissile and vegetal tissues of herbage plants are distributed chiefly in twigs and joints. There is no fissile or vegetal tissue at the hypodermis. When wheat grows, the stem of wheat straw does not become very thick across the grain direction but extends along the grain direction. Wheat straw consists of the spike, stem, leaf, and root—all distinguishable with the naked eye. Generally, the stem ranges in height from 29 cm to 97 cm, and the stalk diameter ranges from 2 mm to 4 mm. The thickness of the walls ranges from 0.3 mm to 0.7 mm. The diameter of the marrow cavity ranges from 0.9 mm to 1.9 mm. The joints of the stem above the ground consist of 3 to 6 units. The thickness of the stalk wall increases from top to bottom (Paper and Pulp Manual 1987).

The chemical components of wheat straw are similar to those of wood. The major chemical components of wood are cellulose, hemicellulose, lignin, and some extractives. The main difference between wheat straw and wood is in the high silica and wax content, concentrated primarily on the surface of wheat straw. This surface layer interferes with the moisture absorption and adhesive penetrating in water-based adhesives like UF resin. Thus, the layer acts as a barrier to bonding (Markessini et al. 1997; Han et al. 1998). Removing this bonding-barrier layer from straw materials has been an ongoing technical problem in the performance enhancement of straw panels.

Wheat straw is an anisotropic material: adhesive wetting behavior may be different along the grain than across the grain. With the wetting model developed by Shi and Gardner (2001), the spreading and penetrating abilities of a liquid/solid system can be quantified. This is more useful and easier for the comparison of adhesive wettability. The final expression of the wetting model, in which the contact angle (θ) changes as a function of time (t), is:

$$\theta = \frac{\theta_i \bullet \theta_e}{\theta_i + (\theta_e - \theta_i)\exp\left[K\left(\frac{\theta_e}{\theta_e - \theta_i}\right)t\right]}$$

where θ_i represents the instantaneous (initial) contact angle, θ_e represents the equilibrium contact angle, and K is the contact angle change rate constant.

K is also a constant referring to the intrinsic relative contact angle decrease rate. The rate of contact angle change is related to the rate of liquid spreading and penetrating on the solid surface. Therefore, the intrinsic relative contact angle change rate constant (K) in the equation can also be referred to as the spreading and penetrating constant. Among the wettability parameters, the K value represents, physically, how fast the liquid spreads and penetrates into the structure of wheat straw. By knowing the K value, spreading and penetrating in an adhesive/straw system can be quantified. The greater the K value is, the faster the spreading and penetrating occur. θ_i refers to the wettability of the adhesive on the straw surface. The smaller the contact angle is, the better the wettability is. The greater the percent decrease is, the better the spreading and penetrating abilities of the adhesive become. These parameters are normally used to describe wettability, and adhesive spreading and penetrating on the straw surface from different aspects.

The purposes of this study were, first, to observe the structural features of the wheat straw surface using an Optical Microscope (OM) and a Scanning Electron Microscope (SEM); second, to observe the structural features of the wheat straw surface after treating with a 0.6% NaOH solution at room temperature; and, finally, to quantitatively evaluate the adhesive wettability of different adhesive types on different wheat straw surfaces using a contact angle measuring apparatus.

MATERIALS AND METHODS

Materials

Wheat straw was obtained from outside Zhengzhou City, He'nan Province, China. It was dried in sunlight after harvest and then cut into 3- to 5-cm-length pieces. The cut straw was air-dried for a few months in airing shelves.

Three adhesive resins commonly used in wood composite industry were evaluated in the wetting experiments: urea-formaldehyde (UF), phenol-formaldehyde (PF), and polymeric diphenylmethane diisocyanate (PMDI). The PF resin was obtained from the Institute of Forest Products in Heilongjiang Province; the UF resin and PMDI resin were prepared in the adhesives laboratory of Northeast Forestry University. Table 1 shows the major specifications of the three resins.

Methods

Observing surface structure.—First, clean flakes were chosen from the air-dried wheat straws, the moisture content of the untreated wheat straw was 7.73%. These flakes were then made into slices and observed on the holder within fastening liquid. A photograph was taken after adjusting the ocular and the objective of the Olympus BX51 Optical Microscope (OM, made in Japan) as soon as the field of vision became very clear. The interior and exterior surfaces were observed using the OM. Second, the samples of the interior and exterior surfaces were observed using a KYKY-100B Scanning Electron Microscope (SEM).

In addition, wheat straws were saturated by spraying them with a 0.6% NaOH solution. During the course of spraying and wetting, almost

TABLE 1. *Specifications of the UF, PF, and PMDI resins used in the study.*

Specification	UF	PF	PMDI
Color	Opalescent to maroon	Pale red-brown to black	Dark brown
Nonvolatile content (%)	60.5	41.3	71.5
pH value	7.8	11.4	6.3
Viscosity (cps)/20°C	200	220	260
Density ($g \bullet cm^{-3}$)	1.20	1.12	1.29
Free–NCO (%)	—	—	14.5
Surface free energy*(mJ/m^2)	56.23	58.92	46.75

* The surface free energies of resins were measured using the method of suspending drop.

no diluted alkali leached out from the treated wheat straws. After 8 h, the wheat straws were dried to a moisture content of approximately 4% in an oven at 95°C to 102°C in order to be used for manufacturing particleboard. In the wettability experiment, the treated straws were stored in the air for 2 weeks after drying. Finally, the interior and exterior surfaces of the treated wheat straws were observed with the OM and the SEM. At the same time, we could observe the structural changes of wheat straw surfaces.

Contact angle measurement.—In adhesive wetting measurements, a drop of adhesive was placed on the surface of wheat straw. The conditions of adhesive wetting measurements were as follows:

Resins: UF, PF, and PMDI
Dimension of the specimens: 2 mm wide by 15 mm long
Temperature of the lab: 16.0 ± 2.0°C
Drop volume: 5 μl
Reduplicate: 5
Measuring sites: Exterior surface along (//) the grain direction
Exterior surface across (⊥) the grain direction
Interior surface along (//) the grain direction
Interior surface across (⊥) the grain direction

Wetting measurements were made using the JC2000A Contact Angle Measuring Apparatus (CAA, made in Shanghai, P.R. China). Images of the adhesive drop shape on the straw surface were captured by a camera and saved every 2 s. During the wetting measurements, as time elapsed, the drop shape tended to stabilize (equilibrium contact angle was obtained). The contact angles at both ends of the measured drop were averaged. Nine data points were taken for each recorded drop to obtain a curve of contact angle vs. time. Five replicates were averaged for each sample. Representative images of the adhesive drops are shown in Fig. 1. As seen in Fig. 1, the length of contact between the sessile drop and the straw surface increased as time elapsed due to liquid spreading. It is also seen from Fig. 1 that the drop volume decreased as a function of time. The volume decrease was mainly due to liquid penetrating into the porous structure of the wheat straw surface, especially to the interior surface. Therefore, these phenomena confirmed that the contact angle change as a function of time was caused both by liquid spreading and penetrating into the substrate.

The equation was applied to the experiment data. The K value, the asymptotic standard error (SE), and the R^2 value of the constant were calculated based on curve-fitting results by using

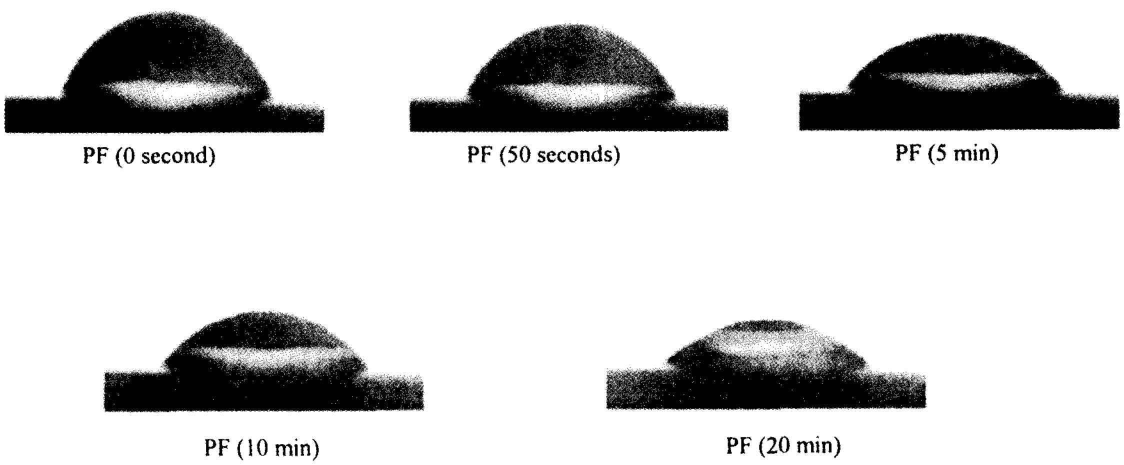
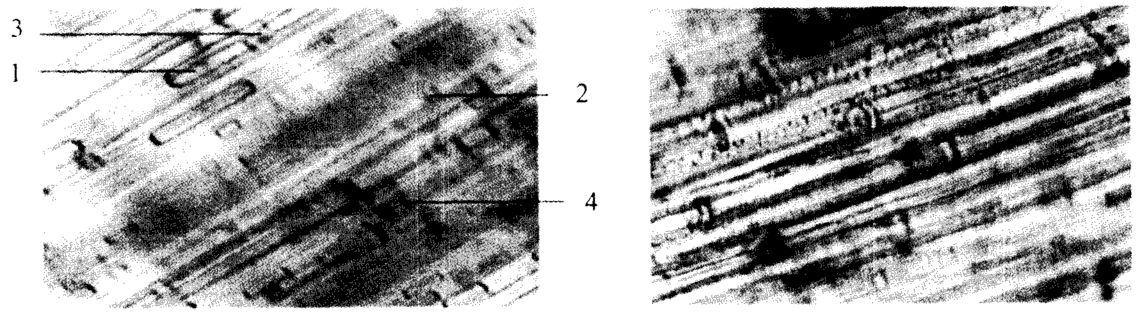


FIG. 1. Images of the same sessile drop of PF on the exterior surface along the grain direction at different time intervals.



2a 2b

FIG. 2. The comparison of exterior surfaces for untreated straw (2a) and treated straw (2b) LM × 200 (OM). 1. Long cell; 2. Suberization cell; 3. Silicon cell; 4. Stoma.

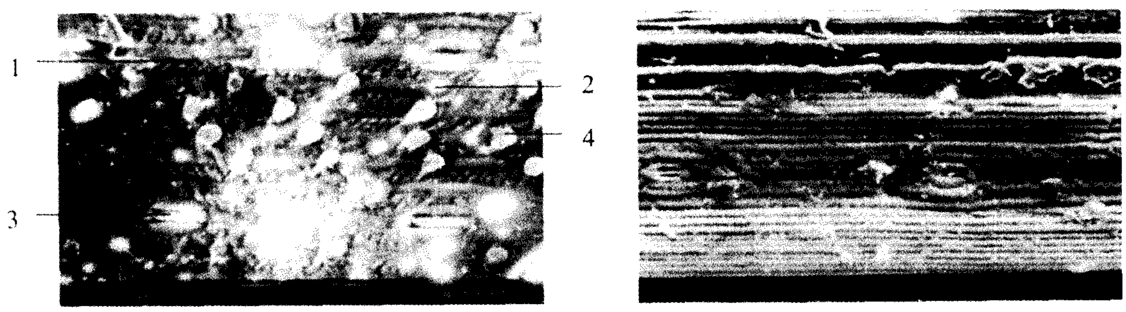
statistical analysis software (JMP3.2-SAS and SPSS8.0).

RESULTS AND DISCUSSION

Structural features of wheat straw

The exterior surface structure of wheat straw was observed with the OM and SEM. The images are shown in Figs. 2a and 3a, respectively. As we know, the epidermis is the exterior layer of cells. These cells are both long and short. One long cell and two short cells are arrayed alternately in the epidermis. The short cells are divided into two kinds—silica cells and suberization cells. The cuticle is composed mostly of cutin and waxes. Cutin is a biopolyester made of interesterified hydroxyl, and epoxy-hydroxy C16 and C18 fatty acids. The cuticle prevents excessive interior water evaporation and bacteria incursion and affects the resin's wetting, spreading, and penetrating abilities. The cuticle is easily formed in the epidermis because of suberization and mineralization.

In Fig. 2a, we see that the straw surface is smooth. There are long cells, suberization cells, silica cells, and stomas in the straw walls. In Fig. 3a, we see the platode mastoid epidermal hairs in the straw surface features as well as long cells, silica cells, and stomas. It was observed that there were parenchyma cells in the interior surface, as shown in Figs. 4 and 5. The parenchyma cells are distributed throughout the basic parenchyma tissues, which are thinner, de-



3a 3b

FIG. 3. The comparison of exterior surfaces for untreated straw (3a) and treated straw (3b) LM × 300 (SEM). 1. Long cell; 2. Silica cell; 3. Stoma; 4. Platode mastoid epidermal hair.

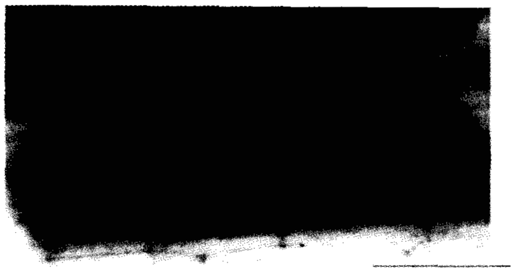


FIG. 4. Interior surface of wheat straw LM × 200 (OM). 1. Parenchyma cell.

FIG. 6. Interior surface of treated wheat straw LM × 260 (OM).

formed, and fragile. The shape and dimension of the parenchyma cell vary with straw species (Wu 1991). The shape in Fig. 5 seems to be oblong. These parenchyma cells were not closely arrayed and did not form cuticle. In general, it was found that the exterior surface was smoother than the interior surface. Further observation showed that there were cuticles and stomas in the exterior surface. The tissue of the interior surface consisted mainly of parenchyma cells.

The images of exterior surfaces of NaOH treated straw are shown in Figs. 2b and 3b. If we compare Fig. 2a with Fig. 2b, and Fig. 3a with Fig. 3b, we see that the exterior surfaces have a lot of small rugged canals. We have called this phenomenon "chemical etching." Due to this "chemical etching," the cuticle was either partially visible or entirely absent under the OM and SEM. The change of the exterior surface was significant, while the change in the interior surface, shown in Figs. 4 and 6, was insignificant.

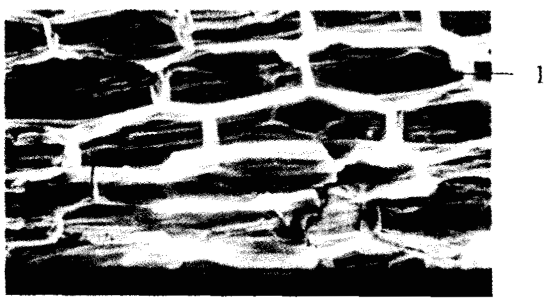


FIG. 5. Interior surface of wheat straw LM × 304 (SEM). 1. Parenchyma cell.

Wettability of wheat straw

Figures 7, 8, and 9 show the contact angle changes as a function of time for the different surfaces of wheat straw and the three adhesive systems. The experiment data fit the wetting model curve well. The penetration/spreading constants (*K* values) of all the samples and their asymptotic standard errors (SE), and the R^2 values of the fit, calculated using SPSS8.0, are shown in Table 2. In Table 2, we see that the R^2 values of the wetting model were over 0.95 for the wheat straw surfaces examined. Therefore, the developed wetting model could accurately describe the adhesive wetting process on the wheat straw surface. Based on these results, the adhesives and adhesives wettability for the different wheat straw surfaces, whether treated or not, were discussed separately.

The alkali treatment apparently had an effect on the contact angles of the three resins on either the exterior or interior surfaces. The *K* value of the three resins on the treated wheat straw was approximately 10 times greater than that of the untreated surface. This proved that the spreading and penetration of the three resins on the treated surface were substantially improved. Table 2 shows that PMDI resin for the whole column had a greater percent decrease in contact angle from initial to equilibrium (21.5–76.9%), compared to UF (13.3–36.3%) and PF (20.2–53.0%) resins. This trend indicates that PMDI resin exhibits better spreading and penetrating ability than UF and PF resins. Previous publications

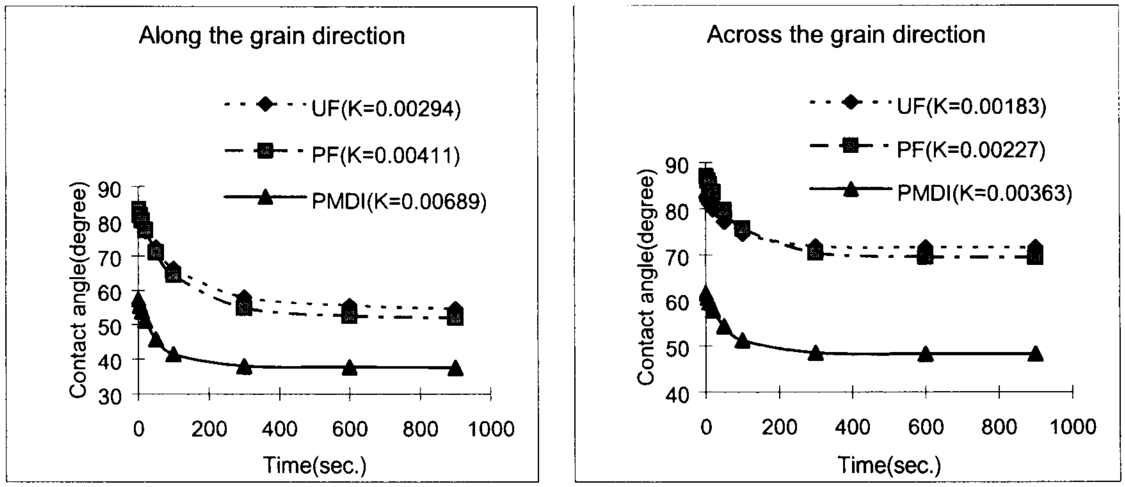


FIG. 7. Contact angle changes as a function of time on the exterior surface for untreated wheat straw.

have shown that wood or straw bonded with PMDI resin exhibited better physical and mechanical properties than when bonded using PF and UF resin at the same resin content (Sun et al. 1994; Shi and Wang 1997). When wheat straw was treated by extracting treatment or silane coupling agents, the bonding strength was improved for UF resin (Han et al. 1999). The results of this current study show that treating wheat straw with dilute alkali substantially improved the wettability of the three adhesives. After the wheat straw was treated by dilute alkali, equilibrium time of the adhesive resin decreased a great deal. The spreading and penetrating abilities of the three resins improved very quickly. Although the treated straws were dried at close to 100°C, the temperature may have affected NaOH treatment and may have caused some chemical components to decompose slightly. However, the cuticle, which is

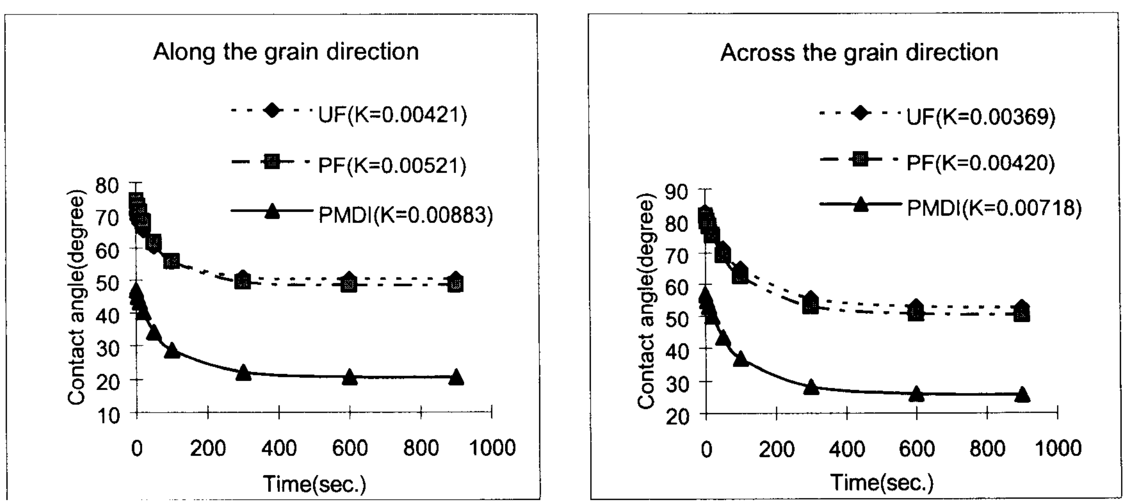


FIG. 8. Contact angle changes as a function of time on the interior surface for untreated wheat straw.

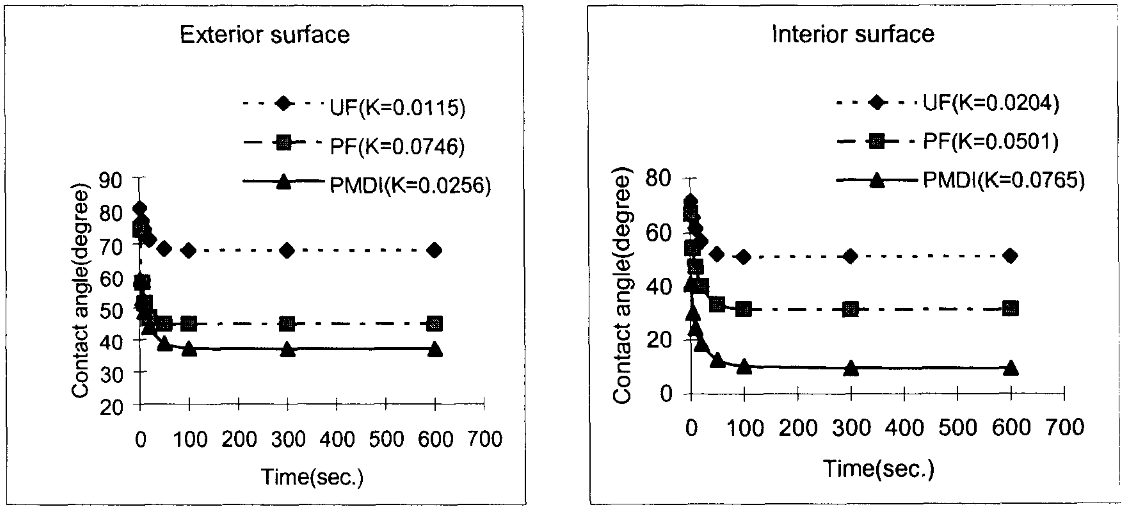


FIG. 9. Contact angle changes as a function of time along the direction for treated wheat straw.

TABLE 2. *Contact angles and* K *values on different wheat straw surfaces in different conditions.*

Sample	Surface	Direction	Resin	Contact Angles* θ_i (Degree)	θ_e (Degree)	Percent decrease (%)	*K* value Value (l/s) ($\times 10^{-3}$)	SE ($\times 10^{-3}$)	R^2 model fit
UW	OS	//	UF	81.5 (4.28)	54.5 (3.67)	33.1	2.94	0.055	0.98
			PF	83.5 (3.73)	52.0 (4.50)	37.7	4.11	0.019	0.97
			PMDI	57.3 (4.20)	37.5 (5.75)	34.6	6.89	0.073	0.98
		⊥	UF	82.5 (3.82)	71.6 (4.81)	13.3	1.83	0.031	0.96
			PF	87.1 (3.20)	69.5 (4.95)	20.2	2.27	0.067	0.97
			PMDI	61.5 (3.01)	48.3 (4.48)	21.5	3.63	0.140	0.97
	IS	//	UF	70.3 (3.25)	50.3 (4.55)	28.4	4.21	0.079	0.98
			PF	74.5 (3.46)	48.5 (5.19)	34.9	5.21	0.073	0.98
			PMDI	46.9 (3.34)	20.5 (3.95)	56.3	8.83	0.160	0.98
		⊥	UF	82.7 (3.69)	52.7 (3.81)	36.3	3.69	0.075	0.98
			PF	81.6 (3.21)	50.5 (2.93)	38.1	4.20	0.048	0.98
			PMDI	56.8 (3.84)	25.6 (4.75)	54.9	7.18	0.031	0.98
TW	OS	//	UF	80.5 (5.42)	68.0 (5.56)	15.5	11.50	1.08	0.95
			PF	74.3 (4.47)	44.9 (3.38)	39.6	74.60	2.70	0.98
			PMDI	59.0 (4.92)	37.0 (5.35)	37.3	25.60	1.30	0.98
	IS	//	UF	71.5 (3.42)	51.5 (3.84)	28.0	20.40	0.93	0.98
			PF	67.0 (5.20)	31.5 (4.23)	53.0	50.10	2.10	0.97
			PMDI	41.2 (3.31)	9.5 (3.22)	76.9	76.50	2.20	0.98

UW: untreated wheat straw; TW: treated wheat straw; OS: exterior surface; IS: interior surface; θi: instantaneous contact angle; θe: equilibrium contact angle; UF: urea formaldehyde; PF: phenol formaldehyde; PMDI: polymeric diphenylmethane diisocyanate;

SE: asymptotic standard error; //: along the grain direction; ⊥: across the grain direction.

*Contact angle represents an average of ten measurements over five straw samples (two measurements per sample) for each resin. Values in parentheses are standard deviations.

mainly composed of fatty acids, has a relatively high melting point: the drying temperature would not be high enough to cause the cuticle on the exterior surface of straw to decompose. Therefore, these manifestations showed that alkali treatment could be effective in improving the wettability of the wheat straw surface.

Adhesive type: UF, PF, and PMDI

From the comparison curves plotted in Figs. 7, 8, and 9, it is apparent that the resin type (UF, PF, and PMDI) had a significant effect on the adhesive wetting process. The PMDI resin exhibited lower instantaneous and equilibrium contact angles than the PF and UF resins. The lower contact angles for the PMDI resin were expected since the PMDI had a lower surface tension (46.75 mJ/m^2) than those of the UF resin (56.23 mJ/m^2) and PF resin (58.92 mJ/m^2). According to Young's equation for the classical case of the three-phase line of solid, liquid, and vapor, we can deduce that a higher solid surface tension or a lower liquid surface tension will form a lower contact angle in the solid/liquid system. In the experiment, PMDI resin had a lower surface tension and had a lower contact angle than the UF and PF resins. The lower contact angle of the PMDI resin on the wheat straw surface indicates that the PMDI resin has a greater wettability than the UF and PF resins.

In the untreated wheat straw surfaces, the PMDI resin exhibited higher K values than the UF and PF resins (Table 2). For example, the K value of PMDI resin (0.00689) on the exterior surface along the grain direction was 68% greater than that of PF (0.00411) and 134% greater than that of UF resin (0.00294), while the K value of PMDI (0.00883) on the interior surface along the grain direction was 69% greater than that of PF resin (0.00521) and 110% greater than that of UF resin (0.00421). The greater K values of PMDI indicate that the PMDI resin has a greater wettability than the UF and PF resins. But, for the treated wheat straw, the K value of PF resin (0.0746) on the exterior surface along the grain direction was the highest among the three resins. The major reason was likely that the PF resin took advantage of the residual alkali and the chemical etching, and may have been more easily adsorbed by the exterior surface of the straw. The higher K value of PF resin indicated that it penetrates and spreads more quickly on the treated wheat straw surface than UF and PMDI resins. Also, for the exterior surface treated by dilute alkali, the percent decrease of K value for the PF resin was the greatest among the three resins. Based on the increased K value of PF resin, we judged that treatment of wheat straw by alkali may contribute to improved spreading and penetration.

Wheat straw surface: Exterior vs. interior

As seen in Table 2 and Figs. 7, 8, and 9, the K value of the interior surface was higher than that of the exterior surface for the same resin on the untreated wheat straw. This indicates that the spreading and penetration of the interior surface were better than that of the exterior surface. For the treated straw, the K value of the interior surface was higher than that of the exterior surface for UF and PMDI resin, while PF resin had a higher K value on the exterior surface compared to the interior surface. In fact, the wettability for exterior and interior surfaces improved after being treated with NaOH. As discussed above, the structures of the exterior and interior surfaces are different. After treating the exterior surface with NaOH, the chemical etching was more obvious on the exterior surface than on the interior surface. Thus, the etched exterior surface with the residual alkali on it was likely to accelerate the adsorption of PF resin and then increased the spreading and penetrating rate.

Grain direction: Along vs. across

The mechanical and physical properties along and across the grain directions are different. This characteristic of wheat straw material was also present in the adhesive wetting process on the wheat straw surface. Figures 7 and 8 show a comparison of contact angles as a function of time on the exterior and interior surfaces. As shown in Table 2 and Figs. 7 and 8, both the instantaneous (initial) and equilibrium contact angles were lower along the grain direction than across the grain direction for the same resin on the exterior surface of the untreated wheat straw. The *K* values along the wheat straw grain direction were greater than those across the grain direction for all three adhesives (seen in Table 2). Based on the greater *K* values, it can be concluded that the adhesive has better spreading and penetration along the grain direction than across the grain direction. This was likely due to the wheat straw surface structure causing the liquid spreading and penetrating difference. Therefore, when measuring contact angles on wheat straw, the grain direction on the surface cannot be ignored.

CONCLUSIONS

Using an OM and a SEM, three kinds of tissue (epidermis, parenchyma, and vascular tissue) were observed on the cross section of wheat straw. There were long cells, suberization cells, silica cells, and stoma on the exterior surface. The interior surface tissue consisted mainly of parenchyma cells. These parenchyma cells were not closely arrayed.

The exterior surface of NaOH-treated wheat straw appeared to be chemically etched. Little changes were observed on the interior surface after NaOH treatment.

The adhesive wetting process on the adhesive/straw system was described by using a dynamic wetting model. The wetting model could be used to accurately describe contact angle changes as a function of time. The constant (*K* value) in the model could be used to quantify the spreading and penetrating rate of the adhesive/straw system. On the natural (untreated) wheat straw surfaces evaluated in this study, PMDI resin had a lower contact angle (both initial and equilibrium) and greater spreading and penetrating constant compared to UF and PF resins. The *K* value of the interior surface was higher than that of the exterior surface for the same resin on the untreated wheat straw. In general, the *K* values of the three resins on the treated wheat straw surfaces were greater than on untreated wheat straw surfaces. This indicates that alkali treatment was an effective method for improving the wettabilty of wheat straw surfaces. The wheat straw grain direction also significantly affected the adhesive wetting process. Both instantaneous (initial) and equilibrium contact angles were lower along the grain than across the grain. The *K* values of adhesive wetting along the wheat straw grain direction were greater than those of adhesive wetting across the grain direction for the same resin.

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