

# Surface Properties of Poplar Wood after Heat Treatment, Resin Impregnation, or Both Modifications

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To investigate the surface properties of different modified poplar (*Populus tomentosa* Carr.) wood samples, the color, surface roughness, and wettability of untreated poplar wood (control) and poplar modified *via* heat treatment, resin impregnation, and impregnation combined heat treatment were analyzed and compared in this study. The impregnant used in the test was a modified urea-formaldehyde resin with a low molecular weight and low viscosity. The results showed that the lightness of the samples was sorted in order as follows: the control was lighter than the resin impregnated sample, which was lighter than the impregnation combined heat treatment sample, which was lighter than the heat treatment sample. The surface of the control samples was relatively smooth, while after the impregnation, heat, and impregnation combined heat treatments, the  $R_a$  and  $R_z$  values increased, which indicated increased surface roughness due to the modifications. Among them, the heat-treated samples had the roughest surface, and the surface roughness of the impregnation combined heat treated samples at 160 °C had no major difference from the resin impregnated sample. The wettability of the samples decreased after heat treatment and increased after impregnation combined heat treatment. It was concluded that after the modification treatments, the color of the wood became darker, and the surface roughness and hydrophobicity increased.

*Keywords:* Heat treatment; Impregnation; Impregnation combined heat treatment; Surface properties

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## INTRODUCTION

Poplar is a fast-growing plantation tree species; however, its density, mechanical properties, and dimensional stability are relatively inferior to those of natural wood species. In order to improve the properties of the fast-growing timber, researchers have conducted many studies and used different modification techniques. High-temperature (usually 160 to 220 °C) heat treatment (HT) technology is the most environmentally friendly physical modification method widely used to improve the dimensional stability of wood, but the mechanical strength of wood is reduced by it. Resin impregnation (IMPG) modification not only can enhance the mechanical properties of wood, *e.g.*, its density, bending strength, hardness, *etc.*, but it also improves its flame retardancy, bioerosion resistance, as well as other applicability properties (Huang *et al.* 2013; Qin *et al.* 2015; Sandberg *et al.* 2017; Cai *et al.* 2020). Both impregnation and high-temperature heat treatment can improve certain properties of wood. In addition, the changes in color, surface roughness, wettability, and other surface properties of wood due to impregnation and heat treatment have been widely

accepted by wood industry and market. The color is an important indicator that reflects the visual characteristics of the wood surface. Therefore, it is of great significance to carry out research on the effect of modified wood's color on its application in the furniture, flooring, and decoration industries. However, these single modification methods cannot improve all the previously mentioned properties. The modification lead to certain deficiencies, such that the modified wood often cannot meet the requirements for an application. Therefore, in order to obtain modified wood with better properties, it is desired to seek an impregnation combined heat treatment process to expand the application range of fast-growing wood.

There has been related research on the modification treatment of impregnation combined with heat treatment, but the research is still in its infancy. Baysal *et al.* (2014) studied some mechanical properties such as modulus of rupture (MOR) and some physical properties such as glossiness, color, surface roughness, and water absorption (WA) of adolit-KD 5 impregnated and then heat treated of Scots pine (*Pinus sylvestris* L.) wood specimens, They found that adolit-KD 5 treatment before heat treatment caused decreases in the glossiness and color. Heat treatment induced a strong decrease of luminance (darkening). Moreover, increasing treatment temperature and duration, resulted in decreasing values of MOR, WA, glossiness, and surface roughness. Turkyay *et al.* (2016) investigated mechanical properties such as the modulus of rupture (MOR) and compression strength parallel to grain (CSPG) of impregnated by Wolmanit CX-8 (WCX-8) and Celcure AC-500 (CAC500) and heat-treated Oriental beech (*Fagus orientalis* Lipsky) wood. It was found that both impregnation and heat treatment decreased the MOR and CSPG of Oriental beech wood and higher treatment duration and temperature resulted in lower MOR and CSPG of Oriental beech. Liu *et al.* (2013) investigated the influence of melamine-urea-formaldehyde (MUF) resin impregnation combined with heat treatment on the dimensional stability and mechanical properties of eucalyptus. It was found that the dimensional stability and mechanical properties of the MUF resin combined heat treatment at a temperature of 240 °C were better than the properties of the heat treated only wood. Xu *et al.* (2015) used different heat treatment processes to treat timbers impregnated by PF resin and found that the heat treatment improved the dimensional stability of timbers impregnated with PF resin. In addition, the mechanism of the reduction of the hygroscopicity was studied using infrared spectroscopy. Quan *et al.* (2017) found that the heat treatment temperature had a greater impact on the hygroscopicity and dimensional stability of impregnated poplar wood than the heat treatment time. A study by Lahtela and Kärki (2016) showed that the proper heat treatment of impregnated timber can improve the mechanical properties. It was believed that the modified wood *via* combined treatments had the potential to be used in outdoor structures and furniture.

Studies on wood modification primarily focus on basic physical properties, *e.g.*, the hygroscopicity, dimensional stability, and macroscopic mechanical properties. Limited studies on the surface properties of the modified wood have been completed. In addition, the wood samples used in experiments are mostly small sized (with the size ranging from 50 mm × 50 mm × 10 mm to 400 mm × 120 mm × 25 mm), which is quite different from the actual situation in industrial production (Sivrikaya *et al.* 2019). Therefore, in this experiment, the surface properties of industrial scaled specimens were examined using industrial equipment in order to provide further guidance for the actual manufacturing.

## EXPERIMENTAL

### Materials and Equipment

The wood species used in this study was poplar (*Populus tomentosa* Carr.) with a diameter at breast height greater than 30 cm (purchased from Siyang, China). The sawn timber with a size of 2400 mm × 100 mm × 28 mm (L × W × T) was first kiln-dried until the moisture content reached 12%, and then it was surface planed to a thickness of 22 mm. A total of 150 timber samples were selected with straight grains and no defects, which were numbered as #1, #2, #3... #150 sequentially.

The impregnant used in the test was a modified urea-formaldehyde resin with a low molecular weight (less than 500 g/mol) and low viscosity (1.5 mPa·s at a temperature of 23 °C ± 0.5 °C *via* the rotary viscometer method), and a solid content of 19.56% (the original solid content was approximately 50%), which was independently developed and produced by Fuyang Jinmu Arts and Crafts Co. Ltd. (Anhui, China). The reaction raw materials primarily included urea, ammonia, formaldehyde, melamine, and other ingredients. The primary performance indicators were tested according to GB/T standard 14074 (2017).

To reduce the differences among the test groups, two boards with a length of 330 mm were cut by band saw from all timber samples. One board was unmodified, while the other board was heat-treated at a temperature of 180 °C for 3 h. The remaining timber (with a length of 1740 mm) was first impregnated with the modified urea-formaldehyde resin at a vacuum and then kiln-dried and cut into four equal pieces. One of which was not processed as a control, while the other three were heat-treated at a temperature of 160, 180, and 200 °C for 3 h, respectively. Therefore, there were 6 groups of samples in this experiment, including the control, heat treatment at a temperature of 180 °C, resin impregnation, and impregnation combined with heat treatment at 3 different temperatures (Table 2).

### Methods

#### *Impregnation combined heat treatment process*

The timber samples were impregnated according to LY/T standard 2407 (2015) and the production process of the enterprise. The first step of the impregnation process was to vacuum the dried poplar timber at -0.08 MPa for 30 min in a vacuum impregnated pressure tank. Then, the dried timber was filled with the impregnation liquid in a vacuum state, and the impregnation pressure was increased to 3.0 MPa and maintained for 1 h. After that, the timber samples were taken out and the surface liquid was removed, the pressure was released, and the remaining impregnating liquid was discharged. After 3 d, the impregnated timbers were dried again to a moisture content of 12%; the drying period was 23 d.

#### *Color measurement*

The samples of control, heat treatment at a temperature of 180 °C, resin impregnation, and impregnation combined with heat treatment at 3 different temperatures were respectively processed into samples with a size of 330 mm×100 mm×20 mm. Each group had 10 samples, with a total of 60 samples. The color of the wood surface was measured with a colorimeter according to the CIE wood color characterization method promulgated by the International Commission on Illumination (CIE). The CIELab System was used for color evaluation. A chroma meter (CR-400, Konica Minolta, Tokyo, Japan) was used to measure the  $L^*$ ,  $a^*$ , and  $b^*$  parameters at four specific positions (6 cm between

each two points) on the tangential surface of each sample after regular intervals, and the average values were calculated. The  $L^*$  denotes lightness (+ denotes brighter and - denotes darker),  $a^*$  denotes the red-green index (+ denotes reddish and - denotes greenish), and  $b^*$  denotes the yellow-blue index (+ denotes yellowish and - denotes bluish). The  $\Delta L^*$ ,  $\Delta a^*$ ,  $\Delta b^*$  parameters and the  $\Delta E$  were calculated according to Eqs. 1 through 4,

$$\Delta L^* = L_t^* - L_i^* \quad (1)$$

$$\Delta a^* = a_t^* - a_i^* \quad (2)$$

$$\Delta b^* = b_t^* - b_i^* \quad (3)$$

$$\Delta E = \sqrt{(\Delta L^*)^2 + (\Delta a^*)^2 + (\Delta b^*)^2} \quad (4)$$

where the  $L_i^*$ ,  $a_i^*$  and  $b_i^*$  are the lightness, red-green index, and yellow-blue index of the untreated wood, respectively,  $L_t^*$ ,  $a_t^*$ , and  $b_t^*$  are the lightness, red-green index, and yellow-blue index of the modified wood, and  $\Delta L^*$ ,  $\Delta a^*$ ,  $\Delta b^*$ , and  $\Delta E$  are the differences in the brightness, red-green index, yellow-blue index, and total color change, respectively (Ding *et al.* 2017).

#### *Surface roughness measurement*

The samples of control, heat treatment at a temperature of 180 °C, resin impregnation, and impregnation combined with heat treatment at 3 different temperatures were respectively processed into specimens with a size of 50 mm×50 mm×20 mm. Each group had 10 samples, with a total of 60 samples. In this experiment, one side of the sample surface was manual-planed, and the other side was sanded with 320# sandpaper and then 400# sandpaper. The roughness after the hand-planing was measured *via* a TR110 portable surface roughness meter (Shanghai Double Produced by Asahi Electronics Co., Ltd.) (indicating error less than or equal to ± 15%). The sampling length was  $L = 2.5$  mm and the evaluation length was  $5L = 12.5$  mm. The surface roughness after sanding was measured *via* a JB-4C precision roughness meter (Shanghai Taiming Optical Instrument Co., Ltd.) (indicating error less than or equal to ± 5%).

Referring to GB/T standard 12472 (2003) and GB/T standard 1031 (2009), 6 areas along the transverse direction of the tangential surface, after the manual planing and sanding, were measured (the probe of the measuring head was perpendicular to the grain direction), and the average surface roughness value was then calculated. The sampling length was  $L = 0.8$  mm and the evaluation length was  $5L = 4$  mm. The measurements were randomized relative to their order to make sure that their conclusions were not affected by the gradual wearing out or filling of the sand paper. In general, the average roughness ( $R_a$ ) and average peak-valley height ( $R_z$ ) can comprehensively represent the roughness of a wood surface and as such were selected for the roughness evaluation. Therefore, only the  $R_a$  and  $R_z$  were discussed and analyzed in this study.

#### *Determination of the contact angle and surface free energy*

The samples of control, heat treatment at a temperature of 180 °C, resin impregnation, and impregnation combined with heat treatment at 3 different temperatures were respectively processed into a size of 50 mm×50 mm×20 mm, 3 samples for each group, a total of 18 samples. The contact angle test reagents were distilled water (polar liquid) and diiodomethane (non-polar liquid). The contact angle of each liquid drop on the tangential surface was measured using an optical contact angle instrument (Theta Lite,

Biolin Scientific, Gothenburg, Sweden). A micro-injector was used to take 2  $\mu\text{L}$  and drop it on the planed surface. The contact angle was measured within 2 s after the droplet dropped onto the surface of the sample. Each sample was measured 4 times for each liquid with different measurement positions. The initial contact angle, *i.e.*, the contact angle when the droplet contacted the wood surface, was recorded and the average value was calculated. After the measurement, the surface droplets were removed with absorbent paper. After obtaining the contact angle value of  $\theta$ , the Owens double-liquid method (Owens and Wendt 1969) was used to calculate the surface free energy. According to the polar force ( $\gamma_{sp}$ ), the dispersion force ( $\gamma_{sd}$ ) (non-polar force), and the surface free energy ( $\gamma_s$ ) (surface tension) of distilled water and diiodomethane (Table 1) and the contact angle value of  $\theta$ , the polar force and the dispersion force ( $\gamma_{Ld}$ ) can be obtained from Eq. 5 and 6, respectively,

$$\gamma_{L_1}(1 + \cos\theta_1) = 2(\gamma_{s^d}\gamma_{L_1^d})^{1/2} + 2(\gamma_{s^p}\gamma_{L_1^p})^{1/2} \quad (5)$$

$$\gamma_{L_2}(1 + \cos\theta_2) = 2(\gamma_{s^d}\gamma_{L_2^d})^{1/2} + 2(\gamma_{s^p}\gamma_{L_2^p})^{1/2} \quad (6)$$

and then the surface free energy ( $\gamma_L$ ) can be obtained according to Eq. 7,

$$\gamma_{s^d} + \gamma_{s^p} = \gamma_s \quad (7)$$

where  $\gamma_L$ ,  $\gamma_{Ld}$ , and  $\gamma_{Lp}$  are the surface tension, dispersion force, and polar force of the test liquid (distilled water and diiodomethane), respectively,  $\gamma_s$ ,  $\gamma_{sd}$ ,  $\gamma_{sp}$  and are the surface free energy, dispersion force, and polar force of the test wood samples, respectively, and  $\theta$  is the contact angle.

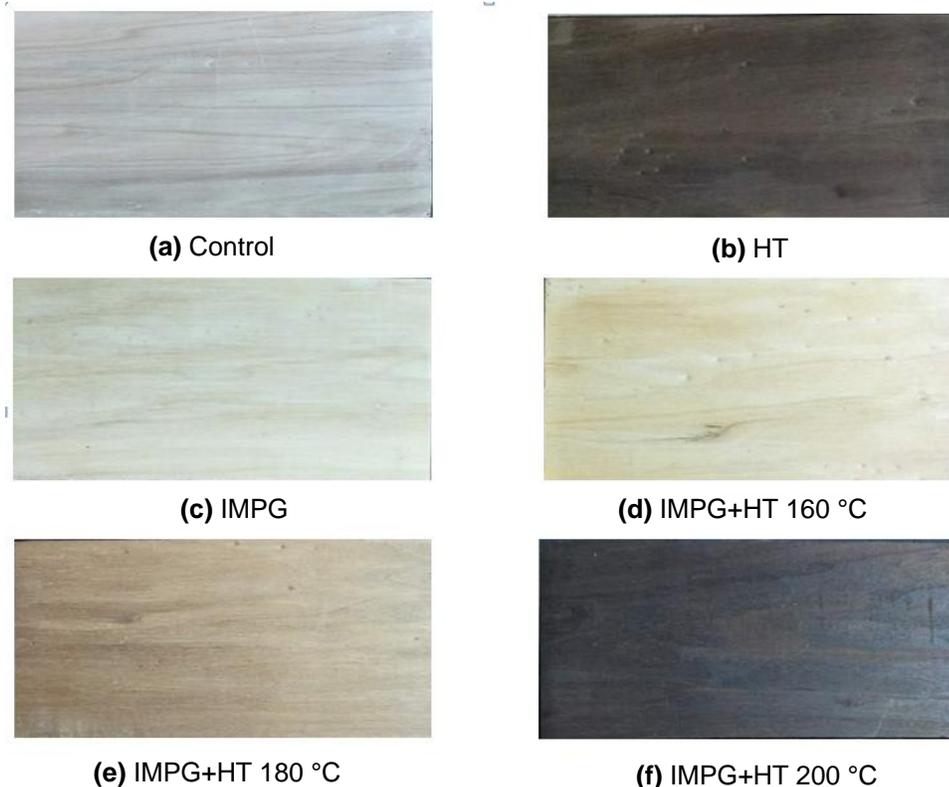
**Table 1.** Surface Tension, Polar Force, and Non- Polar Force of the Test Liquid

Test Liquid	Type	Dispersion Force $\gamma_{Ld}$ (mJ/m <sup>2</sup> )	Polar Force $\gamma_{Lp}$ (mJ/m <sup>2</sup> )	Surface Tension $\gamma_L$ (mJ/m <sup>2</sup> )
Distilled water	Polar	21.80	51.00	72.80
Diiodomethane	Non-polar	48.50	2.30	50.80

## RESULTS AND DISCUSSION

### Color of Modified Materials

Figure 1 shows the colors of the control and the modified samples. The HT at a temperature of 180 °C for 3 h drastically increased the darkness of specimens, while the IMPG changed the surface color to light yellow. The color of the IMPG+HT samples changed considerably with different heat treatment temperatures. As the heat treatment temperature increased, the color gradually changed from light yellow to brown and dark brown. By comparing Fig. 1b and Fig. 1e, it was found that, with the same heat treatment temperature and duration, *i.e.*, 180 °C and 3 h, the color of the HT sample was darker than the color of the IMPG+HT at a temperature of 180 °C, which indicated that the resin impregnation had a certain inhibitory effect on the color change of the wood.



**Fig. 1.** Surface colors of the different samples

Table 2 shows the results of the color index and their changes after different modifications. As can be seen from Table 2, the  $L^*$  of the HT samples was 40.97, which was 38.18% lower than the  $L^*$  of the control, which indicated the lightness of poplar wood decreased after the heat treatment (Militz 2002). The lightness of the IMPG samples slightly decreased (1.37%), which indicated that the resin impregnation had a minor effect on the lightness value of poplar. After the IMPG+HT, the  $L^*$  value ranged from 33.76 to 66.23, and the lightness values were 14.63% to 56.49% lower than the lightness of the control. This was attributed to the fact that the water-soluble UF resin itself was yellowish brown. After being immersed in the wood and cured by heating, the color became darker. In addition, it contained hydroxyl (-OH), which was easy to change after the HT treatment, forming benzoquinone or other structures and causing the color of the samples to deepen. As the heat treatment temperature increased, the lightness value of the IMPG+HT samples became lower, and the color became darker. This was primarily due to the fact that the higher the heat treatment temperature, the more obvious the degradation of the hemicellulose and impregnated resin, and these dark degradation products led to the decrease in the wood lightness. With the same heat treatment process (180 °C and 3 h), the lightness of the HT samples more considerably decreased compared to the IMPG+HT samples. This may be due to the crosslinking of the resin and hemicelluloses in the wood, which inhibited the degradation to some extent, or the alkaline environment caused by the urea-formaldehyde resin inhibited the degradation of the hemicelluloses, thereby decreasing the effect of the heat treatment on the lightness reduction (Zhang *et al.* 2014).

**Table 2.** Changes in the Color Indexes of the Control and Modified Samples

Materials	$L^*$	$\Delta L^*$	$a^*$	$\Delta a^*$	$b^*$	$\Delta b^*$
Control	77.59	-	4.71	-	20.99	-
HT (180 °C)	40.97(2.01)	-36.62	8.10(0.50)	3.39	17.19(1.11)	-3.80
IMPG	76.53(2.77)	-1.06	4.08(0.94)	-0.63	28.48(1.61)	7.49
IMPG+HT 160 °C	66.23(3.26)	-11.36	8.92(0.92)	4.21	36.42(1.91)	15.43
IMPG+HT 180 °C	53.17(3.41)	-24.42	10.63(0.61)	5.92	28.64(2.18)	7.65
IMPG+HT 200 °C	33.76(2.24)	-43.83	7.20(0.95)	2.49	13.88(2.22)	-7.11

Note: the data in the parenthesis refers to the standard deviations

The  $a^*$  of the HT samples increased by 71.97% compared to the  $a^*$  of the control, which indicated that the HT increased the red-green index of the poplar wood. The  $a^*$  of the IMPG samples decreased by 13.38%, which indicated that the IMPG treatment reduced the red-green index of the poplar wood. The  $a^*$  of the IMPG+HT samples ranged from 7.20 to 10.63, which increased by 52.87% to 125.69% when compared to the control. As the HT temperature increased, the  $a^*$  first increased and then decreased. However, the  $a^*$  value of the IMPG+HT was always higher than the  $a^*$  value of the control and IMPG samples.

With the same heat treatment process (180 °C and 3 h), the  $a^*$  of the IMPG+HT increased more than the  $a^*$  of the HT samples, and its color was more reddish. This may be due to the resin and lignin crosslinking at this temperature, which changed the number of chromophoric groups, *e.g.*, carbonyl groups in lignin (Zor *et al.* 2019). When the temperature was higher than 180 °C, the product generated by the crosslinking of the resin and lignin was unstable and further degraded into small molecules at high temperatures, leading to an increase then decrease of the  $a^*$  as the temperature increased (Raisanen *et al.* 2003).

The  $b^*$  values of the HT samples were 18.10% lower than the  $b^*$  of the control, which indicated a reduction of the yellow-blue index of poplar wood after the HT. The  $b^*$  of the IMPG samples increased by 65.68%, which indicated an increase in the yellow-blue index of poplar after the IMPG treatment. The  $b^*$  of the IMPG+HT samples ranged from 13.88 to 36.42, which increased by -33.87% to 73.51% when compared to the control. As the heat treatment temperature increased, the  $b^*$  value gradually decreased, even eventually reaching a negative  $\Delta b^*$  value. This was because there was no obvious resin degradation, and its color was still yellowish at lower heat treatment temperatures. However, as the temperature increased, the resin degradation became more obvious, which resulted in a rapid decrease in the  $\Delta b^*$ . With the same heat treatment process (180 °C and 3 h), the changes in the  $\Delta b^*$  of the HT samples and the IMPG+HT were opposite, which was primarily affected by the addition of resin.

Figure 2 shows the color differences of the samples from different modification groups. The total colour difference of each modified group are given in descending order as follows: IMPG+HT 200 °C was greater than HT, which was greater than IMPG+HT 180 °C, which was greater than IMPG+HT 160 °C, which was greater than IMPG. The total color difference between the IMPG and the control was small. However, the HT had major impact on the absolute color difference. The  $\Delta E$  of the IMPG+HT samples ranged from

19.62 to 44.47. The higher the heat treatment temperature, the greater the  $\Delta E$ . When the temperature was higher than 180 °C, there was a drastic increase in the  $\Delta E$ . With the same heat treatment process (180 °C and 3 h), the total color difference between the combined modified samples and the controls was smaller than the total color difference of the heat treatment, which indicated that the impregnation pretreatment can effectively reduce the effect of the heat treatment on the color.

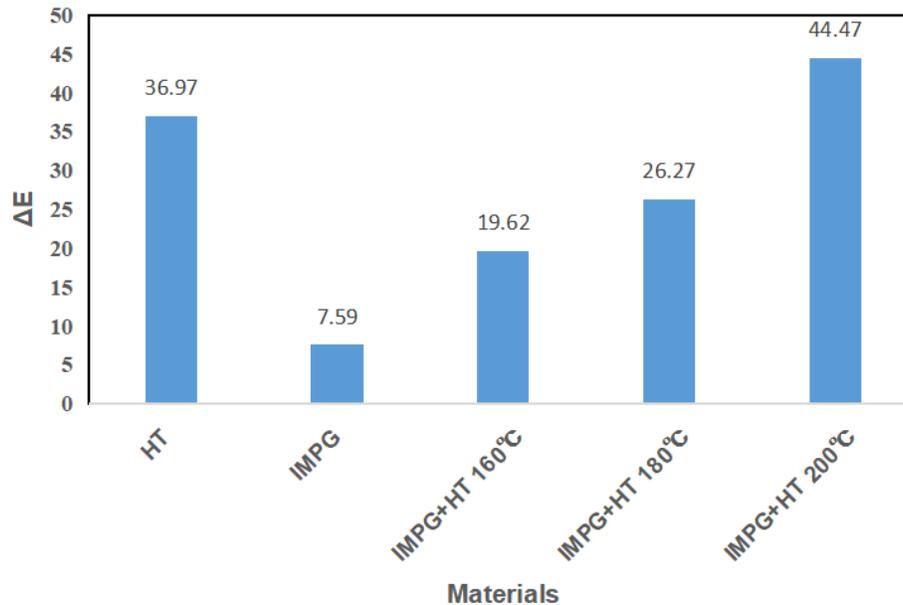


Fig. 2. Color difference values of the different samples

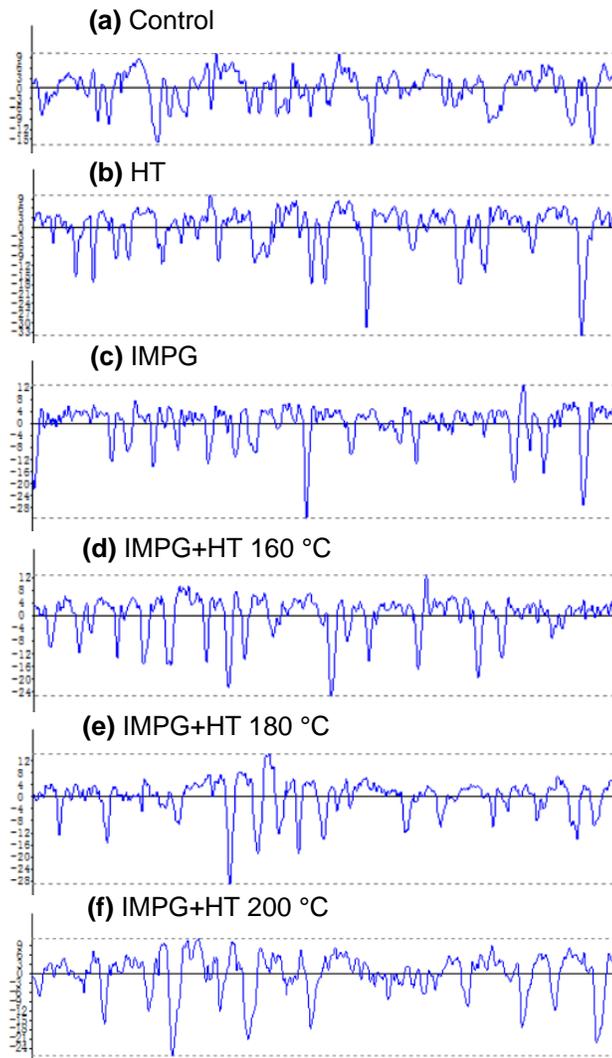
### Surface Roughness of the Modified Woods

Figure 3 shows the surface profile of each test group after sanding. Figures 4 and 5 show the test results of the  $R_a$  and  $R_z$  of each group, respectively.

Figure 3 shows that the surface of the control was relatively flat. After the resin impregnation process, heat treatment, or combined modification, the  $R_a$  and  $R_z$  values became larger, and the wood surface became rougher than the control. Heat treatment made the wood more brittle and easier to crack during cutting; the impregnated resin was plasticized, and the heat generated by sanding caused the plasticized resin to fluff during the sanding process. According to Figs. 4 and 5, the  $R_a$  and  $R_z$  values of the planed wood surface were larger than the  $R_a$  and  $R_z$  values of the sanded wood. This was because the wood surface processing method is an important factor in terms of its surface roughness. The higher the processing accuracy, the lower the surface roughness (Wang *et al.* 2005). However, for both the sanded and planed surfaces, the  $R_a$  had similar trends after different modification treatments.

Both the  $R_a$  and  $R_z$  values of poplar wood increased after HT. After the planing process, the  $R_a$  and  $R_z$  increased from 6.15 and 40.39  $\mu\text{m}$  to 7.16 and 46.32  $\mu\text{m}$ , respectively. After the sanding process,  $R_a$  and  $R_z$  increased from 3.63 and 25.06  $\mu\text{m}$  to 4.57 and 29.30  $\mu\text{m}$ , respectively. These results showed that the surface of the poplar wood became more uneven after HT at a temperature of 180 °C for 3 h, which agreed with the results reported by Bakar *et al.* (2013). The reason for the increased surface roughness in poplar wood after HT might be due to the breakages of fibers of the heat-treated wood

during the planing and sanding processes (Bourmaud and Baley 2009; Butylina *et al.* 2011). Therefore, the surface of the wood fibers split, and the surface roughness increased.



**Fig. 3.** Surface roughness profile of the different test woods after sanding

The results also showed that both the  $R_a$  and  $R_z$  values increased after IMPG. After the planing process, the  $R_a$  and  $R_z$  increased to 7.61 and 52.12  $\mu\text{m}$ , respectively. After the sanding process, the  $R_a$  and  $R_z$  increased to 4.21 and 28.97  $\mu\text{m}$ , respectively. The result showed that the resin impregnation also improved the surface roughness of poplar wood. The primary reason for this result might be that the uneven distribution of the resin in the microstructure of the wood increased the roughness of wood.

The  $R_a$  value of the IMPG+HT sample after planing ranged from 5.92 to 7.32  $\mu\text{m}$ , while the  $R_z$  value ranged from 37.96 to 47.98  $\mu\text{m}$ . After the sanding process, the  $R_a$  and  $R_z$  values decreased from 3.76 to 4.46  $\mu\text{m}$  and 25.95 to 28.16  $\mu\text{m}$ , respectively. As shown in Figs. 4 and 5, the  $R_a$  and  $R_z$  of the IMPG+HT samples first decreased and then increased as the heat treatment temperature increased. The surface roughness of the IMPG+HT samples at 160  $^{\circ}\text{C}$  had no considerable difference from the IMPG samples, and the surface

of the IMPG+HT sample at 180 °C was the smoothest. This might be because the volume of the impregnated resin shrunk at a temperature of 180 °C, which led to the resin filling in the vessels, and the wood fibers tending to be flat, thus reducing the effect of the resin on the surface roughness (Eichhorn *et al.* 2001). However, when the heat treatment temperature reached 200 °C, the resin degradation products and the wood extracts migrated to the wood surface, which resulted in an increase in the surface roughness of the IMPG+HT wood at a temperature of 200 °C (Willner and Brunner 2005; Poletto 2016).

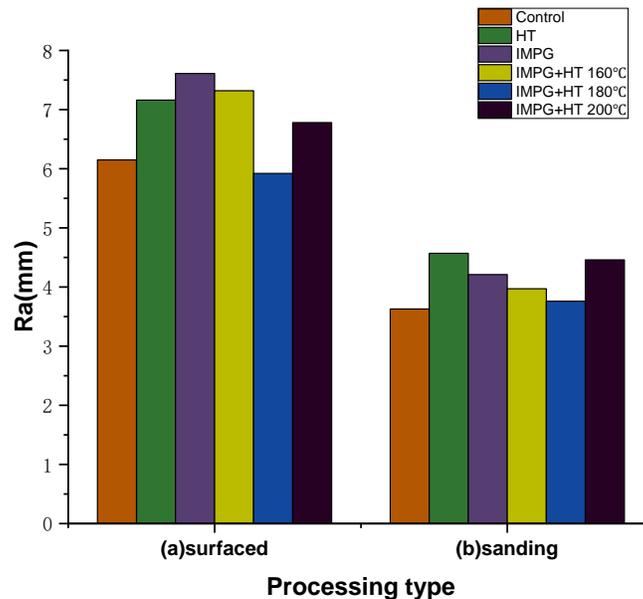


Fig. 4.  $R_a$  of the different test woods

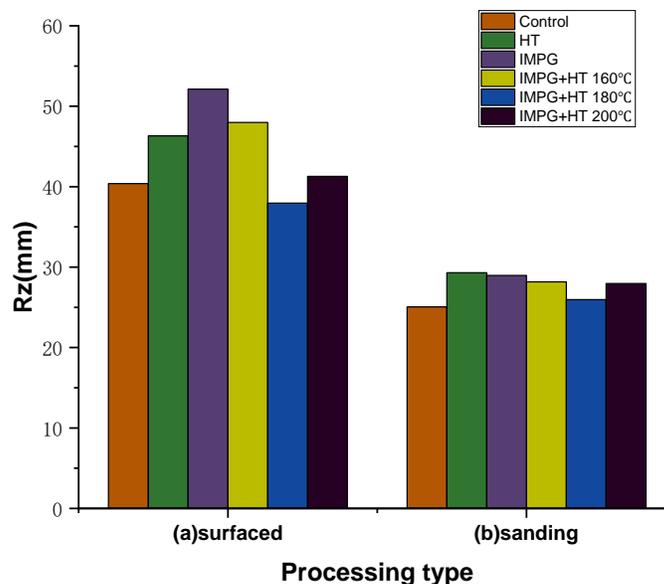
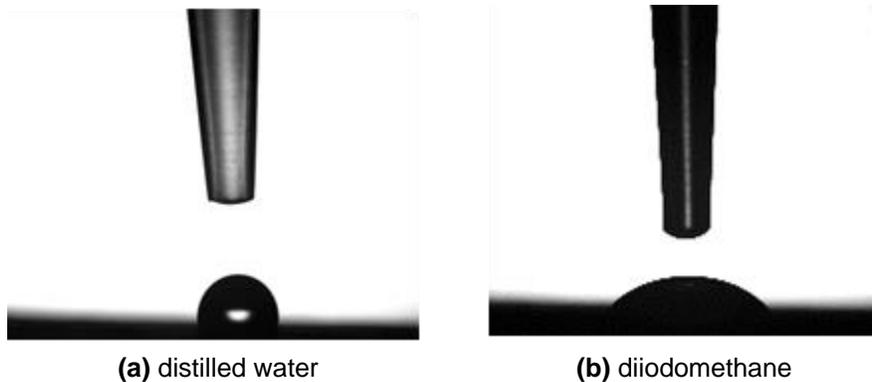


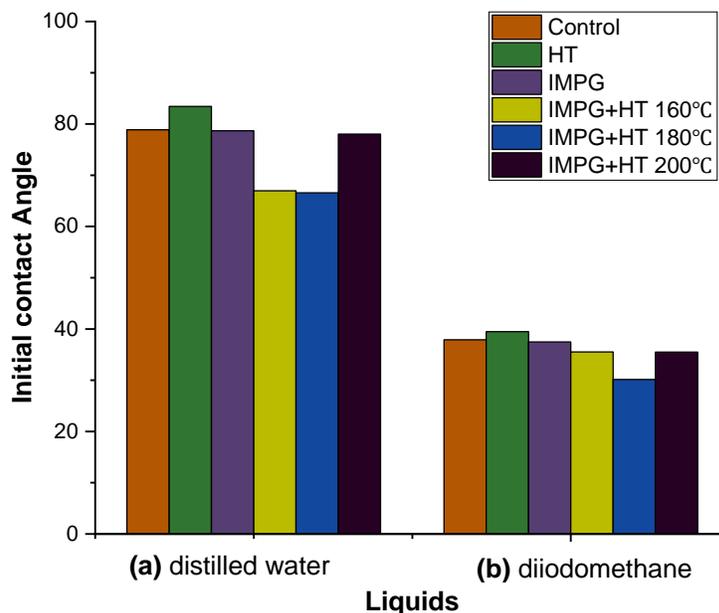
Fig. 5.  $R_z$  of the different test woods

### Surface Wettability of the Modified Woods

Figure 6 shows the initial contact angles of the poplar wood surface with distilled water and diiodomethane. The contact angle of diiodomethane on the wood surface was smaller than the contact angle of distilled water. The diiodomethane quickly wetted the wood at the moment of contact with the wood surface, but the outline of the droplet on the wood surface was still clear. Then, the droplet was gradually absorbed by the wood. This is because diiodomethane is a non-polar liquid, while distilled water has strong polarity and surface tension; therefore, the contact angle between the wood surface and distilled water was larger (Fabiya and McDonald 2010).



**Fig. 6.** Initial contact angle of poplar wood with distilled water and diiodomethane



**Fig. 7.** Initial contact angles of the different test wood samples with distilled water and diiodomethane

Figure 7 shows the results of the contact angles of the different test groups with distilled water and diiodomethane. The contact angle of distilled water on the surface of the samples in descending order was as follows: HT was greater than Control, which was

greater than IMPG, which was greater than IMPG+HT 200 °C, which was greater than IMPG+HT 160 °C, which was greater than IMPG+HT 180 °C. Among all groups, the HT sample had the largest contact angle (83.43°), which indicated that the hydrophobicity was increased after HT treatment. This was attributed to a decrease in the number of hydrophilic groups on the wood surface, caused by the degradation of hemicellulose during the HT treatment. The contact angle of the poplar wood with distilled water slightly decreased from 78.87° to 78.69° after impregnation, which indicated that the IMPG treatment had little effect on the hydrophilicity. The contact angle between the IMPG+HT wood and distilled water, which ranged from 66.57° to 78.02°, first decreased and then increased as the heat treatment temperature increased. This might be due to the fact that the HT did not drastically degrade the wood components and had a limited effect on increasing the hydrophobicity at the lower temperature. However, the resin preferentially degraded and produced hydrophilic degradation products during the low-temperature HT (Mankowski and Morrell 2000; Verhey *et al.* 2001; Tripathi *et al.* 2014). Such a phenomenon predominantly influenced the contact angle and led to the decreased contact angle and increased wettability of the IMPG+HT samples when the wood was heat-treated below 200 °C. When the temperature reached 200 °C, the HT had a noticeable degradation effect on the wood components, and the hydrophilic groups on the wood surface were drastically reduced. Therefore, the influence of the HT on the contact angle was dominant, which made the hydrophobicity of the IMPG+HT samples increase.

The contact angle of diiodomethane on the surface of the samples in descending order was as follows: HT was greater than Control, which was greater than IMPG, which was greater than IMPG+HT 200 °C, which was greater than IMPG+HT 160 °C, which was greater than IMPG+HT 180 °C, which was consistent with the results of distilled water. The contact angle of diiodomethane on the surface of the HT sample was the largest (39.47°), which indicated that the oleophobicity was increased after the HT treatment. After the heat treatment, the number of lipophilic groups decreased. 180 °C heat treatment had the greatest tendency to bring alkyl-type monomers to the wood surface such as fatty acids. Such materials would have a relatively low Hamaker constant, thus tending to give a relatively low dispersion-type interaction energy with the nonpolar probe liquid. The contact angle of the IMPG sample was slightly reduced. The reduction from 37.90° to 37.46° indicated that the resin had little effect on the lipophilicity. The contact angle of the IMPG+HT wood sample with diiodomethane ranged from 30.14° to 35.53°, which also first decreased and then increased as the HT temperature increased, due to the same reason as the change in contact angle of distilled water.

Table 3 shows the surface free energy and its component results of the different modified samples.

**Table 3.** Surface Free Energy and Component Value of Different Specimens

Materials	Dispersion Force $\gamma_{sd}$ (mJ/m <sup>2</sup> )	Polar Force $\gamma_{sp}$ (mJ/m <sup>2</sup> )	Surface Tension $\gamma_s$ (mJ/m <sup>2</sup> )
Control	36.78	4.48	41.26
HT	37.13	2.88	40.01
IMPG	36.97	4.50	41.47
IMPG+HT 160 °C	35.54	9.36	44.90
IMPG+HT 180 °C	37.47	9.47	47.21
IMPG+HT 200 °C	37.83	4.55	42.38

As shown in Table 3, the surface free energy of the different modified samples in descending order was as follows: IMPG+HT 180 °C was greater than IMPG+HT 160 °C, which was greater than IMPG+HT 200 °C, which was greater than IMPG, which was greater than Control, which was greater than HT, which was the result of the contact angle in contrast. After the heat treatment, the dispersion force (non-polar component) changed from 36.78 mJ/m<sup>2</sup> to 37.13 mJ/m<sup>2</sup>, and the polar force (polar component) changed from 4.48 mJ/m<sup>2</sup> to 2.88 mJ/m<sup>2</sup>. This was due to the increase of the number of non-polar functional groups on the wood surface and the decrease of the number of polar functional groups (hydroxyl) through the heat treatment. The surface free energy of the IMPG and its components were slightly increased compared to the control, which indicated that the impregnation had no obvious influence on the wettability of the wood. After the IMPG was heat-treated at different temperatures, its surface free energy changed according to the temperature. Compared with the IMPG samples, the dispersion force of the IMPG-160 °C sample was reduced by 3.87%, and the polar force was obviously increased (the increase rate was as high as 108.00%), which indicated that the heat treatment temperature reduced the number of non-polar functional groups in the IMPG samples, and the number of polar functional groups considerably increased. As the heat treatment temperature increased, the dispersion force of the impregnation combined heat treatment samples gradually increased, while the polar force first increased and then decreased as the heat treatment temperature increased. However, the surface free energy of the impregnation combined heat treatment samples at all three temperatures were higher than the surface free energy of the IMPG samples, which indicated that the heat treatment improved the wettability of the IMPG samples. Among them, the sample after the IMPG+HT 180 °C treatment had the largest surface free energy and the good wettability.

## CONCLUSIONS

1. In this study, the color, surface roughness, and surface wettability of the untreated control wood, and the wood modified *via* heat treatment, resin impregnation, and impregnation combined heat treatment (at 160 °C, 180 °C, and 200 °C) were investigated. The results showed that the lightness ( $L^*$ ) values of each group were ranked as follows: Control was greater than resin impregnation plus heat treatment (IMPG+HT) 200 °C, which was greater than HT, which was greater than IMPG+HT 180 °C, which was greater than IMPG+HT 160 °C, which was greater than IMPG. The wood surface turned a reddish color after HT and IMPG+HT, while it became a greenish color after the IMPG treatment. For the IMPG+HT wood, the color became darker and more blueish as the heat treatment temperature increased. Simultaneously, the reddish color first increased and then decreased, and the total color change always increased as the temperature increased. The IMPG treatment had a small effect on the total color change, while the HT had a major impact on the total color change.
2. Both the HT and IMPG treatments increased the surface roughness of the wood. The  $R_a$  and  $R_z$  of the IMPG+HT wood samples first decreased and then increased as the heat treatment temperature increased. The surface roughness of the IMPG+HT wood samples at a temperature of 160 °C were not considerably different from the surface roughness of the IMPG wood samples. The surface of the IMPG+HT wood at a temperature of 180 °C was the smoothest among all the groups, while the surface

roughness of the IMPG+HT samples at a temperature of 200 °C began to increase. The results showed that both the  $R_a$  and  $R_z$  increased after different modifications.

3. The initial contact angles of all test groups with distilled water were greater than the initial contact angles with diiodomethane. The contact angle of distilled water on the surface of the samples in descending order are as follows: HT was greater than Control, which was greater than IMPG, which was greater than IMPG+HT 200 °C, which was greater than IMPG+HT 160 °C, which was greater than IMPG+HT 180 °C. After the heat treatment, the polar component and surface free energy were reduced, and the wettability was reduced. After impregnation, the surface free energy and its components were slightly increased. Compared with the other modified samples, the surface free energy of the IMPG+HT samples increased, and the wettability was improved. Among them, the polar components of the samples after the IMPG+HT 160 °C and IMPG+HT 180 °C treatments were obviously higher than the polar components of the IMPG samples, but the polar component of IMPG+HT 200 °C (4.55 mJ/m<sup>2</sup>) was reduced to close to the value of the polar component of IMPG (4.48 mJ/m<sup>2</sup>).

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