DYNAMIC ADHESIVE WETTABILITY OF POPLAR VENEER WITH COLD OXYGEN PLASMA TREATMENT

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Effects of cold oxygen plasma treatment on activating the surface of poplar veneers and improving its wettability were investigated. The veneers were treated with cold oxygen plasma for 1, 3, 5, 7, and 9 min, and aged in air for 1, 3, 7, 14, 21, and 28 days. The dynamic adhesive wettability of veneers was assessed using the contact angle, *K-value* analysis, and surface free energy. The shear strength of three-layer panels produced from untreated and cold oxygen plasma treated veneers was examined. The results showed that the wettability of veneer was significantly improved after cold oxygen plasma treatment, leading to the enhancement of shear strength of panels. The optimized treatment time should be 7 min. Aging effect of treated veneers showed that the veneer surface wettability degraded within the first 7 days and thereafter changed slightly.

Keywords: Wettability; Contact angle; Cold oxygen plasma treatment; Poplar veneer; Shear strength

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INTRODUCTION

Veneer-based composites, such as plywood and laminated veneer lumber, outperform all substitute wood-based panels on the market today. However, raw materials with a large diameter for veneer-based panel manufacture are limited due to increasing environmental concerns. Some raw materials with low quality and with resin or gum on the surface, which have adverse effects on bonding, are being used for producing panels. In addition, drying veneer with high temperature for saving energy, as well as carbonization treatment of veneers for producing panels with high dimension stability, will decrease the bonding strength of panels significantly. Therefore, in order to obtain high performance products with these raw materials, surface modification of veneers is necessary.

Some of the most important surface modification techniques, including chemical treatments, mechanical treatments (*e.g.*, sanding and planning), physical treatments (*e.g.*, flame treatment, microwave plasma, corona discharge, vacuum, and low pressure plasma), and heat treatments are widely applied to activate wood surfaces for glue-wood bonds (Rowell *et al*. 1986; Rehn *et al*. 2003; Blanchard *et al.* 2009; Uysal *et al.* 2009; Wolkenhauer *et al*. 2009). Among them, chemical treatment is the most common and effective method. However, the method is costly and results in leaching of toxic materials to the environment. In recent years, increasing concerns about environmental pollution have limited the wide industrial application of chemical surface treatments (Aydin and Demirkir 2010).

Plasma, as the fourth state of matter, has been widely used for modifying the surface of materials. Plasma treatment on wood surface has been investigated, and results have shown that the surface chemical composition is changed effectively, so that the surface wettability and adhesion properties are improved accordingly (Avramidis *et al*. 2011; Aydin and Demirkir 2010; Odraskova *et al*. 2008; Podgorski *et al*. 2000; Wolkenhauer *et al*. 2007). Aydin *et al*. (2010) observed that the surfaces of spruce wood were reactivated with low pressure plasma treatment. Wettability, bonding, and other mechanical strength properties of plywood panels increased. Podgorski *et al*. (2000) reported that the wettability of wood surface was improved to increase the coating adhesion by means of selecting a treatment parameter, such as a type of gas, treatment duration, and power. Liu *et al*. (2010) proved that the wettability of wood/PE composites can be improved by low-pressure glow discharge of air plasma treatment. Acda *et al*. (2012) reported that dielectric barrier discharge was used for wood surface modification to improve adhesion properties. Cold plasma treatment induces physical and chemical changes to the depth of a few micrometers on the wood surface without changing the bulk properties (Chu *et al*. 2002). Cold oxygen plasma is commonly employed to improve wood surface properties. It has been reported that cold oxygen plasma can react with wood to produce a variety of oxygen functional groups such as C—O, C=O, O—C=O, C—O—O, and CO_3 at the surface (Belgacem *et al*. 1995; Klarhofer *et al*. 2010; Yuan *et al*. 2004). Sakata *et al*. (1993) found that an increase in the wettability of plasma-treated wood veneer resulted mainly from the oxidation of the highly hydrophobic surface layer of neutral fraction substances in the extractives, and from the reduction in their hydrophobicity. The advantage of plasma treatment is its clean reaction and short processing time (Liu *et al*. 2010). It represents an efficient, clean, and economic alternative for modification of wood surfaces (Polini and Sorrentino 2003).

Wettability is an essential property of a wood surface, and it influences bonding properties directly. The aim of this study was to investigate the effect of the cold oxygen plasma treatment time on the dynamic adhesive wettability of poplar veneers and the shear strength of panels. The aging behavior of poplar veneers after cold oxygen plasma treatment was also evaluated.

EXPERIMENTAL

Materials

Poplar (*Populus* spp.) veneers were provided by Anhui Tiankang Wood Co. Ltd. Samples without defects or impurities were of the size 10 mm × 3 mm and 1.5 mm thickness. Before plasma treatment, the veneer samples were dried in a vacuum-drying oven at 60°C to a moisture content of 2%. All samples were kept in a desiccator to balance the moisture content.

Urea-formaldehyde (UF) resin from Jiangsu Santai Wood Co. Ltd. was used for evaluating the dynamic adhesive wettability of poplar veneers. Typical properties are shown in Table 1.

Table 1. Typical Physical and Chemical Properties of UF Resin

Specification	UF
Color	Opalescent to Maroon
Solid Content (%)/2h,120℃	49.7
pH Value/20℃	7.2
Viscosity (s)/20℃	19

Cold Oxygen Plasma Treatment

Plasma treatments of veneers were carried out in a plasma reactor (HD-1B, made in Jiangsu, P.R. China) as shown in Fig. 1 with a radio frequency of 13.56 MHz. The plasma volume was 870 cm^{-3}, and the power density in the plasma was 0.23 W • cm^{-3}. Veneers were placed between the electrodes vertically. After the reactor chamber was closed, the system was evacuated to a base pressure level of 1 to 5 Pa in about 3 min, followed by repressurizing cycles with oxygen fed directly into the chamber at a flow rate of about 15 to 20 mL/min and evacuating to the base pressure for removal of any volatile contaminants. Five repressurizing cycles were used. By operating the reactive-gas feeding system valves, the preselected pressure of 15 Pa and steady-state flow rate were created in the reactor. A radio frequency (RF) magnetron sputtering unit was used to produce oxygen plasma. The input power was set at 200 W, which was determined according to our previous paper (Zhang *et al*. 2010) and sustained for a predetermined period of 1, 3, 5, 7, and 9 minutes, respectively. The temperature was 20 to 50 ^{o}C inside the chamber during plasma treatment. After each treatment, Argon gas was fed into the chamber for 10 min to remove traces of extractives inside the chamber. At the end of the reaction, the chamber was pressurized, and the samples were removed and stored in a desiccator for dry conditions for later analysis. Contact angle measurements should be completed in 5 min after plasma treatment.

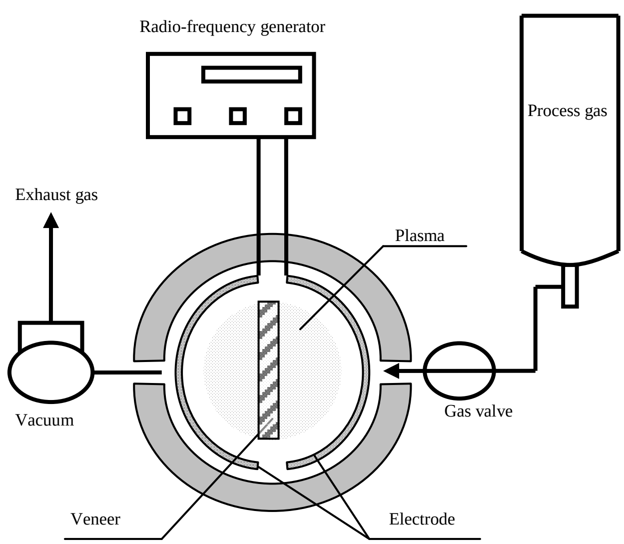

Fig. 1. The schematic diagram of RF plasma reactor

Contact Angle Measurements

The contact angle was defined as the angle through the liquid phase formed between the surface of a solid and the line tangent to the droplet radius from the point of contact with the solid (Ayrilmis *et al.* 2009). To determine the contact angle and the surface free energy of the cold oxygen plasma-treated and untreated poplar veneers, drops of distilled water, diiodomethane, and UF were uniformly dispersed on the veneer (4 mm × 10 mm in size). Surfaces of the side without checks along the grain direction with contact angles were tested by JC2000D Contact Angle Measuring Apparatus (CAA, made in Shanghai, P.R. China). Twenty replicates were tested for each group. The image of each single drop was acquired by a video camera connected to a computer. Contact angle was measured using the imaging software package (JC2000-USB). When a drop of liquid was placed on the veneer surface, a contact angle was formed immediately at the solid and liquid interface, and the result was defined as the "initial contact angle". When an almost constant rate of change of the contact angle is reached, a well-defined contact angle value is determined, which is defined as the "equilibrium contact angle" (Nussbaum 1999). The contact angles of distilled water and diiodomethane were measured starting immediately upon placement of the drops on the wood surface, and the surface free energy was calculated. Due to the high viscosity of UF resin, the drop on the surface of the veneer became smaller gradually. The contact angle of UF resin was measured at 0, 10, 20, 40, 60, 80, and 100 s after it dropped on the veneer surface. The spreading and penetration rate can be quantified by the following wetting model developed by Shi (Shi and Gardner 2001),

$$\theta = \frac{\theta_i \theta_e}{\theta_i + (\theta_e - \theta_i)\exp\left[K\left(\frac{\theta_e}{\theta_e - \theta_i}\right)t\right]} \qquad (1)$$

where θ_i and θ_e represent the initial contact angle and the equilibrium contact angle, respectively, and K is the contact angle change rate constant.

Plywood Manufacture

Three-layer-plywood panels were produced from cold oxygen plasma-treated and untreated veneers for evaluating the bonding strength. In each case, three rotary-peeled poplar veneers with the size of 250 mm × 250 mm × 1.5 mm and the moisture contents of 2.1% and UF resin were used.

Ammonium chloride (NH_4Cl) was used as a hardening agent at 0.5 wt % based on the solid content of resin. Wheat flour as an additive was put into the liquid adhesive at 30 wt %. Approximately 130 g/m^2 resin content (single glue line) was applied on the veneer surface. Hot pressing time and pressure were 4.5 min and 1.0 MPa, respectively at 110 ^{o}C in the manufacturing of panels.

After hot-pressing, panels were stored at an ambient environment for at least 24 h before being tested. For each condition, three replicates were produced.

Shear Strength of Plywood

Prior to the evaluation of the mechanical properties, the panels were conditioned at 25°C and 65 ± 5% relative humidity (RH) until the panels reached equilibrium moisture content for properties examination. The panels were characterized according to the Chinese National Standard for Plywood (GB/T 9846-2004) for shear strength. Panels were measured with a soak test in accordance with GB/T 17657-1999. The panels were soaked in water at (63±3) °C for 3 h. All panels were inspected to see whether they were delaminated, and then they were cooled down with tap water. The shear strength of the panels was determined with a mechanical testing machine (SANS, made in Shenzhen, P.R. China) while they were still wet. The crosshead speed was 1.0 mm/min. Thirty-two samples were tested for each group.

Aging Behavior Evaluation

Aging behavior of the cold oxygen plasma-treated veneers was evaluated by the surface free energy after being stored for some days. The veneers were treated for 7 min with a plasma power of 200 W. After the treatment, the veneer samples were stored in air at room temperature. The aging times were 1, 3, 7, 14, 21, and 28 days.

RESULTS AND DISCUSSION

Effect of Cold Oxygen Plasma Treatment on Dynamic Adhesive Wettability of Poplar Veneer

Contact angle analysis is widely used to evaluate the wettability of solid materials (Aydin 2004; Owens and Wendt 1969; Gindl *et al*. 2001). However, wood, as a complex composite material, has a heterogeneous, rough, and porous surface. It is not comprehensive to evaluate the wettability of wood surface only from the contact angle. Therefore, contact angle, *K*-value analysis, and surface free energy were used to determine the dynamic adhesive wettability of veneer surfaces in this study.

Contact Angle

Untreated and treated poplar veneers presented different dynamic adhesive wetting behavior at different wetting periods. When a drop of UF resin was placed on the veneer surface, at the initial stage, a contact angle was formed at the solid and adhesive interface. Then the adhesive was spread over the veneer surface. It penetrated into the veneer until the equilibrium contact angle was obtained. The initial and equilibrium contact angles of UF resin on untreated and treated veneer surfaces are shown in Fig. 2. It was found that initial and equilibrium contact angles decreased by 6.7 % and 48.2 % after oxygen plasma treatment for 1 min, respectively. Then they continued to lessen after veneers were treated from 1 min to 7 min. After being treated for 7 min, there was a slight change in the initial and equilibrium contact angles.

K-value

The rate of contact angle change, *K-value*, is related to the rate of the adhesive penetration and spreading on the solid surface. The higher the *K-value*, the faster the contact angle reaches equilibrium and the better the veneer is wetted.

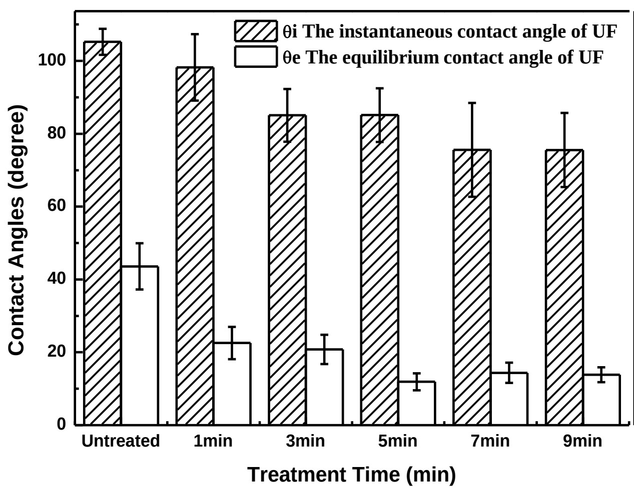

Fig. 2. The instantaneous and equilibrium contact angle values of UF resin

As shown in Fig. 3, the contact angle decreased as a function of wetting time. At the initial stage of the wetting process, the contact angle decreased quickly. After approximately 20 seconds, it changed smoothly and finally attained relative equilibrium. It can be also seen that the wetting model provided an excellent fit to the experimental data. The R^2 values of the wetting model were close to 0.99 for all samples. The *K-value* increased dramatically after oxygen plasma treatment. It was approximately 4 times greater than that of the untreated veneer. This proved that the spreading and penetration of UF resin on the treated veneer surface were significantly improved. The *K-value* increased from 0.260 (L/s) to 0.293 (L/s) after being treated for 1, 3, and 5 min. However, it was slightly smaller after being treated for 7 and 9 min.

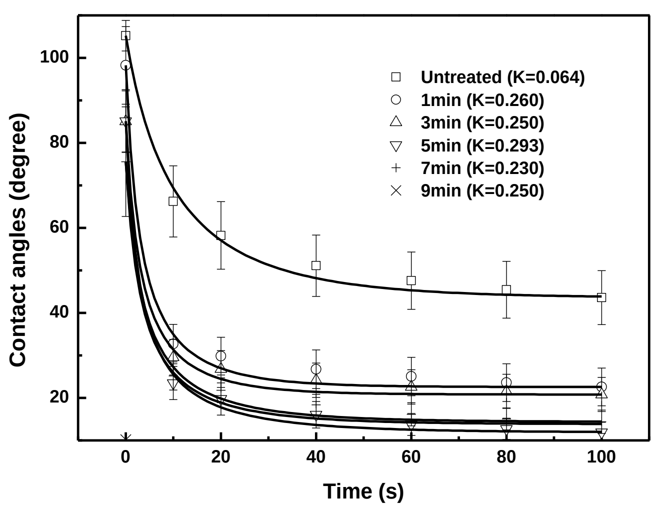

Fig. 3. The contact angle values of UF change as a function of wetting time

Surface Free Energy

The Owens-Wendt method (Owens and Wendt 1969) was used to estimate veneer surface energy. The surface free energy was calculated from the contact angle values of distilled water (polar liquid) and diiodomethane (non-polar liquid), using the geometric mean equation combined with Young's equation (Owens and Wendt 1969),

$$\gamma_{lg}(1+\cos\theta) = 2\left[\left(\gamma_{lg}^{d}\gamma_{sg}^{d}\right)^{1/2} + \left(\gamma_{lg}^{p}\gamma_{sg}^{p}\right)^{1/2}\right] \tag{2}$$

where γ_{lg} and γ_{sg} are the surface energies at the liquid-gas and solid-gas interfaces, respectively. The quantity θ is the contact angle, and the superscripts d and p represent the dispersive and polar components for the liquids. The dispersive and polar components for the liquids used in the calculations are given in Table 2 (Winfield *et al.* 2001).

Table 2. Surface Energy Components of Wetting Liquids (mJ/m^2)

Liquid Type	Surface Energy (mJ/m^2)		
	γ^{p}	γ^{d}	γ^{total}
Distilled Water	51	21.8	72.8
Diiodomethane	1.3	49.5	50.8

Surface free energy of veneers influences the reactivity and interfacial properties of the veneers deeply (Zhang *et al*. 2008). Effects of the cold oxygen plasma treatment on the surface free energy and its components of veneers are shown in Table 3 and Fig. 4. The results show that the total surface free energy of treated veneers was increased by about 55%. The trend of the total surface free energy was consistent with the increasing polar component, and the dispersion components decreased after cold oxygen plasma treatment. The surface free energy and its polar components kept increasing with the duration of treatment time from 1 min to 7 min. They reached a plateau after time periods up to 7 min. The results were in accordance with previous findings (Wolkenhauer *et al*. 2007). The increasing polar component is responsible for wettability enhancement. Some polar or oxygen-containing groups could be implanted on the veneer surface during plasma treatment (Belgacem *et al*. 1995; Klarhofer *et al*. 2010).

Table 3. The Contact Angles (°) of Distilled Water and Diiodomethane of Veneer

Treatment Time	Contact Angle (°)	
	Water	Diiodomethane
Untreated	76.53 (7.13)	27.06 (3.70)
1 min	25.31 (3.69)	25.21 (3.05)
3 min	16.74 (3.22)	16.41 (3.08)
5 min	18.80 (3.41)	16.88 (2.21)
7 min	15.70 (2.8)	15.95 (3.02)
9 min	14.07 (2.73)	14.88 (2.93)

The surface wettability had been improved. One explanation is that the cold oxygen plasma treatment introduced some polar or oxygen-containing groups and produced some new contact sites on veneer surfaces because of chemical reactions and plasma etching (Zhang *et al*. 2009; Jamali and Evans 2011). The wettability increases as the cold oxygen plasma treatment time increases. It becomes stable from a treatment time of 7 min. The results agree very well with the observations of Acda *et al*. (2012) and Ma *et al*. 2010). The improvement of veneer surface wettability implies that plywood shear strength will increase as well.

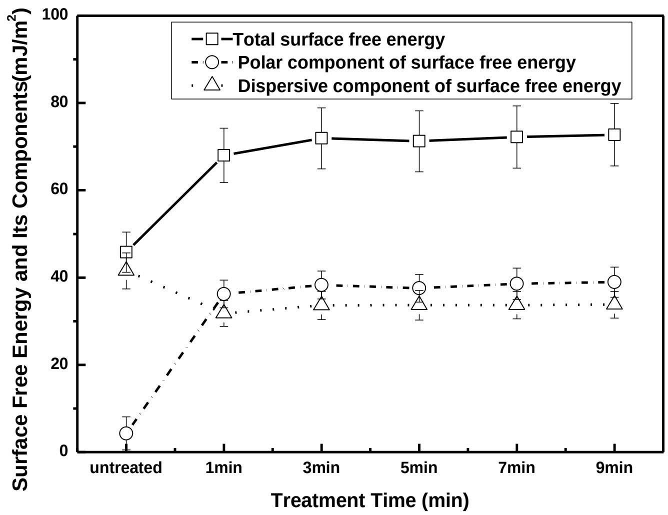

Fig. 4. Surface free energy and its components of untreated and treated veneer

Effect of Cold Oxygen Plasma Treatment on Plywood Shear Strength

Surface wettability of materials influences bonding properties directly. Therefore, the shear strength of plywood was tested to prove the effect of cold oxygen plasma treatment on the surface wettability of poplar veneers. Figure 5 shows the results of the shear strength test for three-layer-plywood panels.

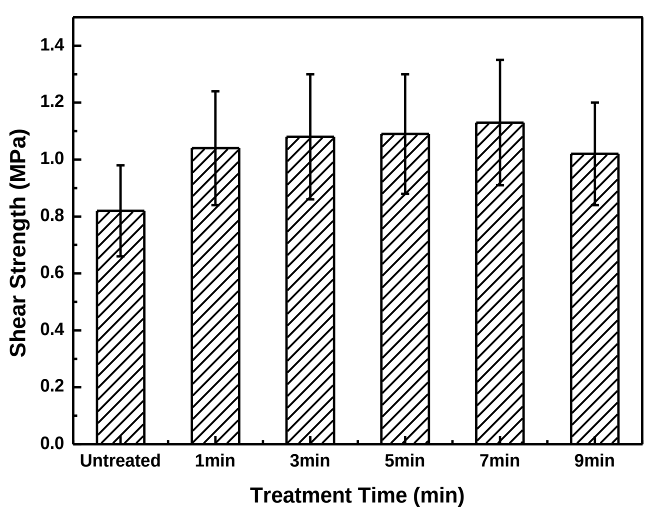

Fig. 5. The shear strength of untreated and treated three-layer-plywood panels

Compared with untreated panels, the samples exhibited a distinct increase in shear strength after cold oxygen plasma treatment. Shear strength kept increasing from 1.04 to 1.13 MPa within the treatment time from 1 min to 7 min. The maximum increase rate of 37.8% was achieved when veneers were treated for 7 min. These findings correlated well with the surface wettability results.

Experimental data were statistically analysed by One-way ANOVA. The probability value was 7.1E-07, which indicated that plasma treatment time was a very significant factor for shear strength.

Effect of Cold Oxygen Plasma Treating Time on Aging Behavior

Aging behavior of the cold oxygen plasma-treated veneers was evaluated by the surface free energy after being stored for some days. The veneers were treated for 7 min with a plasma power of 200 W. The surface free energy and its components of the plasma-treated veneer after aging in the air for different times were calculated from the contact angle of water and diiodomethane, and results are shown in Fig. 6.

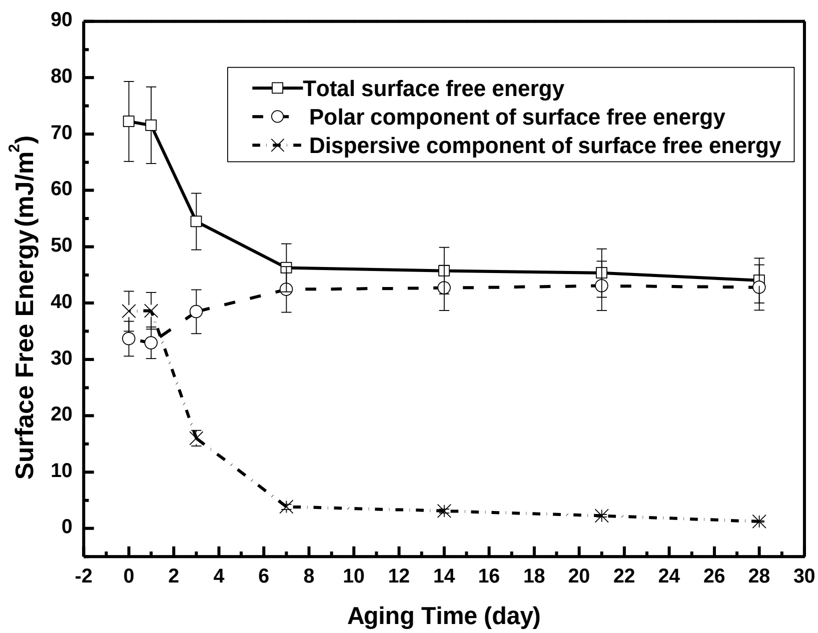

Fig. 6. Surface free energy and its components of veneers after aging in air

It was found that surface free energy and its polar component experienced little change after veneers were stored in air for 1 day. They decreased sharply to 46.25 and 3.84 mJ/m^2, respectively, after storing in air for 7 days. When the aging time increased from 7 days to 28 days, they decreased slowly. The results showed that wettability of veneer surfaces degraded slightly in the first day after the cold oxygen plasma treatment. Therefore, cold oxygen plasma treatment was judged to be feasible for industrial application for modification of veneers.

Table 4. The Contact Angles (°) of Distilled Water and Diiodomethane and Surface Free Energy (mJ/m^2) of Veneer after Aging in Air

Treatment Time	Contact Angle (°)	
	Water	Diiodomethane
Plasma Treated for 7min	15.70 (2.80)	15.95 (3.02)
1 Day after the Treatment	17.15 (2.72)	19.09 (3.95)
3 Days after the Treatment	54.53 (9.84)	20.09 (3.20)
7 Days after the Treatment	77.29 (11.99)	25.50 (3.03)
14 Days after the Treatment	79.46 (11.47)	26.39 (3.71)
21 Days after the Treatment	82.09 (12.70)	27.21 (2.30)
28 Days after the Treatment	86.75 (6.61)	30.75 (3.19)

CONCLUSIONS

The effect of the cold oxygen plasma treatment time on surface wettability, as well as interfacial adhesion of poplar veneers and aging effect of the oxygen plasma modified veneer surfaces were investigated. The main conclusions can be summarized as follows:

(1) Dynamic adhesive wettability of poplar veneer surface was significantly increased after cold oxygen plasma treatment. The treatment time had a remarkable effect on wettability. The decrease of contact angle values and the increase of *K-value* and surface free energy after cold oxygen plasma treatment indicate the improvement of veneer surface wettability.

(2) Shear strength of panels produced by cold oxygen plasma-treated veneers are enhanced. According to wettability of veneer and shear strength of panels, the optimized cold oxygen plasma treatment time should be 7 min.

(3) The aging behavior of the cold oxygen plasma-treated veneer appeared in the first 7 days, and only little additional aging effects were observed with further aging. Therefore, the next process should be conducted within 7 days after cold oxygen plasma treatment.

(4) The advantage of plasma treatment is its clean reaction and short processing time. It represents an efficient, clean, and economic alternative for modification of wood surfaces. We have successfully developed the continuous processing equipment that can be used in the veneer-based composites production line and evaluated the cost of plasma treatment. Since the resin consumption can be reduced 10 to 20% of veneer-based composites produced by the plasma treated veneers, the costs involved for preparing these treated panels could be significantly decreased though the plasma treatment power consumption will increase costs slightly. Obviously, plasma treatment applied to wood modification is advantageous in the economy.

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