Influence of Atmospheric Pressure Dielectric Barrier Discharge Plasma Treatment on the Surface Properties of Wheat Straw

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The effects of atmospheric pressure dielectric barrier discharge (DBD) plasma on the surface properties of wheat straw were investigated in this work. The surface wettability changes in the wheat straw were determined using contact angle measurements, the surface morphology was observed using scanning electron microscopy (SEM) and atomic force microscopy (AFM), and the chemical characteristics were scanned using X-ray photoelectron spectroscopy (XPS). Moreover, the shear strength was measured using a paper tension meter. The results indicated that after plasma treatment, the urea-formaldehyde (UF) resin had lower instantaneous and equilibrium contact angles on the wheat straw surfaces than the untreated specimens did, which decreased by 34% and 64%, respectively. Obvious etching was observed on the wheat straw surface after plasma treatment. There was an increase in the O/C ratio along with an increase in the C2, C3, and C4 proportions based on the XPS analysis after plasma treatment on wheat straw surface. Moreover, the shear strength between glued surfaces of the wheat straw was greatly improved after plasma treatment, indicating that atmospheric pressure DBD plasma treatment was an effective method for improving the surface properties of wheat straw, which were helpful for UF resin penetration and application of wheat straw.

Keywords: Wheat straw; Plasma treatment; Surface property

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INTRODUCTION

As a developing country, China is experiencing many challenges in the context of natural forests. They have been eradicated or severely diminished, resulting in a shortage of wood. Many researchers are discussing possible ways to help China maintain a sustainable forest sector, while at the same time helping to bridge the gap between wood demand and supply (Xu *et al.* 2004).

Agricultural residues such as wheat straw, which is an abundant, inexpensive, and readily available source of renewable lignocellulosic biomass, has drawn the increasing attention of the wood industry (Yu *et al.* 2007). As a traditional agricultural country, China has rich crop straw resources. According to statistics (Yang *et al.* 2013), the crop straw output is more than 700 million tons a year on average, and 60% to 70% of that is wheat straw and rice straw. However, wheat straw and rice straw have few applications in industrial production on a large scale and are mostly burned, which causes environmental pollution. Therefore, there is an urgent demand for the large-scale and comprehensive utilization of straw (Yang *et al.* 2013). In addition, research indicates that wheat straw is an ideal type of lignocellulosic material for biocomposites and has been tested in the laboratory (*e.g.*, processed into shavings, fibers, or used for the preparation of composite materials).

However, another serious challenge has been encountered in the production of biocomposites from wheat straw because, as a lignocellulose material, wheat straw is more complicated than wood. It contains other elements, including actual fibers, parenchymal cells, vessel elements, and epidermal cells, which are the outermost surface cells and covered with a large amount of silica wax and lipophilic extractives (Jiang *et al.* 2009; Wen *et al.* 2013). Such lipophilic substances can prevent liquids (such as UF resin) from penetrating into the wheat straw body and producing strong adhesion (Xu and Zhou 2009). In response to this problem, many researchers have suggested a large number of surface treatment methods for wheat straw to improve its surface performance, including mechanical, hot water, physical, chemical, and biological treatments. Compared with these treatments, plasma modification is a relatively recent technology in biomass materials surface modification and is acknowledged as one of the best methods for rendering cellulosic surfaces more hydrophilic; it is fast, energy efficient, and low polluting (Zhuang *et al.* 2002). Researchers have studied the effects of low temperature plasma treatments on wheat straw fibers, corn stalks, and sorghum stalk surfaces (Busnel *et al.* 2010; Yang *et al.* 2014). The results showed that low temperature plasma treatment can destroy the weak interface layer on the surface of the straw and can introduce hydroxyl, carboxyl, and carbonyl oxygen-containing functional groups on the surface, increasing the surface activity (Ai 2001; Chen 2006). However, these low temperature plasmas were mainly produced in vacuum states. They have significant equipment requirements, complex operation, large energy consumption, and are not conducive to large-scale industrial production. Dielectric barrier discharge (DBD) plasma is a type of low temperature plasma that works at atmospheric pressure. It has been used for surface modification and requires only simple discharge equipment, has high energy efficiency, and is easily implemented in an industrial environment (Zhuang *et al.* 2002; Zhang *et al.* 2013).

In this study, atmospheric pressure DBD plasma was used to treat wheat straw to obtain better surface properties in order for UF resin to penetrate into the wheat straw body and produce strong adhesion to optimize the DBD plasma treatment process. The changes in wheat straw before and after plasma treatment were characterized by measuring surface wettability, surface morphology, surface chemical composition, and shear strength between glued surfaces.

EXPERIMENTAL

Materials

Wheat straw was harvested in an agricultural field in the northern Jiangsu Province in China. It was cleaned with distilled water to remove impurities from the surface and then split and cut into 10-cm-long pieces. Before plasma treatment, the straw samples were dried in a vacuum oven at 60 °C to a moisture content of 2%. All samples were kept in a desiccator to balance the moisture content.

UF resin was prepared in a laboratory at Nanjing Forestry University. It was used to evaluate the surface wettability and shear strength of the wheat straw. The major specifications of the UF resin were as follows: the solids content was 53.3% after drying at 120 °C for 2 h, the pH value was 7.5, and the viscosity was 37 s at 20 °C.

Methods

DBD plasma treatment

The discharge voltage, electrode space, and treatment time are extremely important process parameters for atmospheric pressure DBD plasma treatment. After a preliminary experiment, it was determined that the equipment did not discharge or discharged unevenly when the discharge voltage was less than 25 V or the electrode spacing was larger than 10 mm. The surface of the wheat straw burned or was destroyed when the discharge voltage was higher than 40 V or the electrode spacing was less than 4 mm. Therefore, a discharge voltage of 30 V and an electrode spacing of 6 mm were chosen. The primary focus was the effect of the treatment time on the surface of wheat straw. In the experiment, flattened and dried wheat straw specimens (5 mm × 100 mm) were placed between the two insulating media of the discharge device (shown in Fig. 1) of DBD plasma treatment equipment (CTP-2000K, made in Nanjing, China) for processing. Treatment times of 30, 45, 60, 75, and 90 s were utilized to achieve different treatment effects.

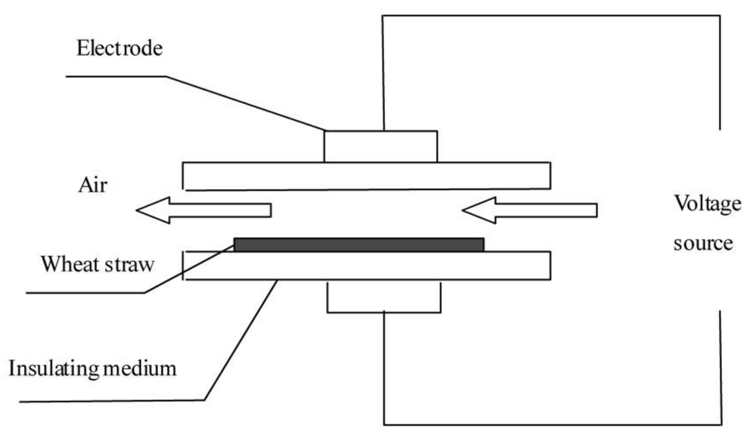

Fig. 1. Atmospheric pressure plasma processor

Contact angle measurements

Drops of UF resin formulation (volume 4 mL, 20 °C) were dispersed on the interior and exterior surfaces of the plasma-treated and untreated wheat straw specimens. Ten replicates were made to measure the contact angle. The tests were performed using an optical contact angle measuring apparatus (Theta, Sweden). Images of the drop shapes on the specimens were captured using a camera connected to a computer and saved. As time elapsed, the drop shape tended to stabilize, reaching an equilibrium contact angle.

Morphology observation by SEM method

The wheat straw was cut into 10 mm × 10 mm squares, then put into a vacuum drying oven at 60 °C and 0.1 Pa for 10 h. Next, the wheat straw surfaces were treated with atmospheric pressure DBD plasma. SEM (JSM-7600F, Japan) images of the wheat straw interior and exterior surfaces were obtained after gold coating in a vacuum.

Morphology observation by AFM method

The wheat straw was cut into 2 mm × 2 mm × 40 mm pieces using the Franklin maceration method and put into boiling water, where it sank to the bottom of the test tube. Next, the water was poured out, a hydrogen peroxide and glacial acetic acid (volume ratio 1:1) solution was added, and the solution was heated until the wheat straw turned a white color. Then, the wheat was washed with distilled water to neutral conditions and shaken until the straw was fully broken. Finally, the sample was stored in a weighing bottle. Distilled water drops, which contained wheat straw fibers, were dripped onto clean mica sheets, placed in a standard machine (KD-P, made in Zhejiang, China) to eliminate moisture, and put into a vacuum drying oven at 40 °C for 2 h. Finally, the wheat straw fibers were treated with atmospheric pressure DBD plasma. After processing, the fiber-loaded mica sheet was attached to a stage for observation using Atomic Force Microscopy (AFM) (XE-100, Korea).

Chemical composition analysis by XPS

The wheat straw was cut into a 10 mm × 10 mm square and put into a vacuum drying oven at 60 °C and a vacuum of 0.1 Pa for 10 h. Then, the wheat straw was treated with atmospheric pressure DBD plasma. Information about the chemical characteristics of the treated and untreated wheat straw surfaces was determined using X-ray Photoelectron Spectrometry (XPS) (K-Alpha, Thermo Scientific, America). The Analysis room vacuum was 10^{-9} Pa, the space resolution was 100 μm, and the energy step size was 0.1 eV.

Shear strength evaluation

Before evaluating the shear strength, two pieces of untreated and plasma-treated wheat straw specimens were coated with UF (1% NH_4Cl as curing agent), then heated and pressed in a hot press (X16, made in Qingdao, China). The experimental conditions were as follows: specimen dimensions: 5 mm × 100 mm; gluing section: 5 mm × 20 mm; gluing surface: exterior-to-exterior, interior-to-exterior, and interior-to-interior; resin content: 200 g/m^2; hot-pressing pressure: 1.5 MPa; hot-pressing temperature: 110 °C; hot-pressing time: 90 s.

This experimental method was established by our research group (Yang *et al.* 2014). The shear strength was measured using the paper tension meter (WZL-300, made in Hangzhou, China) after the prepared specimens were cooled for more than 24 h. Twenty replicates were made for each sample.

RESULTS AND DISCUSSION

Effect of DBD Plasma Treatment on Wettability of Wheat Straw

The wettability of solid materials is largely dependent on the contact angle (Aydin 2004). Therefore, in this experiment, the wettability of wheat straw before and after plasma treatment was measured using the contact angle. The change in the contact angle is shown in Fig. 2 for the untreated and treated wheat straw surfaces after different wetting periods.

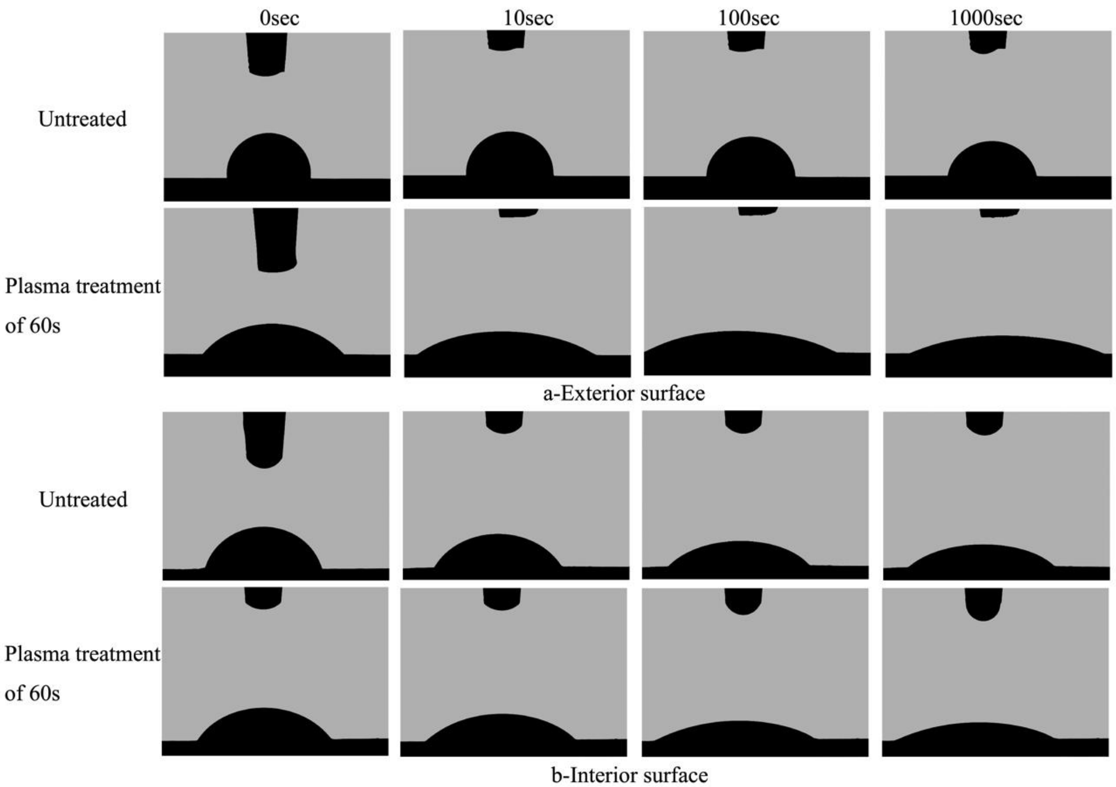

Fig. 2. Contact angle changes on the untreated and treated wheat straw surfaces after different wetting periods

Table 1. Contact Angle Values on the Untreated and Treated Wheat Straw Surfaces

Treatment time (s)	Instantaneous contact angles (°)		Equilibrium contact angles (°)	
	Exterior surface	Interior surface	Exterior surface	Interior surface
0	89.50 ± 3.32	79.15 ± 3.10	67.97 ± 3.43	38.22 ± 2.72
30	74.43 ± 2.77	62.37 ± 3.81	35.07 ± 4.45	27.14 ± 1.42
45	66.72 ± 4.48	54.17 ± 1.61	33.09 ± 3.81	27.03 ± 2.77
60	59.48 ± 0.97	53.86 ± 1.68	24.70 ± 1.02	25.07 ± 1.52
75	60.91 ± 1.33	60.14 ± 3.05	27.08 ± 2.07	27.53 ± 2.16
90	58.73 ± 2.63	58.54 ± 2.71	27.45 ± 4.81	32.00 ± 3.10

As shown in Fig. 2, the images at 0 s were taken at the moment the drop landed on the specimens, and are defined as the instantaneous contact angles. In addition, as the time elapsed to 1000 s, the drop shape tended to stabilize, reaching an equilibrium contact angle. Figure 2 shows that the instantaneous and equilibrium contact angles of the UF resin on plasma-treated wheat straw were much lower than those of the untreated specimens on both the exterior and interior surfaces. It is clear that the atmospheric pressure DBD plasma treatment improved the wettability of the UF resin on the interior and exterior surfaces of the wheat straw. In addition, as shown in Table 1, by increasing the treatment time, the instantaneous and equilibrium contact angles decreased at first and then increased. After a 60-s treatment, the contact angle decreased to a minimum. On the exterior surface, the instantaneous and equilibrium contact angles decreased by 34% and 64%, respectively, and they decreased by 32% and 34%, respectively, on the interior surface. Thus, the best wettability for wheat straw surfaces was achieved when the treatment time was 60 s.

Moreover, there was a significant difference between the contact angles on the interior and exterior surfaces of the wheat straw. As shown in Fig. 2, the instantaneous and equilibrium contact angles of the interior surface were lower than those of the exterior surface for the UF resin on the untreated and treated wheat straw. This indicates that the wettability of the interior is greater than that of the exterior because the structures of the exterior and interior surface of the wheat straw are different. The exterior surface contains many cuticles, composed mostly of cutin and wax, which prevents the resin from spreading and penetrating into the surface (Liu *et al.* 2004).

The plasma etching effect could be one of the reasons for the better wettability, which increases the roughness of the wheat straw surface and the microscopic area of contact between the liquid drop and the wheat straw. In addition, this increase in wettability is attributed to the chemical changes in the surface of straw, especially the exterior surface, which can be caused by an increase in the number of active groups after plasma treatment (Xu and Zhou 2009; Chen *et al.* 2010). As the treatment time increases, the contact time between the high-energy particles and the active groups on the wheat straw is longer; thus, the etching effect on the surface of wheat straw is more obvious, and more active groups are generated. Therefore, the wettability increases. However, the surface contact angle of the wheat straw gradually increases as the treatment time increases because the active groups added to the surface of the wheat straw could be destroyed by the high-energy particles in plasma, decreasing its surface polarity, which affects the wettability (Zhang *et al.* 2010). Therefore, when the treatment time was longer than 60 s, the plasma easily destroyed the wheat straw and its strength was sharply reduced. SEM, AFM, and XPS were used to verify the accuracy of these conclusions.

Effect of DBD Plasma Treatment on Morphology and Surface Roughness of Wheat Straw

The surface roughness has been shown to be a key factor that contributes to mechanical interlocking phenomena (Moghadamzadeh *et al.* 2011). Therefore, two different methods were employed to show the topography of the wheat straw before and after treatment with DBD plasma: SEM was used to observe the interior and exterior surfaces of the wheat straw and AFM was used to scan an area of 3 μm × 3 μm on the wheat straw fiber. Both methods clearly show the roughness of the wheat straw surfaces.

SEM observation

As shown in Figs. 3 and 4, the untreated wheat straw observed using SEM showed a smooth and dense surface. However, atmospheric pressure DBD plasma treatment turned the smooth surface into a coarse surface. On the exterior surface of the wheat straw, different degrees of sagging and cresting occurred around the lenticels, and many dot notches appeared on the interior surface. The coarse appearance and exposed inner structure are favorable for UF resin anchoring; consequently, the wettability of the treated straw was improved. In addition, by increasing the treatment time, the sags and crests around the lenticels and notches on the interior surface became more obvious. However, when the treatment time was too long (over 60 s), small holes appeared on the wheat straw surfaces (Fig. 4), due to the bombardment of high-energy particles in the plasma on the surface of the wheat straw at high speeds. These phenomena were consistent with previous results.

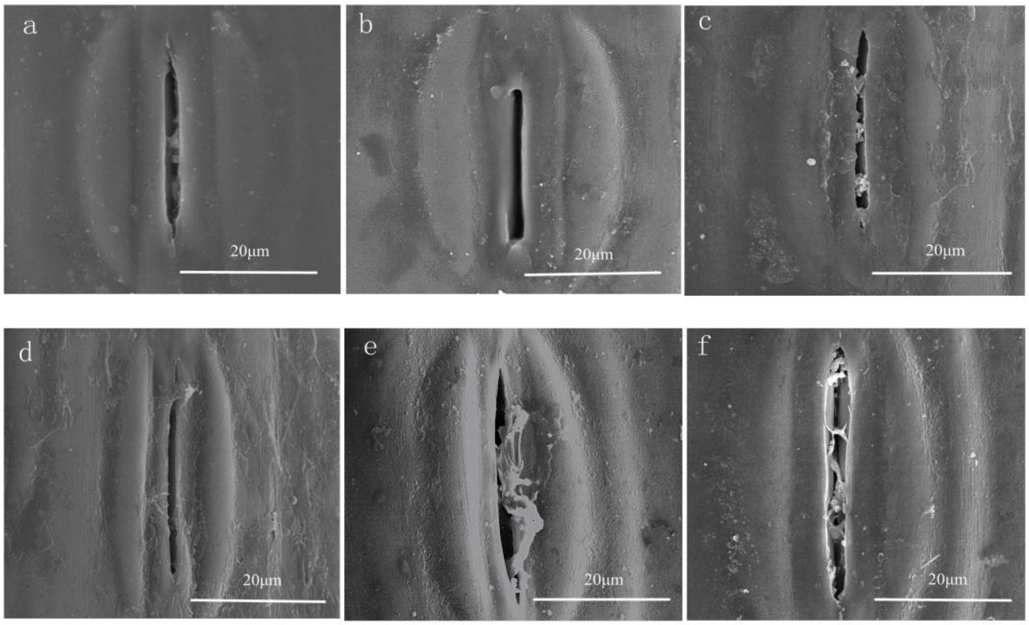

Fig. 3. Exterior surfaces of the wheat straw observed under SEM (a- untreated; b- plasma treatment of 30 s; c- plasma treatment of 45 s; d- plasma treatment of 60 s; e- plasma treatment of 75 s; f- plasma treatment of 90 s)

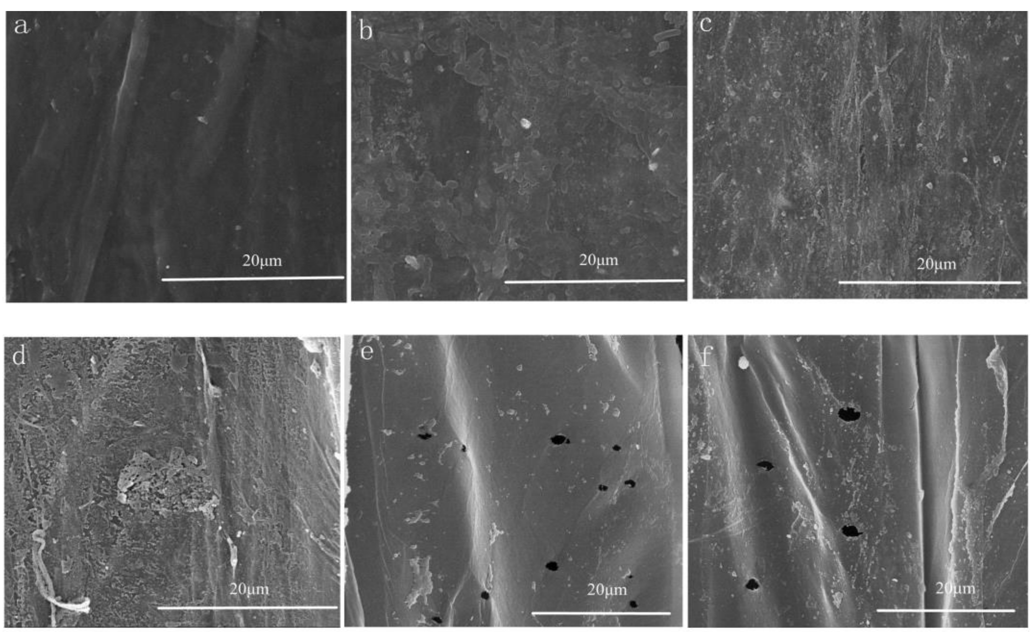

Fig. 4. Interior surfaces of the wheat straw observed using SEM (a- untreated; b- plasma treatment of 30 s; c- plasma treatment of 45 s; d- plasma treatment of 60 s; e- plasma treatment of 75 s; f- plasma treatment of 90 s)

AFM observation

According to SEM observations, the atmospheric pressure DBD plasma had an etching effect on the surface of the wheat straw, which can be approximately determined by the distribution and size of the notches on the microscopic surface. This result is still not very clear or intuitive for elucidating the influence of the atmospheric pressure DBD plasma on the surface of the wheat straw. However, AFM can provide such information with suitably high resolution. During the research, AFM was used in combination with SEM to characterize the wheat straw fibers before and after plasma treatment in three dimensions.

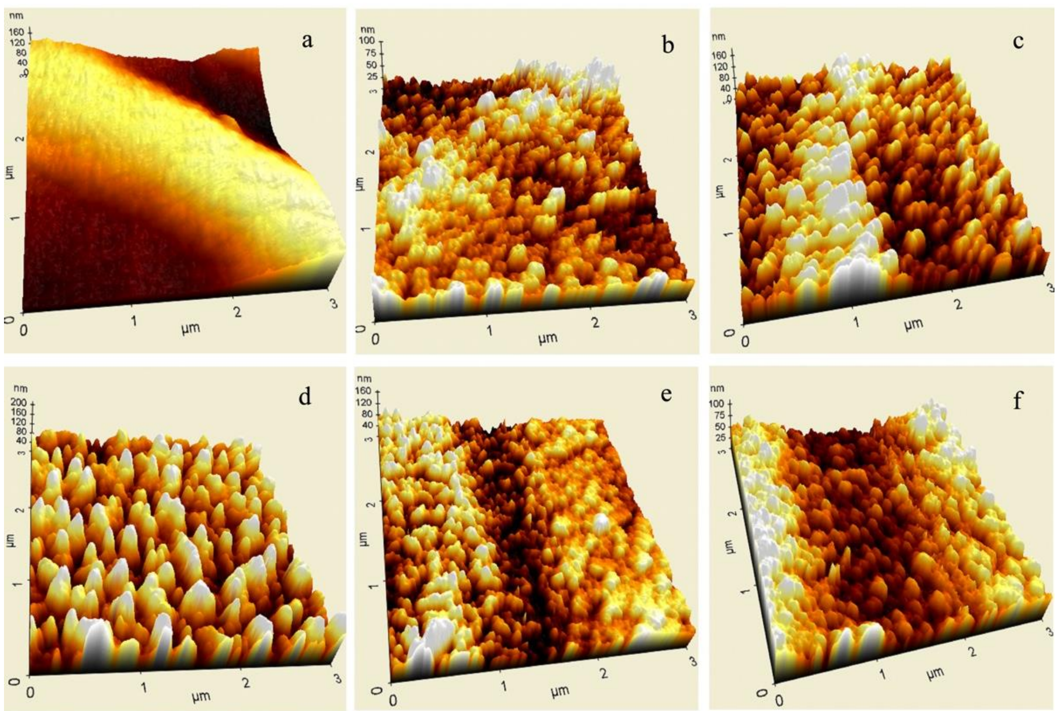

Fig. 5. 3D AFM image of wheat straw fibers (a- untreated; b-plasma treatment 30 s; c- plasma treatment 45 s; d- plasma treatment 60 s; e- plasma treatment 75 s; f- plasma treatment 90 s)

Figure 5 shows 3D images of wheat straw fibers before and after plasma treatment. The untreated wheat straw fiber surface had the shape of a smooth parabola. In contrast, the surfaces of the wheat straw fibers after plasma treatment contained many uneven pits. Moreover, the distribution and size of these indentations became more uniform as the treatment time was increased, especially for the treatment time of 60 s. However, as the treatment time was further increased, the distance between the highest point and the lowest point decreased from 200 to 100 nm, which means the surface roughness decreased as the treatment time increased. The concave and convex formations on the wheat straw fiber surfaces were flattened by the high-energy plasma. This result is consistent with the SEM observation.

After the atmospheric pressure DBD plasma treatment, uneven indentations on the wheat straw interior and exterior surfaces generated a microporous structure on the wheat straw surface, which is useful for the spreading and penetration of UF resin on the surface of the wheat straw and increases the surface wettability. Moreover, this behavior enhances the bonding performance of the wheat straw via the production of glue nails.

Effect of DBD Plasma Treatment on the Surface Chemistry of Wheat Straw

X-ray photoelectron spectroscopy was used to estimate the surface chemical compositions and functional groups of the original and plasma-treated wheat straw (Zhou *et al.* 2013). The effects of the DBD plasma treatment time on the surface chemistry of the wheat straw are shown in Fig. 6 and Table 2. They indicate that the oxygen concentration increased gradually with the treatment time, while the carbon concentration gradually decreased. The ratio of oxygen to carbon atoms (O/C) increased markedly from 0.27 to 1.88 on the exterior surface and from 0.65 to 1.85 on the interior surface with increasing plasma treatment time. However, there was a decrease in the oxygen concentration and the ratio of oxygen to carbon atoms when the plasma treatment time was longer than 60 s. These results indicate that the wheat straw surface was activated as a result of the increase in oxygen-containing functional groups. Moreover, the Si content increased at first and then decreased as the treatment time increased because the C content decreased from the exterior to the interior, but the O content always increased. The Si content was the largest in the middle section (Liu *et al.* 2002).

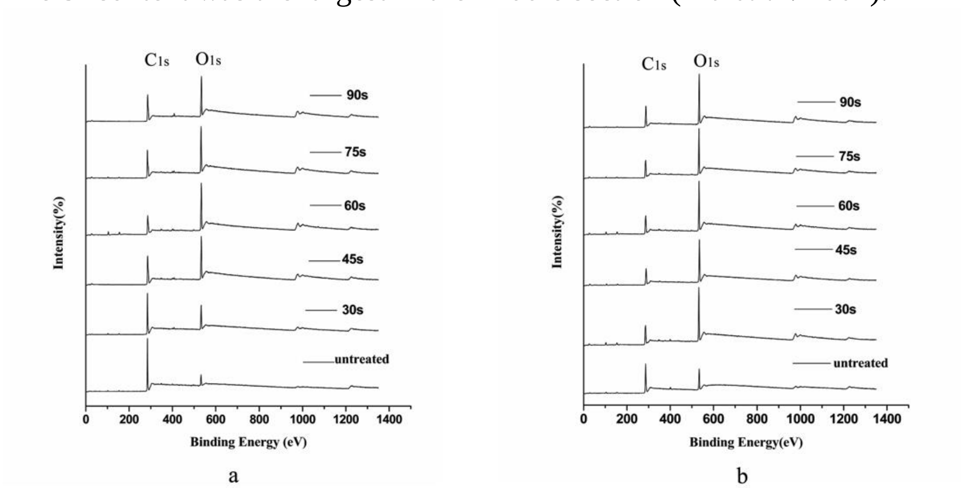

Fig. 6. XPS survey spectra of the wheat straw before and after plasma treatment (a- exterior surface; b- interior surface)

Table 2. Experimental Atomic Compositions and O/C Ratios Obtained via XPS Analysis of the Wheat Straw Surface

Treatment (s)		%C	%O	%Si	O/C
Exterior surface	Untreated	75.66	20.21	1.54	0.27
	30 s	58.11	37.77	1.55	0.65
	45 s	43.47	51.29	2.49	1.23
	60 s	32.67	61.27	3.48	1.88
	75 s	41.36	54.30	1.17	1.31
	90 s	42.24	53.19	1.30	1.26
Interior surface	Untreated	57.19	37.26	1.68	0.65
	30 s	33.68	61.42	2.44	1.82
	45 s	33.97	62.41	1.82	1.84
	60 s	33.44	61.85	3.22	1.85
	75 s	35.56	60.80	1.79	1.71
	90 s	34.45	62.49	1.74	1.81

To investigate which chemical functional groups were introduced onto the wheat straw surface after DBD plasma treatment, deconvolution analysis of the C1s peaks was performed. The C1s XPS spectra of the untreated and DBD plasma-treated wheat straw after various plasma treatment times are shown in Fig. 7 and Table 3.

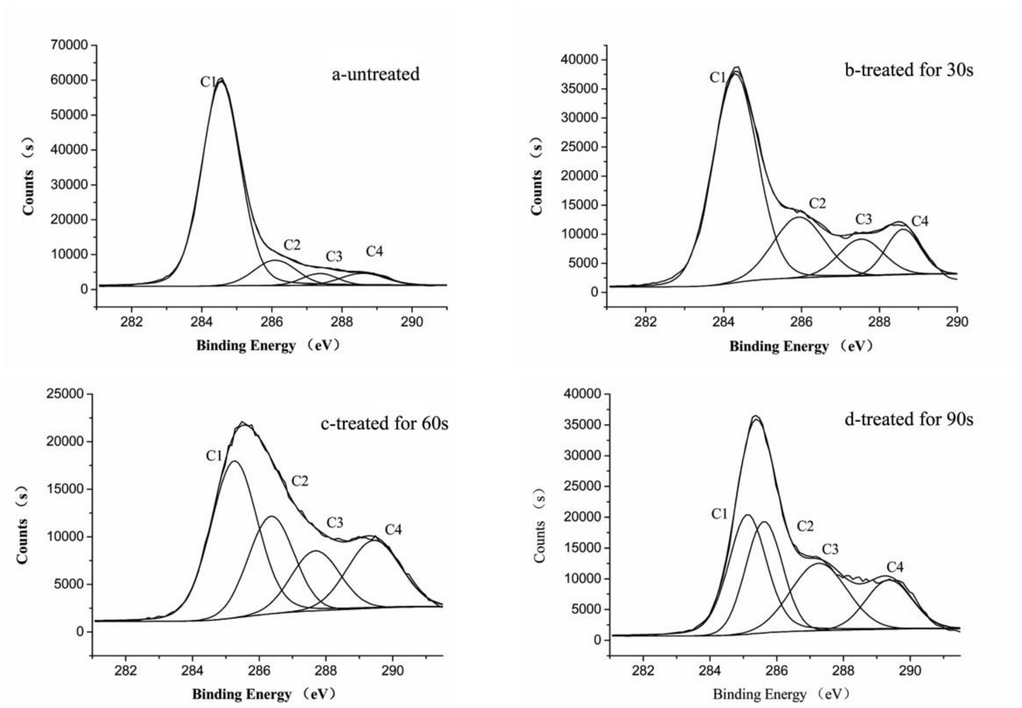

Fig. 7. The C1s peaks of the exterior surfaces of the wheat straw

Table 3. The Test Data for the C1s Peak of Treated and Untreated Wheat Straw Surfaces

Treatment Time (s)		Content (%)			
		C1	C2	C3	C4
Exterior surface	Untreated	80.89	13.56	4.90	6.60
	30 s	63.46	18.85	9.90	9.60
	45 s	52.43	20.41	11.29	15.87
	60 s	44.14	24.35	13.63	17.88
	75 s	52.28	19.84	13.51	14.72
	90 s	53.36	19.74	14.61	12.29
Interior surface	Untreated	47.79	28.32	11.21	12.69
	30 s	41.11	32.57	12.81	13.51
	45 s	42.31	30.22	13.85	13.62
	60 s	15.28	35.35	25.30	24.07
	75 s	21.71	33.00	26.91	18.39
	90 s	41.17	28.47	11.67	18.70

As can be observed in Fig. 7 and Table 3, the C1 peak, which corresponds to carbon atoms only linked to carbon and/or hydrogen (C-C, C-H), decreased significantly with increasing plasma treatment time. However, the concentrations of the C2/C3/C4 peaks, which represent the oxygen-containing functional groups such as hydroxyl, carbonyl, and carboxyl groups, increased, in particular for the DBD plasma-treated wheat straw samples after 60 s. This indicates that the introduction of functional groups to the surface of wheat straw occurred during plasma treatment (Zhou *et al.* 2013). In addition, the concentrations of the oxygen-containing functional groups decreased gradually when the plasma treatment time was greater than 60 s, which implies that 60 s of plasma treatment is sufficient for wheat straw surface modification to increase the wettability and permeation of UF resin (Chen *et al.* 2010).

Effect Of DBD Plasma Treatment on the Interfacial Adhesion of Wheat Straw with UF Resin

The results for the shear strength of wheat straw with UF resin are shown in Fig. 8. It is evident that the shear strength of the specimens with exterior-to-exterior surfaces, exterior-to-interior surfaces, and interior-to-interior surfaces of wheat straw improved dramatically after it was exposed to atmospheric pressure DBD plasma treatment, particularly for the exterior-to-exterior surface specimens, which had nearly no shear strength (only 10% glued together) before plasma treatment. In addition, the shear strength increased at first then decreased as the treatment time further increased. At 60 s, the shear strength was the highest for all sample types.

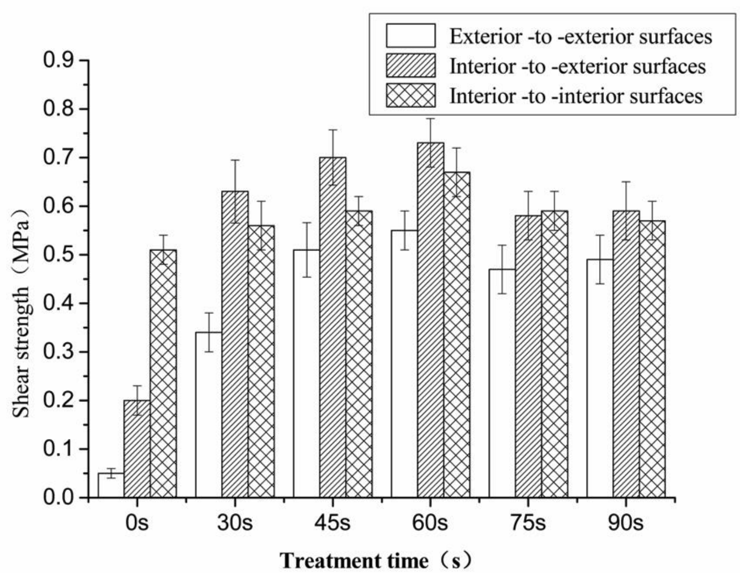

Fig. 8. Shear strength of the wheat straw

CONCLUSIONS

1. Atmospheric pressure DBD plasma treatment was found to be an effective method for improving the surface properties of wheat straw, which is beneficial for UF resin penetration into the wheat straw body and production of strong adhesion.
2. As the treatment time increased, the wettability and shear strength of the wheat straw surfaces increased at first and then decreased. The changes in the topography and chemical composition of the wheat straw surfaces are powerful evidence that confirmed this result. After 60 s of treatment, the properties of the wheat straw surface were optimal.

3. Physical etching and chemical changes were responsible for the better wettability between the wheat straw and the UF resin.

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