Effect of Plasma Processing Rate on Poplar Veneer Surface and its Application in Plywood

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Dielectric barrier discharge (DBD) plasma at atmospheric pressure in air was applied to a poplar veneer surface. Effects of plasma processing rate on surface morphology, chemical property, and surface wettability of the poplar veneer were investigated. The adhesion strength of urea formaldehyde (UF) glued plywood manufactured from the modified veneer was also studied. Atomic force microscopy (AFM), electron spin-resonance spectroscopy (ESR), X-ray photoelectron spectroscopy (XPS), contact angle tests, and shear strength tests were carried out. AFM indicated that the surface roughness increased after plasma treatment and was the maximum at a processing rate of 14 m/min. Both ESR and XPS tests showed more oxygen accumulation on the wood surface, forming various oxygen-containing chemical groups. Contact angle tests showed better wetting at a decreased plasma processing rate. Consequently, the adhesion strength of plywood increased after plasma treatment and showed higher strength at lower processing rates.

Keywords: Dielectric barrier discharge (DBD) plasma; Poplar veneer; Surface modification; Adhesion; Processing rate

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INTRODUCTION

With an increasing concern for the environment, composites obtained from natural materials are required for sustainable development (Sanghvi *et al*. 2005; Kumar *et al.* 2008; Olsson *et al*. 2010). Wood has been widely used in the manufacturing of plywood (Cheng and Wang 2011; Choi *et al.* 2011), fiberboard (Ye *et al*. 2007; Li *et al.* 2009), strandboard (Lei *et al*. 2006; Lei and Wu 2006), and wood-plastic composites (Kazayawoko *et al*. 1999) for a long time. Wood-based composites with high adhesion strength have especially attracted plenty of interest from researchers and industry.

Modification of adhesives is considered to be one of the most effective methods in improving adhesion strength (Wang and Pizzi 1997; Li and Geng 2004; Liu and Li 2007). Some researchers have modified the urea-formaldehyde wood adhesive using borax (Çolakoğlu and Demirkir 2006) or proteins (Wang and Pizzi 1997), leading to improved adhesion strength. However, the modification of adhesives usually leads to increased formaldehyde emission (Que *et al*. 2007) or higher costs (Liu and Li 2002). In recent years, surface modification was studied to improve the bonding strength of wood-based products. Kazayawoko *et al.* (1999) modified a polypropylene surface with maleic anhydride and combined it with wood fiber. Improved interfacial adhesion between wood fiber and modified polypropylene was obtained.

In plywood manufacturing, surface modification is common. Both chemical (Gardner and Elder 1988) and mechanical (Aydin 2004) surface treatments have been

reported for plywood. In recent years, plasma treatment on wood surfaces has been considered the most effective way to improve adhesion strength (Acda *et al*. 2012). Xie *et al.* (2015) roughened the wood surface to control interaction with another phase by using radio-frequency oxygen plasma. Tang *et al*. (2012, 2015) treated poplar veneer surface with radio-frequency oxygen plasma. They found that the plasma treatment would introduce polar chemical groups onto the surface, change the surface morphology, and increase the roughness, all of which improved the interfacial adhesion of the plywood product. Acda *et al*. (2012) studied dielectric barrier discharge (DBD) oxygen plasma treatment on three different wood specimens and found that *S. contorta* surfaces could be well modified, with greatly enhanced shear strength of adhesive joints. However, the reported plasma treatments on wood surfaces require either low pressure during processing (*e.g.* radio-frequency plasma) or the existence of pure oxygen (*e.g.* DBD oxygen plasma). These processing conditions would be costly for industrial processes.

In the present research, a DBD plasma generator at atmospheric pressure in air developed in our lab (Zhou *et al.* 2011) was applied to treat poplar veneer surfaces. Plasma-induced surface chemical and physical modifications of wood veneers could be achieved by passing through the gap between the electrodes. The process could be conveniently operated in air atmospheric condition, without any special requirements on gas or vacuum. Surface morphology, chemical properties, and surface wettability of the poplar veneer, as well as the adhesion strength of the plywood product, were studied as a function of the plasma processing rate. The results indicated increased adhesion strength with reduced processing rate as a result of better wetting and increased surface roughness.

EXPERIMENTAL

Materials

Commercial poplar veneers (1200 × 800 × 1.5 mm^3) were purchased from Anhui Tiankang Timber Co., Ltd., China. The moisture content of the veneers was adjusted to approximately 6% by keeping them at constant temperature and humidity for at least seven days before plasma treatment.

The urea formaldehyde (UF) resin was synthesized at 90 °C for 1 h in water solution with formaldehyde/urea mole ratio of 1.20. The solids content of the UF resin was 43.26%, with a flow-out viscosity of 25 s from a DIN 4 cup at 25 °C and curing time of 75 s.

Methods

Plasma treatment of poplar veneer

Surface treatments of poplar veneer were performed by means of DBD plasma treatment, as described in our previous patent (Zhou *et al.* 2011). The veneers were uniformly plasma-treated by passing through the gap between two electrodes on a roller conveyor. The veneers were cut to 300 × 900 mm^2 to fit the width of the electrodes. The processing power was 4.5 kW at atmospheric pressure in air, with an electrode gap of 10 mm, and the plasma processing rate was set to 2, 8, 14, or 20 m/min.

Roughness of poplar veneer

Atomic force microscopy (AFM) of the macerated poplar fibers were carried out on an XE-100 AFM (Park Systems Corp., Korea) to evaluate the surface roughness

before and after plasma treatment. The macerated fibers were obtained according to Franklin (1945), and plasma treated at the same conditions as the poplar veneers. AFM images were scanned within an area of 5 × 5 μm^2 on a mica plate. The surface roughness of the fibers was calculated by the associated software (XEI, Park Systems Corp., Korea).

Surface chemical characterization

Electron spin-resonance spectroscopy (ESR) (X-Band spectrometer, Type Elexsys ER-4150, Bruker-Biospin, Germany) was used to evaluate the free radical concentration induced by plasma treatment on wood surface, and conducted in a magnetic field range of 100 G, with an amplification factor of 10^4 and standard sample spin of 2.6×10^{14}. The spectra were collected immediately (within 15 min) after plasma treatment to minimize the influence of free radical lifetime.

X-ray photoelectron spectroscopy (XPS) was used to analyze surface oxidization by plasma, and performed on an ESCALAB 250 system (Thermo, USA) using an Al Kα X-ray source (1486.6 eV) operated at 50 eV. The XPS tests were carried out immediately (within 15 min) after plasma treatment.

Wettability of poplar veneer

Surface wetting of poplar veneer was evaluated by contact angle and surface free energy. Contact angles of UF adhesive, water, and diiodomethane on the veneer surface were tested using an Attension Theta Optical Tensiometer (Biolin Scientific, Sweden). By putting a drop of liquid with volume size of 5 μL on veneer, the contact angles were recorded every 5 s for 110 s after the liquid reached the veneer surface. The free energies of the poplar veneer surface before and after plasma treatment were calculated using the contact angles of water and diiodomethane on the poplar veneer surface according to Owens' two-liquid method (Owens and Wendt 1969). The veneer specimens were previously conditioned to a moisture content of 6% and sanded with 100-grit sandpaper before use. All tests were repeated at least 10 times.

Assembly of plywood and shear strength test

Three-layer plywood was made of poplar veneers after plasma treatment, with a UF adhesive loading level of 260 g/m^2 (double glue lines). The hot pressing time, pressure, and temperature applied were 1.3 min/mm, 1.0 MPa, and 110 °C, respectively. For each plasma treatment condition, three replicates were prepared. The adhesion strength was evaluated on a mechanical testing machine (HD-500, Shenzhen Sans Material Test Instrument Co., Ltd., China) according to the Chinese national standard for plywood (GB/T 9846.3 2004). Thirty-two specimens were tested for each group.

RESULTS AND DISCUSSION

Roughness Analysis

Increased surface roughness of wood induced by plasma etching on a scale of several micrometers have already been reported by using laser scanning confocal microscope tests (Jamali and Evans 2011; Tang *et al*. 2015; Xie *et al.* 2015). However, surface etching is difficult to observe for wood veneer, as rotary cutting makes the veneer surface terribly rough with high roughness variation (Dundar *et al.* 2008). To better compare the etching effect of plasma, poplar wood fibers were macerated according to

Franklin (1945), and the surface roughness of the macerated fibers was characterized by AFM, as shown in Fig. 1. The 3-D images in Fig. 1(A) show that the smooth surface of poplar fibers became rough after plasma treatment. By drawing a line across the 3-D image, line profiles of the surface height can be obtained (shown in Fig. 1(B)).

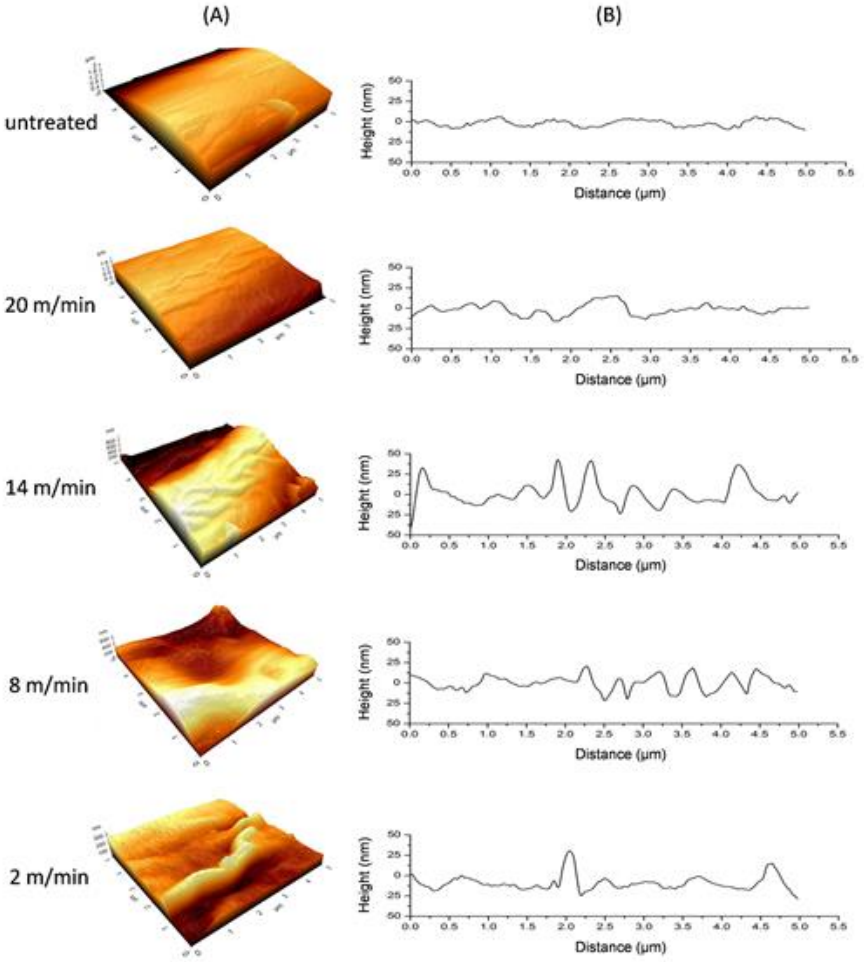

Fig. 1. (A) AFM 3-D images of poplar fiber surfaces at the indicated plasma processing rate. The line profiles (B) obtained from the corresponding 3-D images is also presented.

The line profiles exhibited the roughest surface at a processing rate of 14 m/min, and reduced roughness at a higher or lower processing rates. Also, by calculating the arithmetic mean roughness (R_a) and quadratic mean roughness (R_q) from at least eight images (presented in Table 1), maximum roughness for R_a (10.18 nm) and R_q (14.61 nm) were present at 14 m/min. The high energy eximers were generated by plasma (Shen *et al*. 1998; Bhat and Upadhyay 2002; Tang *et al*. 2015), and struck the wood surface. As a result, the wood surface was etched and became rougher. At a higher processing rate, shorter processing time made the surface etching less severe. At a lower processing rate, the plasma etching made the cell wall become thinner (Jamali and Evans 2011). Consequently, the surface roughness decreased compared to that at 14 m/min. The significant level p-value was smaller than 0.05 by using ANOVA analysis, indicating the numerical differences were statistically significant.

Table 1. Surface Roughness Obtained from AFM Images (errors are indicated in the parenthesis)

Plasma processing rate (m/min)	Ra (nm)	Rq (nm)
Untreated	3.29 (0.90)	4.05 (1.03)
20	5.32 (1.77)	6.69 (1.95)
14	10.18 (3.11)	14.61 (5.01)
8	8.70 (2.01)	12.39 (6.63)
2	6.27 (1.68)	8.89 (2.28)

Chemical Analysis of the Poplar Surface

Surfaces can be both chemically modified (Tang *et al*. 2015) and etched (Jamali and Evans 2011) by plasma treatment. In our research, chemical changes were evaluated by free radical concentration and atomic composition. Free radical concentration on the poplar surface was evaluated by ESR spectra, which are shown in Fig. 2 as a function of the DBD plasma processing rate. During plasma treatment, air was excited in terms of atoms, molecules, ions, free radicals, and electrons (Shen *et al*. 1998; Bhat and Upadhyay 2002; Tang *et al*. 2015). The excited particles attacked the wood surface, generating free radicals through absorption and proton exchange (Fischer 1965). With reduced plasma processing rate, the poplar veneer was exposed to the plasma for a longer time. As shown in Fig. 2, the free radical concentration increased at a slower processing rate. The excited particles accumulated on the wood surface and generated more free radicals with a longer exposure time.

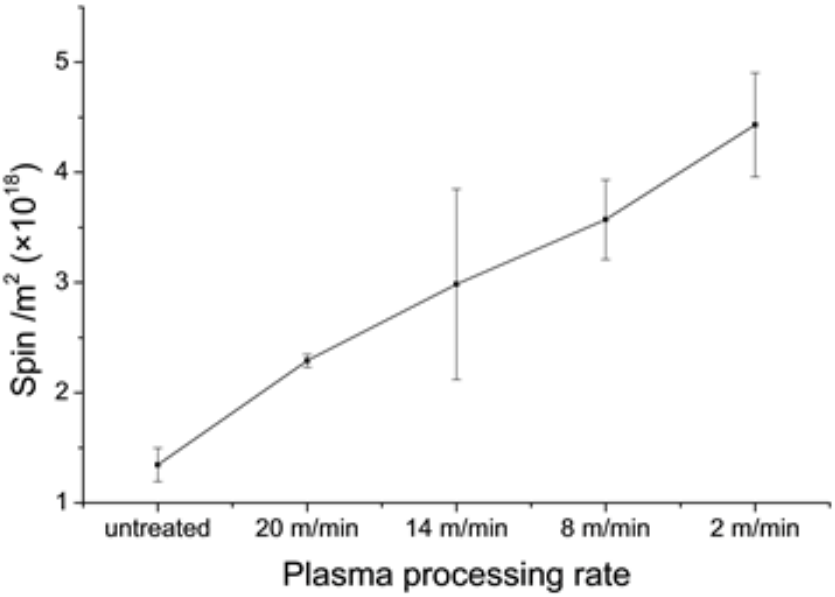

Fig. 2. Free radical concentration on poplar veneer surface as a function of DBD plasma processing rate

As oxygen in air is easily excited, the accumulated free radicals were primarily oxygen-based (Kossyi *et al.* 1992; Tang *et al*. 2015). The excited oxygen-based free

radicals would introduce more oxygen-containing groups on the wood surface. Figures 3a and 3b through 3f are low-resolution XPS spectra and high-resolution C1s spectra for the samples. The appearance of peaks at 284 to 290 eV and 532 eV in the low-resolution spectra indicated the existence of oxygen and carbon on the poplar wood surface. By comparison with the atomic composition shown in Table 2, the content of carbon decreased after plasma treatment, while the content of oxygen increased. A slower plasma processing rate led to lower carbon contents and higher oxygen contents. The content changes of carbon and oxygen indicated oxygen accumulation during plasma treatment. The accumulated oxygen-based excimers reacted with poplar wood and resulted in an increasing number of oxygen-containing chemical groups.

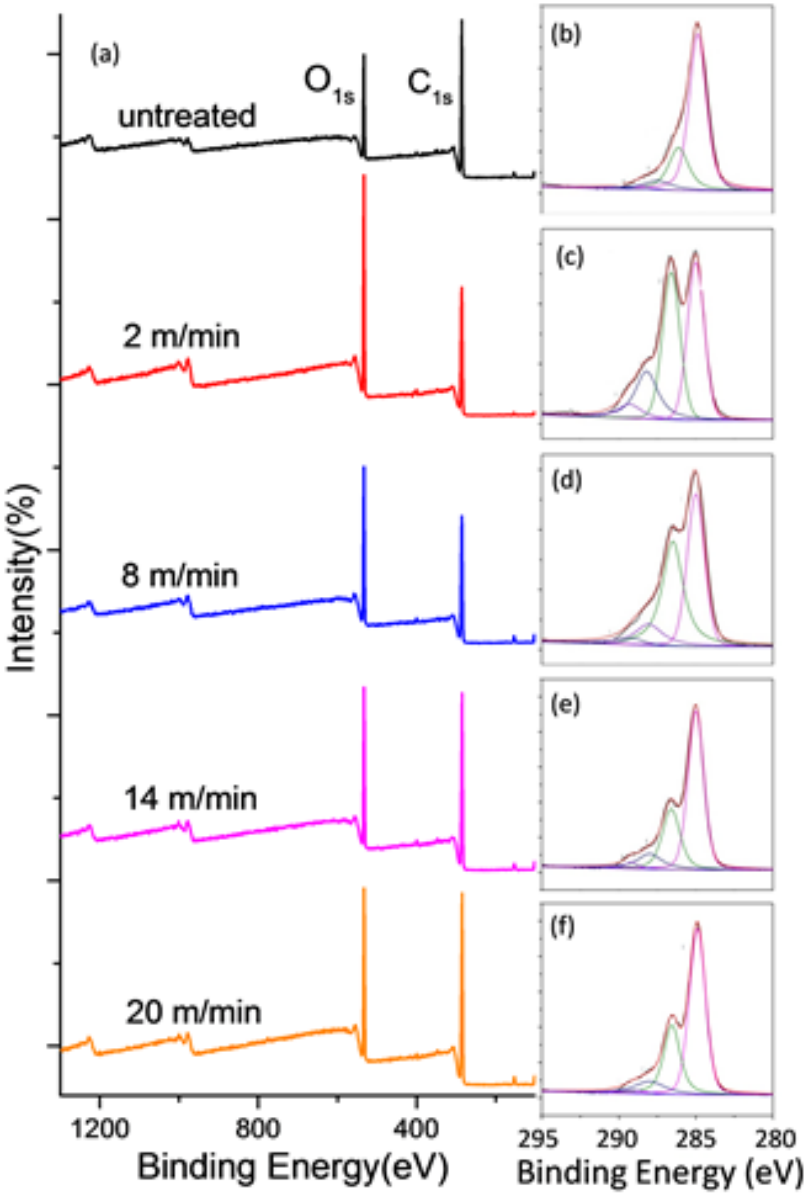

Fig. 3. (a) Low-resolution XPS spectra before and after plasma treatment, and high-resolution XPS spectra of C1s for (b) the untreated specimen and (c to f) specimens plasma-treated with processing rates of 2, 8, 14, and 20 m/min, respectively. All high-resolution spectra were de-convolved.

By deconvolving the high-resolution C1s spectra in Fig. 3b through 3f, the carbon peak could be separated into four groups: C1 for C-C/C-H; C2 for C-O; C3 for C=O, and C4 for O=C-O (Klarhöfer *et al*. 2010; Tuong and Li 2011; Tang *et al*. 2015). Comparing the four types of carbons in Table 3, the plasma processing rate has a great influence on C1's composition. The composition of C1 decreased after plasma treatment, while C2, C3, and C4 increased. With decreasing processing rate, C1 composition fell from 66.84% to 37.15%, while C3 and C4 compositions increased from 8.02% and 1.47% to 20.08% and 6.27%, respectively. The C2 composition increased from 23.66% to 44.20% when the processing rate slowed down to 8 m/min. However, when the processing rate further decreased to 2 m/min, the C2 composition dropped from 44.20% to 36.51%. The changes in the C1s compositions indicated that the alkyl groups on poplar wood were oxidized to oxygen-containing groups during plasma treatment (Chang *et al.* 2010). At a fast processing rate, the alkyl chains on poplar wood converted to alcohol, ether, ketone, acid, ester, and other oxygen-containing groups, through a reaction with oxygen-based excimers. At a slower processing rate, the C2 groups (alcohol, ether, *etc*.) would be further oxidized to more oxidized groups, like ketones, acids, and esters. In this way, the surface chemical properties of poplar wood could be controlled by adjusting the plasma processing rate.

Table 2. Experimental Atomic Composition (%) and O/C Ratio Obtained by XPS Analysis

Plasma processing rate (m/min)	%C	%O	O/C
Untreated	79.33	19.19	0.24
20	74.55	24.09	0.32
14	74.35	24.15	0.33
8	70.98	27.27	0.39
2	66.07	32.33	0.49

Table 3. C1s Composition Obtained by XPS Analysis

Plasma processing rate (m/min)	Binding energy (eV)				Composition (%)			
	C1	C2	C3	C4	C1	C2	C3	C4
Untreated	285.20	286.53	288.00	289.10	66.84%	23.66%	8.02%	1.47%
20	284.90	286.55	288.00	289.08	58.15%	28.90%	10.61%	2.34%
14	285.00	286.60	288.00	289.18	57.99%	27.72%	11.80%	2.49%
8	285.00	286.50	288.06	289.00	40.73%	44.20%	12.61%	2.46%
2	285.00	286.60	288.20	289.30	37.15%	36.51%	20.08%	6.27%

Evaluation of Surface Wettability

Surface chemical changes usually result in surface free energy changes (Wolkenhauer *et al.* 2008), and a higher surface free energy indicates better wettability. The surface free energy can be divided into a polar component and a dispersion component. In the present research, surface free energies of poplar veneers before and after plasma treatment, as well as their components, were measured according to the Owens two-liquid method (Owens and Wendt 1969), as shown in Fig. 4. After plasma treatment, both the polar component and the dispersion component increased. With a decreasing processing rate, the polar component increased continuously. As discussed above, more oxygen-containing groups were introduced onto the wood surface at a

reduced processing rate. As oxygen is highly electronegative, the introduced chemical groups were largely of high polarity and resulted in an increased polar component. As presented in Fig. 4, as the processing rate was decreased, so was the dispersion component of surface free energy. The dispersion component of surface free energy is related to the polarizability of the outermost electrons at the surface (Hubbe *et al.* 2015). Lignin is considered to have higher contribution to the dispersion component in wood, due to the delocalization of electrons within the aromatic rings (Shen 2009). At a fast processing rate, plasma etching may decompose hemicellulose in wood first, because of its lower pyrolysis temperature (Gasparovic *et al.* 2010). As a consequence, higher relative content of lignin increased the dispersion component of surface free energy. With reduced processing rate, plasma may further decompose lignin, and reduced the dispersion component.

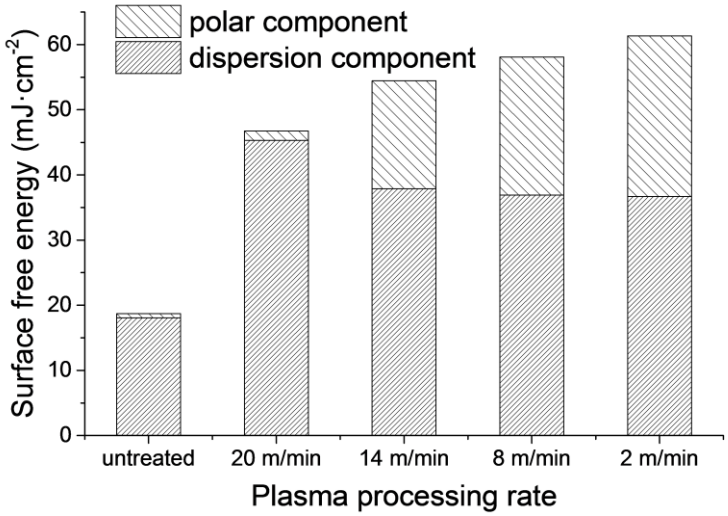

Fig. 4. Surface free energy and its components on poplar veneer surface at indicated plasma processing rates

Shown in Fig. 5 are the contact angles of UF on poplar surfaces as a function of the plasma processing rate. In the present research, the initial angle is defined as the contact angle at the moment the UF droplet reaches the veneer surface, and the equilibrium angle is obtained when the contact angle changing with time became a constant. For all the tests, the UF adhesives came to an equilibrium in 1 min, and the absorption and evaporation of solvent was not taken into account. Surface wettability was evaluated by the equilibrium angles. A lower equilibrium angle indicates better wetting on the surface. After plasma treatment, better surface wetting could be observed. When the processing rate decreased to 14 m/min, both increased surface oxidation and surface roughness made the equilibrium angle drop quickly from 43.05° to 25.90°. When the processing rate further decreased to 2 m/min, the equilibrium angle slightly decreased, from 25.90° to 22.85°. According to Wenzel's model of surface wetting, both the surface chemical properties and the surface roughness affect wetting on a rough surface. The contact angle on a rough surface follows the following equation (Wenzel 1936),

$$\cos \theta^* = r \cos \theta \quad (1)$$

where θ^* is the apparent equilibrium contact angle; r is the ratio of the true area to the apparent area of the surface, representing the surface roughness, and is always larger than

1; and θ is the contact angle of the material with an ideal flat surface. For a hydrophilic surface, cos θ ranges between 0 and 1, and θ^* decreases with larger r. As a result, a rough surface would increase the hydrophilicity of an ideal hydrophilic surface. The wettability of an ideal surface can be evaluated by the surface free energy and increases with a decreasing processing rate, as shown above. From the AFM analysis, the wood surface roughness was the maximum at a processing rate of 14 m/min. When the processing rate decreased from 20 to 14 m/min, both ideal wettability and roughness increased the apparent surface wetting, resulting in a rapid decrease of the equilibrium angle. When the processing rate was further decreased from 14 to 2 m/min, the increased ideal wettability increased, while the reduced surface roughness provided a decreased r. These opposing effects caused the θ^* to slightly decrease, as shown in Fig. 5.

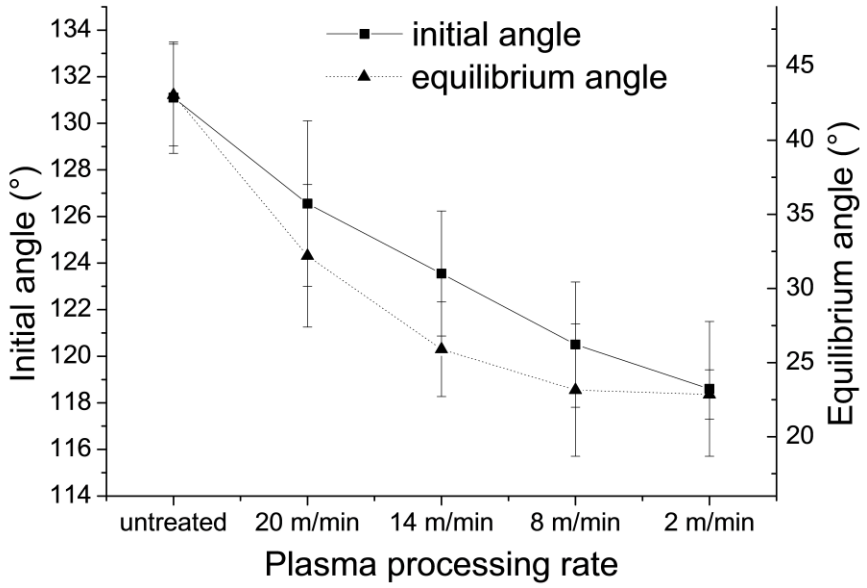

Fig. 5. The initial and equilibrium contact angles of UF on poplar veneer surface at indicated plasma processing rates

Evaluation of Adhesion Strength

Poplar veneer is used for manufacturing plywood in the wood industry. As shown above, the plasma-treated poplar veneer presented high surface free energy and good UF wetting. Therefore, the adhesion strength of UF-glued poplar plywood was tested according to GB/T 9846.3 (2004). Shown in Fig. 6 is the adhesion strength as a function of the plasma processing rate.

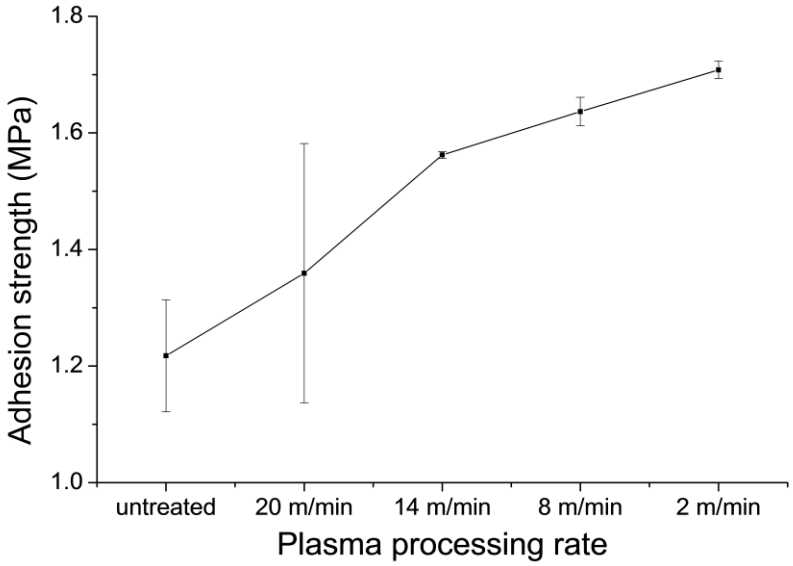

Fig. 6. Adhesion strength of poplar plywood at indicated plasma processing rates

The adhesion strength increased from 1.22 MPa for the untreated plywood to 1.36 MPa after plasma treatment at 20 m/min, and continuously increased to 1.71 MPa when the plasma processing rate decreased to 2 m/min. The relatively large errors for untreated and 20 m/min samples were probably due to the large variation of wood. The increased adhesion strength could be interpreted by the wettability and roughness. After plasma treatment, the UF wetting on poplar veneer surface increased and the UF adhesive could be better spread and absorbed on the veneer. At the same time, the surface roughness increased after plasma treatment, and the absorbed adhesive could better penetrate into the wood surface. After curing, the absorbed adhesive cured at the interface and strengthened the adhesion surface.

CONCLUSIONS

1. The UF-glued plywood presented increased adhesion strength after plasma treatment, and strong adhesion was present at a low processing rate. The mechanism responsible incorporated aspects of surface roughness change, chemical change, and wettability change. This work presents an effective method to improve the adhesion strength of plywood and may be of practical use to the industry.
2. The roughness increased after plasma treatment, and the R_a increased from 5.32 to 10.18 nm when the processing rate decreased from 20 to 14 m/min; further decreasing the processing rate to 2 m/min made the R_a drop to 6.27 nm. The 14 m/min exhibited the highest roughness, which would be beneficial for surface wetting.
3. Chemical analysis suggests that oxygen-containing groups were introduced to the veneer surface, and the oxygen content increased at a lower processing rate. At a medium processing rate (8 m/min), large amounts of C2 (from alcohol and ether) were observed, and increasing amounts of C3 and C4 (from acids and esters) appeared at 2 m/min.

4. At reduced plasma processing rates, the modified surface showed an increasing surface free energy, where the polar component increased and the dispersion component decreased. As a result, the wettability of UF adhesive on poplar surfaces also increased at a decreasing processing rate.

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