Surface Characteristics of Poplar Wood with High-Temperature Heat Treatment: Wettability and Surface Brittleness

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The many uses of wood are greatly affected by its surface properties, which are significantly altered by heat treatment. Investigated here are the wettability and surface brittleness when treating poplar wood with heat at 160, 180, 200, and 220 °C for 2 h. Contact angles were measured by the sessile drop method, and surface free energy was calculated. Surface brittleness was expressed by hardness (HD value), roughness (R_a , R_q , R_y , and R_z values), and abrasive resistance (K value). Next, non-destructive Fourier transform near-infrared spectroscopic (FT-NIR) and X-ray photoelectron spectroscopic (XPS) measurements were employed to analyze the surface chemical changes. Scanning electron microscopy (SEM) revealed the post-heating microscopic structure. The results demonstrated that heat treatment reduces the surface wettability while increasing the surface brittleness, which becomes more apparent with increased temperature. Significant differences were determined (p < 0.05) between the surface parameters at four different temperatures. The degradation of cell wall components and the deterioration of microstructures was further expounded by FT-NIR, XPS, and SEM analyses. Furthermore, the abrasive resistance and hardness values decreased in line with the rate of weight loss (WL, %) and temperature. This indicates a strong correlation between the surface characteristics and the WL or temperature. The intensity of heat treatment appears to be predictable and easy to regulate.

Keywords: Thermal treatment; Poplar wood; Hydrophobicity; Surface brittleness; Surface abrasion resistance; Near infrared spectroscopy (NIR); X-ray photoelectron spectroscopy (XPS)

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INTRODUCTION

Because of the declining production and increasing protection of natural forest wood, poplar is one of the most important plantation trees in the world, providing the industrial requirements of timber. There are more than 50 kinds of poplar wood in China, growing mostly in northern China. Jia *et al.* (2013) studied the carbon storage and density of poplars in China, and according to data from the seventh national forest inventory, found that poplar plantations accounted for 15.9% of the carbon storage relative to the whole of the carbon stored in plantation biomass. Moreover, carbon storage of young and middle-aged poplar plantations accounted for 65.9% of the total carbon storage. However, plantation poplar timbers cannot be used as structural materials because of their low values of density, mechanical strength, dimensional stability, *etc.* (Xu *et al.* 2015). Additionally,

poplar timbers have a light color and no obvious surface texture. Generally, they are used as raw material for artificial boards and papermaking (Ye and Wang 2003; Jia *et al.* 2009).

Many studies have used different modification processes, including acetylation, fire retardants, preservatives, impregnation with resins, as well as heat treatment, to improve wood performance (Esteves and Pereira 2009; Hamdan and Islam 2012; Ajdinaj *et al.* 2013; Adebawo *et al.* 2015; Kumar *et al.* 2015; Zhang *et al.* 2015). Unlike other modifications, heat treatment is one of the oldest and most environmentally friendly, and it has recently received renewed interest because of the limitations on the use of natural lumber and restrictive regulations on the use of toxic chemicals (Hillis and Rozsa 1985; Boonstra *et al.* 2006a,b). Moreover, heat treatment could be an alternative modification to make it possible for poplar to be applied as furniture and interior decoration material because of the improvement of dimensional stability and aesthetic characteristics during the treatment.

In recent years, researchers in Finland, France, and the Netherlands have carried out systematic studies on high-temperature heat treatment of wood, forming a relatively mature technology (Esteves and Pereira 2009; Salca and Hiziroglu 2014). Heat treatment is usually applied at temperature levels and treating times ranging from 160 to 260 °C and from 15 min to 24 h. The treatments differ in terms of process conditions, such as the presence of a shielding gas (nitrogen or steam), humid or dry processes, and the use of oils (Candelier *et al.* 2013; Salca and Hiziroglu 2014). The intensity of heat treatment can be appreciated by means of mass loss during the treatment. Color change and mass loss are best described by a logarithmic regression equation, while dynamic wetting behavior is linearly related to the mass loss (Hakkou *et al.* 2006; Olarescu and Campean 2014; Wang *et al.* 2015).

Heat-treated wood possesses novel properties, such as improved decay resistance, higher dimensional stability, aesthetic coloration, and photo-degradation (Dubey *et al.* 2012; Persze and Tolvaj 2012; Bal and Bektaş 2013; Bekhta *et al.* 2014; Aytin and Korkut 2015). However, adverse influences on mechanical characteristics, like impact strength, compression strength, and shear strength, restrict the heat-treated wood from being used in structural applications (Kasemsiri *et al.* 2012; Bakar *et al.* 2013). In addition, some mechanical properties of juvenile wood are greater than those that occur in mature wood at the same treating conditions, as wood is a highly heterogeneous and anisotropic material (Bal and Bektaş 2013).

In actuality, the aesthetics, as well as performance of wooden products are closely associated with surface properties such as color, texture, hardness, roughness, and wettability. The chemical or physical characteristics of wood surfaces affect the gluing and coating processes, while the decorative surface of wooden wares attracts consumers to purchase them. Existing studies have shown that chemical, physical, and structural properties of a heat-treated wood surface also change during heat treatment (Inari et al. 2006; Popescu et al. 2011; Popescu et al. 2013; Li et al. 2015). Wood surfaces turn out to be warmer and more attractive because of the formation of degradation products from hemicelluloses, changes in extractives, and the formation of oxidation products such as quinones (Dubey et al. 2012; Wastiels et al. 2012). On the other hand, heat treatment reduces the hygroscopicity and increases the hydrophobicity of wood surfaces (Gérardin et al. 2007; Mitsui et al. 2008; Kamperidou et al. 2013). Pétrissans et al. (2003) found that the increase in cellulose crystallinity of heat-treated wood could explain the higher contact angle on the wood surface. Another study using CP/MAS (cross-angle spinning) ¹³C NMR spectra, and Fourier Transform Infrared Spectrometric (FTIR) analyses found that wettability changes may be caused by changes to the conformational arrangement of wood biopolymers or plasticization of lignin (Hakkou et al. 2005). Additionally, it was shown that the surface roughness of heat-treated wood decreases after the treatment (Ozcan et al. 2012; Bakar et al. 2013). Rekola et al. (2013) found that the mean arithmetic deviation of profile values (R_a) and associated standard deviations of the European downy birch (*Betula* pubescens Ehrh) wood surface were reduced from 3.53 (1.36) to 2.02 (0.58) µm after heating at 200 °C for 2 h. In addition to the surface properties mentioned above, abrasion resistance is another important parameter that contributes to the chemical constitution and microstructural properties of the wood surface. Another study investigated the friction mechanisms in wood cutting and friction between wood and various materials. It was found that the integrated effects of meshing, the adhesion effect, and the effect of the furrows friction could be used to explain the abrasion loss of the wood surface (Klamecki 1976; Murase 1984). Hu and Liu (1992) studied the abrasion resistance of different species of wood surfaces and found that the density, hardness, and microstructural characteristics of the wood surface were the results of abrasion and structure features. As many researchers have shown, not only does the chemical composition of heat-treated wood decompose, but the cell wall structure also changes during treatment. The abrasion resistance can constitute an alternative synthetic evaluation of heat-treated wood surfaces, whereas it was previously rarely used to assess the influence of heat treatment on wood surfaces.

Near-infrared spectroscopy and X-ray photoelectron spectroscopy have been widely used for the investigation of the surface chemical composition, providing information regarding the chemical composition and molecular structure of wood (Schwanninger *et al.* 2011; Muñiz *et al.* 2013). Mitsui *et al.* (2008) studied the variation of the molecular structure of heat-treated wood using NIR spectroscopy and found that the hydroxyl groups in the cellulose became degraded in the following order: amorphous, semi-crystalline, and crystalline regions. XPS results showed that the oxygen/carbon ratio (O/C) of beech decreased from 0.55 before treatment to 0.44 after treatment at 240 °C (Inari *et al.* 2006).

Many studies have focused on the mechanical properties, as well as surface changes, of heat-treated wood surfaces, especially those of natural forest timbers. Poplar wood was thermal-treated to improve its dimensional stability and resistance against fungi and insects, and the degradation of some hydrophilic components reduced significantly the ability of bound water to migrate through the wood (Giebeler 1983; Rousset *et al.* 2004). Nevertheless, the effect of heat treatment on the surface properties of plantation poplar timbers, especially wettability and surface brittleness, has rarely been studied. Wood surface changes can affect the distribution of adhesives on the wood surface, the penetration of adhesive into the porous wood structure, the curing of adhesives, and finally, the bonding strength (Boonstra *et al.* 2006a,b; Inari *et al.* 2006; Sernek *et al.* 2007; Dilik and Hiziroglu 2012). Exact knowledge of heat-treated poplar wood's surface characteristics, including wettability, surface hardness, roughness, and abrasive resistance, is essential to optimize coating, painting, and gluing processes.

In this study, the effects of heat treatment on the wood surface of poplar were investigated. The surface embrittlement was represented by surface hardness, abrasion resistance, and roughness after the sanding process. Furthermore, the correlations between the surface properties (HD or K values) and the intensity of heat treatment (WL or temperature) were also studied. Chemical and microstructural changes to the heat-treated poplar wood surface were analyzed by FT-NIR, XPS, and SEM methods. The aim of this study is to offer the timber products industry some useful information and explanations for the surface changes to heat-treated poplar wood.

EXPERIMENTAL

Thermal Treatment

Poplar (*Populus beijingensis* W. Y. Hsu) with an initial moisture content of 20% was purchased from a local timber market in Beijing, China, and wood blocks (1500 mm \times 400 mm \times 25mm) were cut with longitudinal faces parallel to the grain. Next, the blocks were polished with 240-grit sandpaper to ensure uniform surface roughness. Prior to treatment, samples were dried to 10% equilibrium moisture, divided into five groups, and numbered in series. The first four groups of samples were then treated under vapor protection in a laboratory oven at 160, 180, 200, and 220 °C in sequence for 2 h. From ambient to final temperature, oven temperature increased by 20 °C/min, after which the specimens were dried in a climate chamber to average equilibrium moisture of 10% for further tests, wherein the test specimens of surface characteristics, chemical component and morphological analysis were cut from the same wood surface. Samples before and after heat treatment was oven dried for 12 h at 60 °C and 12 h at 103 ± 2 °C to a constant weight. The weight loss during heat treatment was calculated according to Eq. 1,

$$WL = \frac{w_{bf} - w_{af}}{w_{bf}} \times 100\%$$
(1)

where w_{bf} is the oven dry weight before treatment (g) and w_{af} is the oven dry weight after treatment (g).

Measurement of Surface Characteristics

Using the sessile drop method, time-dependent contact angles were measured on the earlywood area of tangential sections on a Dataphysics OCA20 contact angle analyser (DataPhysics Instruments GmbH, Filderstadt, Germany). Data were collected randomly from three sites on each specimen (50 mm × 20 mm × 6 mm), and each group contained five replicates. An automatic micro-syringe was used to dispense approximately 1.5-µL drops of either distilled water or diiodomethane at a rate of 0.5 µL/s. The total surface free energy (γ_s), polarity surface free energy (γ_s ^P) and dispersive surface free energy (γ_s ^d) were calculated according to the Owens-Wendt-Kaelble method (Bao *et al.* 2004).

Surface roughness measurements were obtained by the stylus method in the direction perpendicular to the fiber on the wood surface and carried out using a Taylor Hobson Surtronic 3+ instrument (Metrology Instrument Taylor Hobson Ltd., England) at a constant speed of 1 mm/s over 15 mm of tracing length and a 2.5-mm cut-off across the sample grain. After the heat treatment, all untreated and heat-treated samples were sanded with 240-grit sanding paper twice according to the cross-sanding method (using a 2B pencil draw two lines at an equal distance of 10 mm across the surface perpendicular to the grain, and then scrubbing to wash away those lines with 240-grit sanding paper) before the surface roughness measurement. Four main roughness parameters, mean arithmetic deviation of profile (R_a), mean peak-to-valley height (R_z), root mean square roughness (R_q), and maximum roughness (R_y), were calculated according to international standard ISO 4287 (1997) to evaluate the effect of heat treatment on the surface characteristics of the specimens.

A Shore D Hardness Tester TH210 (Beijing Era South Light Technology Co., Ltd., Beijing, China) was used to measure the hardness. Hardness (HD) was calculated according to Eq. 2,

$$HD = 100 - \frac{L}{0.025}$$

(2)

where *L* specifies the displacement of the needle when the pressure foot surface is in full contact with the sample surface. Surface abrasion resistance was determined by the mass loss rate (*K* value) of samples sanded with a 240-grit sanding wheel. The mass loss was measured with a JM-IV instrument (Wuhan Gelaimo Testing Equipment Co., Ltd., Hubei, China). This protocol is based on international standard ISO 7784-1 (1997). Mass loss values were calculated after 100, 200, 300, 400, 500, and 1000 rotations.

For the surface roughness, hardness, and abrasion resistance parameters, multiple comparisons were first subjected to analysis of variance (ANOVA) and significant differences between average value of untreated and heat-treated samples were determined using Duncan's multiple range test.

Chemical Component and Morphological Analysis

The diffuse reflectance spectra for the five groups of samples were obtained using a Bruker FT-NIR spectrophotometer (MPA, USA) and the Bruker software OPUS7.2 by a cutoff wave number giving a total spectral range from 12,800 to 4000 cm⁻¹. Thirty-two scans of the tangential section of the sample were averaged with a spectral resolution of 8 cm⁻¹, and averaged and stored as log (1/reflectance) spectrum. Each group contained five replicates. Spectra scans were performed on the tangential section, as it is the most commonly used surface, also avoiding the color variation between latewood and earlywood.

Small wood chips (approximately 5 mm × 5 mm × 1 mm) were cut from heattreated and untreated wood surfaces, and all preparations were carried out just before the analysis, avoiding any contact with bare hands. The XPS analyses were carried out on an Escalab 250Xi (Thermo Scientific, USA) spectrometer with a pass energy of 10 eV and no monochromatic MgK α or AlK α radiation (h γ = 1253.7 eV and 1486.7 eV, respectively). The ratio of elemental oxygen to carbon (O/C) was determined from the low-resolution spectra, from 0 to 1100 eV. The C1s were determined from a high-resolution spectrum of the C1s region, from 280 to 300 eV. C1s peaks were divided into three components as follows. The C₁ peak corresponds to carbon atoms bonded with carbon or hydrogen atoms, C-H, or C-C. The C₂ peak corresponds to carbon atoms bonded to one oxygen atom, -C-O. The C₃ peak corresponds to carbon atoms bonded to a carbonyl or two non-carbonyl oxygen atoms, C=O or O-C-O, as well as carbon atoms bonded to a carbonyl and a noncarbonyl oxygen atom, O-C=O.

Scanning electron microscopy (SEM) scans were obtained using a scanning electron microscope FEG-XL30 (FEI, USA). Samples were covered by galvanic gold deposition using a MC1000 ion sputter (FEI, USA) with a current of 5 mA for 45 s. The analyses were performed with an acceleration voltage of 20 kV.

RESULTS AND DISCUSSION

Effects of Temperature on Contact Angle and Surface Energy

Table 1 shows the contact angle data for diiodomethane and distilled water, along with the surface free energy, for untreated and various heat-treated wood surfaces. The decline in wetting property and surface free energy were the highest when the wood was exposed to 220 °C for 2 h. The initial contact angles of two kinds of test liquid were higher

for heat-treated samples, and the surface energy of heat-treated wood increased with treating temperature.

Treatment	Weig	Density	Contac	Surface Energy				
	ht	(kg/m ³)				(mN⋅m ⁻¹)		
	Loss		Diiodomethane	H ₂ O	H ₂ O	γs ^d	γs ^p	γs
	(%)		(0 s)	(0 s)	(30 s)			
Control		446.94	34.93	71.59	18.85	34.98	8.07	43.05
		(71.69)	(4.2)	(3.1)	(4.0)			
Ht _{160 °C}	1.04	437.74	37.62	80.74	31.82	36.99	3.77	40.76
	(0.25)	(70.16)	(5.0)	(3.0)	(2.9)			
Ht _{180 °C}	1.40	431.92	37.95	79.50	39.25	36.31	4.35	40.66
	(0.48)	(62.04)	(4.9)	(4.2)	(6.5)			
Ht _{200 °C}	2.33	397.01	41.07	87.67	48.01	37.63	1.67	39.31
	(0.86)	(61.23)	(6.6)	(3.4)	(5.2)			
Ht _{220 °} c	5.11	385.58	42.08	90.32	64.78	37.55	1.13	38.68
	(0.56)	(58.95)	(5.2)	(3.9)	(4.4)			

Numbers in parentheses are standard deviation.



Fig. 1. Effect of temperature on time-dependent contact angle of distilled water

The contact angles of distilled water on the wood surface are displayed in Table 1. The increments of contact angles at 30 s reached 68.81%, 108.22%, 154.70%, and 243.66% as compared with untreated wood, which demonstrated the deterioration of the wetting property of heat-treated samples. Figure 1 displays the changes in the contact angle for untreated and heat-treated samples within 30 s after the drops of distilled water contact the surface of wood. The trend in the change of the contact angle on wood heat-treated at 160 and 180 °C resembles that of untreated wood. In contrast, a sharp decrease in the contact angle appears on wood heat-treated at 200 and 220 °C within 5 s, then maintains a relatively high and stable value. The decrease of the contact angle is primarily due to the interaction between water and wood surfaces, which changes with the absorption of droplets by the wood and their decrease in volume over time (Wang *et al.* 2015). However, it can be seen clearly from the Fig. 1 that the contact angles of heat treated wood were larger than that of untreated wood at the same moment. Moreover, the higher temperature of heat treatment,

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and the larger contact angles the wood surface possesses, which demonstrated that the deterioration affected the wetting property of heat-treated wood samples. The increased contact angle of heat-treated poplar wood was primarily due to the removal of the hydroxyl group.

The surface free energy of heat-treated samples decreased with increasing temperature, and it is noteworthy that the fraction of the polarity part of surface energy (γs^{P}) decreased prominently with increasing temperature. The fraction of the non-polarity part changed inversely. The slight decrease in the surface energy of heat-treated wood was attributed to the loss of free reactive hydroxyl groups present in hemicelluloses, which are strongly involved in wood wetting phenomena (Mitsui *et al.* 2008; Kamperidou *et al.* 2013). Gérardin *et al.* (2007) investigated the surface energy of beech (*Fagus sylvatica* L.) heartwood and pine (*Pinus sylvestris* L.) sapwood using the Lifshitz-van der Waals acid-base approach. The results showed that the electron-accepting component was less affected by thermal treatment, and that the decrease in the electron-donating component appeared to be closely related to the degradation of the hemicelluloses and the decrease of the O/C ratio.

Effects of Temperature on Surface Brittleness

Table 2 displays data for surface properties, including surface roughness, hardness values, and abrasion mass loss, for untreated samples and samples heat-treated at various temperatures.

Treatment	Unit	Surface	Surface Roughness (um) Surface Abras					
rioutiloni	Orme	Hardness			Mass Loss (mg)			
		Shore D						
		HD	Ra	R_q	Rz	Ry	r 100	r 200
Control	Avg.	51.80	4.92	6.80	34.44	47.22	-28.38	-57.18
	±S	1.65	0.53	0.81	3.09	4.67	8.83	12.13
	S ²	2.71	0.73	0.66	9.57	21.79	78.02	147.16
	V	3.18	10.77	11.95	8.98	9.89	-31.12	-21.22
Ht _{160 °C}	Avg.	50.61	5.08	7.36	42.58 [*]	60.08**	-48.82 [*]	-97.45 [*]
	±S	2.18	0.31	0.55	2.92	6.76	6.44	10.66
	S ²	4.74	0.10	0.31	8.50	45.75	41.44	113.58
	V	4.30	6.13	7.47	6.85	11.26	-13.19	-10.94
Ht _{180 °C}	Avg.	48.24*	5.94*	8.16 [*]	44.86**	60.08**	-71.3 [*]	-140.6 [*]
	±S	1.65	0.54	0.67	3.51	5.35	6.50	7.73
	S ²	2.72	0.29	0.44	12.33	28.66	42.27	59.75
	V	3.42	9.03	8.16	7.83	8.91	-9.12	-5.50
Ht _{200 °C}	Avg.	47.57 [*]	6.62**	9.12**	50.90**	67.10**	-83.88*	-165.43 [*]
	±S	1.05	0.36	0.59	4.32	7.05	10.38	15.31
	S ²	1.11	0.13	0.35	18.70	49.75	107.71	234.41
	V	2.21	5.49	6.51	8.49	10.51	-12.37	-9.26
Ht _{220 °C}	Avg.	42.26 [*]	7.14**	9.64**	51.70**	65.52**	-112.68 [*]	-224.03 [*]
	±S	1.79	0.80	1.06	3.25	5.69	6.89	6.60
	S ²	3.21	0.64	1.12	10.60	32.33	47.53	43.50
	V	4.24	11.19	10.99	6.30	8.68	-6.12	-2.94

Table 2. Surface Hardness, Roughness, and Abrasive Mass Loss of PoplarWood Subjected to Heat Treatment at Various Temperatures ^a

^a ±*s*=standard deviation, s^2 =Variance, V=coefficient of variation. Asterisks denote significant difference compared with the untreated control. All data in the variance and one-way ANOVA test were completed with confidence level p^{*} < 0.05, p^{**} < 0.01.

Significant reduction of surface hardness and abrasive mass loss values and an increase in surface roughness were observed for heat-treated wood. It can be speculated that the surface of the heat-treated poplar wood became more brittle, resulting in higher mass loss and surface roughness under a set sanding process, as well as lower surface hardness values.

Table 3. Percent Decrease in Hardness, Abrasion Resistance, and Surface Free
Energy, and Increase in Surface Roughness of Poplar Wood Subjected to Heat
Treatment at Various Temperatures

Treatment	5	Surface Ro	oughnes	s	Surface	Surface	Surface Free
		(%	5)		Hardness	Abrasive	Energy
					(%)	Resistance	(%)
					(%)		
	Ra	R_q	Rz	Ry	HD	K	γs
Ht _{160 °C}	3.25	8.24	23.64	27.23	2.30	123.53	5.32
Ht _{180 °C}	20.73	20.00	30.26	27.23	6.87	182.35	5.55
Ht ₂₀₀ ∘c	34.55	34.12	47.79	42.10	8.17	300.00	8.69
Ht _{220 °C}	45.12	41.76	50.12	38.75	18.42	411.76	10.15

An increase in the temperature of the heat treatment resulted in higher percent decrease value relative to untreated samples. The 220 °C heat-treated samples had the lowest values of surface hardness but the highest values of surface roughness and abrasion mass loss. A smaller effect of heat treatment was observed when the samples were exposed to 160 °C for 2 h. All differences were statistically significant (p < 0.05). Additionally, the results in Table 3 demonstrate that the variations of surface roughness values, hardness, and abrasion resistance increased with increasing treatment temperature. The smallest changes were found at the lowest treatment temperature, 160 °C. Among these surface characteristics, the largest decrease was found for surface abrasion resistance, followed by surface smoothness (increase in R_y , R_z , R_a , and R_q values), surface hardness, and surface free energy.

The decreases in surface hardness of heat-treated wood were found to be 2.30%, 6.87%, 8.17%, and 18.42%, respectively, when treated at 160 to 220 °C with an interval of 20 °C for a treatment time of 2 h. When exposed to temperatures lower than 200 °C, a slow decrease of hardness value was detected. However, the HD of specimens exposed to a temperature of 220 °C was 11.16% lower than that of samples treated at 200 °C within the same treating time of 2 h. Salca and Hiziroglu (2014) reported that the surface hardness value of red oak decreased by approximately 8.38% to 14.7% as a consequence of heating between 120 and 190 °C for 6 h, showing greater wood surface deterioration when exposed to a higher temperature (approximately 200 °C) for the same amount of time. Gündüz et al. (2008) studied the effect of heat treatment on compression strength and surface hardness of hornbeam (Carpinus betulus L.) wood, and the results showed that both compression strength and hardness decreased with increasing temperature. The hardness values in the tangential, radial, and longitudinal directions decreased by approximately 55%, 54%, and 38%, respectively, when treated at 210 °C for 12 h, with a density loss of 16.12%. The heat treatment process can improve the surface smoothness of wood, which is important for many applications (Ozcan et al. 2012; Bakar et al. 2013). The enhanced surface roughness of the wood exposed to a higher temperature could possibly be related to the degradation of cellulose and plasticization of lignin, as well as the migration of extractives from within the wood structure to the surface (Bakar et al. 2013; Tasdemir and Hiziroglu 2014).

However, a heat-treated wood surface usually needs to be sanded or planed before further processing and use. Therefore, studies of the roughness on a sanded wood surface could be more valuable for industrial production. The results from Table 2 indicate that surface roughness values of heat-treated samples, after being sanded twice according to the cross-sanding method, increased with increasing temperature under the conditions used. Exposure to a temperature of 220 °C resulted in an increase of 45.12% in R_a value and 50.12% in R_z value when compared to that of untreated poplar, after sanding equally. This result is confirmed by the observation that the roughness of a heat-treated wood surface, ranging from 160 to 220 °C for a certain time, increased substantially after the same sanding process. Surprisingly, the surface roughness values of sanded poplar wood increased relatively little when the treating temperature was 160 °C, but increased substantially when the treating temperature was higher than 180 °C. The maximum increasing amplitude value of surface profile values appeared when the temperature was 180 °C. At this temperature, the R_a value increased from 5.08 to 5.94, compared with that of wood treated at 160 °C. Previous studies have shown that the deterioration of hemicelluloses begins at or below 180 °C, while heat treatment of wood in the temperature range between 200 and 260 °C causes strong hemicellulose degradation, with an important release of acetic acid (Tjeerdsma et al. 1998; Shanahan 2002; Cademartori et al. 2013). Pelit et al. (2015) found that surface roughness values increase and brightness decreases with increasing temperature of heat treatment, which could be attributed to the grinding process performed before varnishing. In addition, Budakçi et al. (2011) studied the effects of different circular saws on surface roughness for heat-treated wood and found that heat treatment caused an increase in surface roughness after cutting with circular saws. The decrease in density, shown in Table 1, may be one reason for the increase of the roughness parameters (Gurău 2014).

As can be seen in Table 2, the surface abrasive mass loss values of heat-treated samples were distinctly higher than those of untreated samples within the same abrasive condition of 100 or 200 rotations. Samples from higher treatment temperature levels had a higher mass loss value when sanded equally. Increases in surface abrasive mass loss of heat-treated samples treated at 20 °C intervals from 160 to 220 °C were found to be 72.02%, 151.23%, 195.56%, and 297.04%, respectively, when compared with untreated samples under the same sanding condition of 100 rotations. Aysal *et al.* (2015) stated that a considerable decrease in the abrasion resistance of wooden material occurs in a manner dependent on the temperature increase after the heat-treatment process.

Moreover, a linear relationship between abrasion mass loss and sanding rotations for heat-treated samples was found to have a high confidence level ($\mathbb{R}^2 > 0.926$), as shown in Fig. 2(a). The surface abrasion resistance of heat-treated samples is reflected by the slope of the fitted regression lines, here termed *K* values. Results in Table 3 demonstrate that the K values of heat-treated poplar wood decreased compared with untreated samples and that higher treating temperature resulted in lower surface abrasion resistance.

The relationship between K values and treatment temperature, shown in Fig. 2(b), shows a significant linear correlation, with a high confidence level of 0.903. Furthermore, as shown in Fig. 2(c), the relationship between surface abrasion resistance (K value) or surface hardness (HD value) and weight loss (WL, %) indicates a strong correlation between the K or HD value and weight loss during heat treatment. The described correlation indicates that the abrasion resistance of heat-treated wood declined in a linear fashion. Therefore, the surface hardness or abrasion resistance values could be used to predict the intensity of heat treatment.



Fig. 2. Effects of temperature on (a) abrasion mass loss, (b) the relationship between *K* value and temperature, and (c) the relationship between HD and *K* values and weight loss during heat treatment of heat-treated specimens

As presented in Table 1, the mean density of heat-treated poplar decreased distinctly, from 446.94 to 385.58 kg/m³ when exposed to a temperature of 220 °C for 2 h, accounting for the degradation of cell wall components.

The breakage of the intermolecular and intramolecular bonds in the wood cell wall and the deterioration of microstructures during the heat treatment are potential reasons for the decrease in surface brittleness, as manifested by the decline in surface hardness and abrasion resistance and the increase in roughness. In addition, the increased fiber crystallinity during heat treatment may have caused the brittleness of the wood surface (Sun *et al.* 2010; Tomak *et al.* 2014; Pelit *et al.* 2015).

Effects of Temperature on Chemical and Morphological Changes

The FT-NIR spectra for both untreated specimens and specimens heat-treated at various temperatures for 2 h are shown in Fig. 3. Table 4 presents the assignment of absorption bands for the FT-NIR spectra, where bands numbered in the derived spectra are previously referenced in published reports (Tsuchikawa and Siesler 2003; Boeriu *et al.* 2004; Mitsui *et al.* 2008; Muñiz *et al.* 2013).

Among spectra for heat-treated specimens, some corresponding bands showed smaller absorption peaks. The degradation of cellulose and hemicellulose is evidenced by the changes in characteristic absorption peaks.

Wavenumber (cm ⁻¹)		Assignment				
1	7000	OH stretching first overtone, amorphous region of cellulose (Tsuchikawa and Siesler 2003)				
2	6900	OH stretching vibration of lignin (Heise and Winzen 1997; Tsuchikawa and Siesler 2003)				
3	6287	Crystalline regions of cellulose (Tsuchikawa and Siesler 2003)				
4	5974	Aromatic ring of lignin (Muñiz <i>et al.</i> 2013)				
5	5950	Overtone of aromatic skeletal CH stretching vibrations (Heise and Winzen 1997; Tsuchikawa and Siesler 2003)				
6	5800	CH stretching first overtone, furanose/pyranose functional group present in hemicellulose (Tsuchikawa and Siesler 2003)				
7	5587	Crystalline and semi-crystalline cellulose (Tsuchikawa and Siesler 2003; Muñiz <i>et al.</i> 2013)				

Table 4. Assignment of Absorption Bands of FT-NIR Spectra

In the case of wood, absorbance at wavenumbers 7000, 6287, and 5587 cm⁻¹ is assigned to the amorphous crystalline, crystalline, and semi-crystalline regions of cellulose, respectively. Relatively smooth regions in the peaks reflect decreased cellulose, especially at the amorphous region. Band 6, at 5800 cm⁻¹ is assigned to hemicellulose and represents large-scale reduction. Bands 2 and 4, at 6900 cm⁻¹ and 5974 cm⁻¹, respectively, correspond to OH stretching vibration in lignin and its aromatic ring, while the obvious enhancement in absorption peak at 6900 cm⁻¹ indicates increased lignin content.



Fig. 3. (a) FT-NIR and (b) second-derivative FT-NIR spectra for heat-treated specimens

The XPS analysis results in Table 5 show the atomic percentages and the three components of C1s, decomposed by Origin 8.0 software (OriginLab, USA) according to the total areas of the peaks, for heat-treated and untreated specimens. The typical C1s XPS survey spectra, as well as the high-resolution scans of the XPS spectra of C1s of untreated and heat-treated wood, are presented in Fig. 4.

Table 5. Atomic Percentages, O/C ratio, and C_1 , C_2 , and C_3 Distribution of Poplar Wood Exposed to Various Temperatures

Treatment	C%	O%	O/C	C ₁	C ₂	C ₃
Control	64.60	30.46	0.47	23.96	54.95	21.09
Ht _{160 °C}	75.18	24.82	0.33	36.57	45.47	17.96
Ht _{180 °C}	77.73	22.27	0.29	40.29	46.97	12.74
Ht _{200 °C}	79.93	20.07	0.25	41.16	44.79	14.05
Ht _{220 °C}	80.81	19.19	0.24	55.90	39.93	4.17

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Fig. 4. XPS spectra showing the influence of heat treatment on surface contaminations of poplar wood. Here, (a) represents the low-resolution spectra of untreated and heat-treated poplar wood (b) represents untreated wood, and (c) through (f) represent wood heat-treated at 160, 180, 200, and 220 °C, respectively.

Table 5 shows that the O/C ratio of heat-treated specimens decreased with increasing temperature. This could further improve the degradation of hemicellulose and the regeneration of lignin because the O/C ratio of holocellulose was much higher than that of lignin. High-resolution scans of the XPS spectra of C1s, along with their decomposition into three components, are displayed in Fig. 4. According to previous studies, the C₁, bonded with carbon or hydrogen atoms (C-H, or C-C bonds), can be attributed primarily to lignin and extractives like aliphatic acid, while the C₂, bonded to one oxygen atom (C-O), can be attributed to hydroxyl groups from cellulose and hemicellulose (Inari *et al.* 2006). It is worth noting that the C₃ in this study incorporated both carbon atoms bonded to a carbonyl and a non-carbon carbonyl oxygen atom (O-C=O). This is because

carbon atoms bonded with two oxygen atoms and three oxygen atoms are less abundant (Inari *et al.* 2006; Wang *et al.* 2015). The C₁ contribution of heat-treated samples increased, whereas C_2 and C_3 decreased markedly, indicating the degradation of the content of hydroxyl groups in hemicellulose and cellulose, or phenolic hydroxyl groups in lignin.

The degradation of hemicellulose and cellulose, manifested as shown in Figs. 3 and 5, could be the main reason for the decline in surface strength. The decrease of -OH groups and the contributions of C_2 and C_3 illustrate the reduction of oxidized carbon atoms and the deterioration of wetting. An increased proportion of the C_1 component could further certify the decrease in the surface free energy of heat-treated wood.

Previous studies have reported that the amount of holocellulose and α -cellulose decreased, and the proportion of lignin increased, under heat treatment. For example, the lignin content of treated specimens increased by 123.67% as compared with untreated specimens when Chinese white poplar wood was treated at 230 °C for 5 h (Huang *et al.* 2010). The degradation reactions, as well as crosslinking reactions of lignin, were active in the heat treatment process, while oxidation products from lignin contributed to surface color changes (Dubey *et al.* 2012; Olarescu and Campean 2014).



Fig. 5. SEM photos of untreated samples and samples heat-treated at 220 °C. Here, (a) represents untreated specimens, while (b), (c), and (d) represent specimens treated at 220 °C for 2 h.

To explore the effects of heat treatment on morphological changes of wood cell, the specimens treated at 220 °C for 2 h were chosen to compare with untreated wood, according to the larger decrease of surface brittleness discussed above.

The morphological characteristics of untreated specimens and specimens treated at 220 °C for 2 h appear in Fig. 5. These SEM photos (Fig. 5(b), (c)) show a more open

structure with distinctly increased size and number of pores, visible changes in pitting, and residual degradation products around the pores. Figure 5(d) presents stratified microstructures and some deformation of libriform fibers near the vessels. Similar results were found in previous research. For instance, Fengel *et al.* (1989) found cracks between the S1 and S2 layers and the median lamella of spruce wood treated at temperatures from 180 to 200 °C. These microstructure changes of the heat-treated wood cell wall may be explained by the degradation of cellular wall components, suggested by FT-NIR and XPS results, as well as vapor pressure during the heat modification (Boeriu *et al.* 2004). The cracks around the pits (Fig. 5(c)) and the laminarization of the cell wall could further certify the embrittlement and mechanical decline of a heat-treated poplar wood surface.

The strength of the cell wall on the heat-treated wood surface decreased, resulting in a decline of surface hardness and resistance to aluminum oxide powder on sand paper. Simultaneously, the cell wall of heat-treated wood became more brittle and easily delaminated by the shear force, which led to a larger amount of abrasion mass loss and deeper scratches, during the sanding process.

In further studies, it would be interesting to investigate the effects of surface brittleness of heat-treated poplar on bonding and painting to have a better understanding of the behavior of poplar wood exposed to heat treatment.

CONCLUSIONS

- 1. The wettability of heat-treated poplar decreased, while the surface brittleness increased. The largest decrease was found for surface abrasion resistance, followed by surface smoothness, surface hardness, and surface free energy.
- 2. The surface brittleness could be reflected by HD and K values, which show strong correlation with temperature or mass loss during heat treatment. Therefore, the K or HD values could be used as indicators to predict or regulate the degree of heat treatment.
- 3. FT-NIR and XPS spectroscopy were useful methods for investigation of chemical changes in wood under heat treatment, wherein hemicellulose, as well as amorphous and semi-crystalline cellulose, all evidenced degradation. SEM images further established the degradation of cell walls and explained the surface embrittlement of poplar wood.

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