EFFECT OF pH ON CHEMICAL COMPONENTS AND MECHANICAL PROPERTIES OF THERMALLY MODIFIED WOOD

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Abstract. To investigate the correlation between acidity and degradation during thermal treatment of wood, Cathay poplar (*Populus cathayana* Rehd.) wood samples were impregnated with solutions of different pH values, which included disodium octoborate tetrahydrate (DOT, pH = 8.3), monoethanolamine (MEA, pH = 12), and four buffering solutions composed of boric acid and sodium hydroxide (BA/NaOH, pH = 6, 7, 8, 9). Samples were then heated for 4 h at 180, 200, and 220°C, respectively. Bending MOR and MOE, mass losses, pH values, and percentages of lignin and hemicelluloses were subsequently determined in thermally modified samples and compared with control samples without pretreatment and/or thermal treatment. Results of the experiments indicated that DOT and buffering solutions decreased mass loss of thermally treated wood and increased bending MOR and MOE, whereas MEA pretreatment increased mass loss and showed comparable or even lower bending MOR and MOE than the untreated control with or without thermal treatments. Chemical analyses suggested that degradation of hemicelluloses was inhibited by DOT and BA/NaOH pretreatments within the temperature range 180-200°C, which may explain the mechanical property improvement.

Keywords: Thermally modified wood, pH value, mass loss, chemical components, mechanical properties.

INTRODUCTION

As an alternative to preserving wood with chemicals such as creosote, pentachlorophenol, and heavy metal-based systems, thermal modification

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(TM) of wood has been widely used in many areas. Previous studies showed that wood TM has the potential to improve dimensional stability, hygroscopic properties, and biological resistance without contaminating the environment (Kamdem et al 2002; Popper et al 2005; Tuong and Li 2010). However, the decrease in mechanical properties of TM wood can hardly be ignored and makes an undesirable impact on its structural characteristics (Bekhta and Niemz 2003; Gakhan and Deniz 2009).

Reasons responsible for the decrease of mechanical strength, which is the main limitation of TM wood, have been extensively discussed by researchers. The reasons are considered to be closely related to changes in chemical constitution of the wood during the heating process. Thermal treatment led to a variety of chemical changes, which depended on duration and heating temperature (Bourgois and Guyonnet 1988). Hemicelluloses were the first component to be degraded. Their degradation was identified as the major factor causing bending strength loss during TM (Esteves et al 2008). Degradation of hemicelluloses releases acetic acid, which acts as a catalyst for depolymerization of cellulose (McDonald et al 1999; Sivonen et al 2002). Higher treatment temperatures and longer treatment times will result in higher acid concentration, leading to more severe mass and mechanical property loss (Sundqvist et al 2006). Other changes include the apparent increase of cellulose crystallinity and lignin content (Dirol and Guyonnet 1993; Akgul et al 2007).

Consequently, pre-impregnation was introduced as a method to moderate the decline of mechanical strength caused by TM. Winandy (1997) found that adding borate buffers to the fire retardant treatment chemicals appeared to significantly mitigate subsequent thermal degradation. Awoyemi (2008) suggested that preimpregnation of borate as an alkali-buffering medium decreased the severity of strength loss during TM. This was invariably caused by the buffering effect of alkali on wood acidity. Kartal et al (2008) evaluated effects of boron impregnation and heat treatment on mechanical properties of Sugi sapwood and discovered that boric acid (BA) and disodium octoborate tetrahydrate (DOT) treatments obviously changed the pH value of wood, which made the decrease in MOE in the untreated wood slightly higher than that in the treated specimens. However, little attention has been devoted to correlations among decrease in strength loss, chemical changes, and pH value of pretreated wood. Little data exist in the literature about the effect of pH value on various properties of wood.

The objective of this study was to investigate the impact of pH on chemical component changes and resulting changes in mechanical properties of thermally modified wood.

MATERIALS AND METHODS

Preparation of Wood Samples

Wood samples measuring 20 (R) × 20 (T) × 300 (L) mm were obtained from sapwood of Cathay poplar (*Populus cathayana* Rehd.) harvested from Lesser Khingan Range in northeastern China. Initial moisture content of the samples was about 8% after air-drying. Samples were weighed and sorted into 28 groups (Table 1) with six replicates in each group. All samples were free of visible defects and evidence of infection by mold, stain, or wood-destroying fungi.

Pretreatment of Wood Samples

Wood samples were impregnated with solutions of different pH values by a full-cell process (Freeman et al 2003). Initial vacuum was at –0.1 MPa for 1 h, and the pressure cycle was at 0.5 MPa for 2 h at room temperature. Solutions used for pretreatment included DOT (2% concentration, pH = 8.3), monoethanolamine (MEA, 2% concentration, pH = 12), and four buffering solutions composed of BA and sodium hydroxide (NaOH, 2% BA neutralized to pH = 6, 7, 8, 9). After impregnation, samples were taken out from the treating tank, wiped with a clean paper towel to remove excessive solutions, air-dried for 3 da at room temperature, and then oven-dried at 105°C for 4 more da.

Table 1. Grouping of test samples used for different pretreatments and thermal treatments.

Labeling of samples	Treatments	
	Pretreatment	Temperature of thermal treatment
C-C	—	—
C-D	DOT	—
C-BN6	BA/NaOH (pH = 6)	—
C-BN7	BA/NaOH (pH = 7)	—
C-BN8	BA/NaOH (pH = 8)	—
C-BN9	BA/NaOH (pH = 9)	—
C-M	MEA	—
180C	No	180°C
180D	DOT	180°C
180BN6	BA/NaOH (pH = 6)	180°C
180BN7	BA/NaOH (pH = 7)	180°C
180BN8	BA/NaOH (pH = 8)	180°C
180BN9	BA/NaOH (pH = 9)	180°C
180M	MEA	180°C
200C	No	200°C
200D	DOT	200°C
200BN6	BA/NaOH (pH = 6)	200°C
200BN7	BA/NaOH (pH = 7)	200°C
200BN8	BA/NaOH (pH = 8)	200°C
200BN9	BA/NaOH (pH = 9)	200°C
200M	MEA	200°C
220C	No	220°C
220D	DOT	220°C
220BN6	BA/NaOH (pH = 6)	220°C
220BN7	BA/NaOH (pH = 7)	220°C
220BN8	BA/NaOH (pH = 8)	220°C
220BN9	BA/NaOH (pH = 9)	220°C
220M	MEA	220°C

DOT, disodium octoborate tetrahydrate; BA/NaOH, boric acid and sodium hydroxide; MEA, monoethanolamine.

Thermal Treatment

All impregnated and untreated samples were oven-dried and weighed before thermal treatment. Afterward, they were heated at 180, 200, and 220°C, respectively, for 4 h in a modified drying oven connected with a water vapor supplying system, which used a big container to hold water and then produce water vapor as a protecting medium. Thermally modified samples were oven-dried again and reweighed. Mass loss (ML) was calculated according to Eq 1:

$$\mathrm{ML}(\%) = (\mathrm{M1} - \mathrm{M2})/\mathrm{M1} \times 100 \qquad (1)$$

where M1 = mass of samples before heat treatment (g) and M2 = mass of samples after heat treatment (g).

Determination of pH Value

For pH measurement, wood samples were ground and passed through 40-60 mesh screens. Three grams of oven-dried sawdust samples were soaked in 30 mL of distilled water and then stirred for 5 min, let sit for 15 min, stirred for another 5 min, and then let sit for another 20 min. After the procedure, pH values of the liquid were determined using a pH meter (PHB-5; Shanghai LIDA Instrument Factory, Shanghai, China).

Chemical Analysis of Wood Components

Wood specimens for chemical analysis were ground, passed through 40-60 mesh sieves, and dried at 60°C for 2 da. Prepared sawdust was extracted with a benzene–ethanol solution (2:1) to remove the nonpolar extractive fraction and was then dried. About 1 g dried extracted sawdust was used to determine acid insoluble lignin (AIL) according to ASTM D 1106-96 (ASTM 2007) using sulfuric acid. About 2 g dried extracted sawdust was used to determine holocellulose content by the sodium chlorite method, and 1 g dried extracted sawdust was used to determine cellulose content by the nitric acid–ethanol method. Hemicellulose content was calculated by subtracting cellulose content from holocellulose content.

Fourier Transform IR Analysis

Fourier transform IR (FTIR) spectra were collected using an FT-IR spectrophotometer (Nicolet 510; Nicolet Thermo Corp, Edina, MN). The potassium bromide disk containing 1% finely ground samples was prepared and then analyzed and recorded in transmission mode within the range 4000-400 cm^{-1}. All spectra were recorded at 2 cm^{-1} resolution, and 32 scans were accumulated.

Mechanical Test

All sample groups were conditioned in a humidity chamber at 20°C and 65% RH for 15 da before testing. Static bending tests were performed using

the universal mechanical test machine (WDW-350A; Jinan Shijin Group Co., Jinan, Shandong, China) using the three-point bending test method with a span of 240 mm. Average MOE and MOR of six replicates for each condition were obtained. Then percentage decreases in MOR and MOE after heat treatments at different temperatures were calculated from ratios of average MOR and MOE values of heat-treated samples to those of unheated samples with the same pretreatment. For example, percentage decrease in MOR for 180D was calculated from the ratio of MOR of 180D to MOR of C-D.

RESULTS AND DISCUSSION

pH Value

As shown in Fig 1, pH values of wood specimens after pretreatment were positively correlated with pH values of treating solutions. pH values of control samples without pretreatment were the lowest. With increasing pH value of the treating solution, pH value of treated wood increased accordingly. After thermal treatments, pH levels of untreated specimens and specimens treated with low pH solutions hardly changed, whereas those of specimens treated with high pH solutions showed obvious decrease at 200 and 220°C. MEA-treated specimens showed a different trend from DOT- and BA/NaOH-treated specimens, which was considered to be related to its volatility. Organic acids, especially acetic acid, released during the heat treatment, catalyzed the degradation of polysaccharides and then volatilized out of wood at high temperatures (Alén et al 2002). However, added alkaline substances neutralized the released acid, which made pH value of wood specimens treated with high pH value solutions decrease significantly with elevating heating temperature. Similar changes have been observed in DOT- and BA-pretreated thermally modified Sugi (Kartal et al 2008).

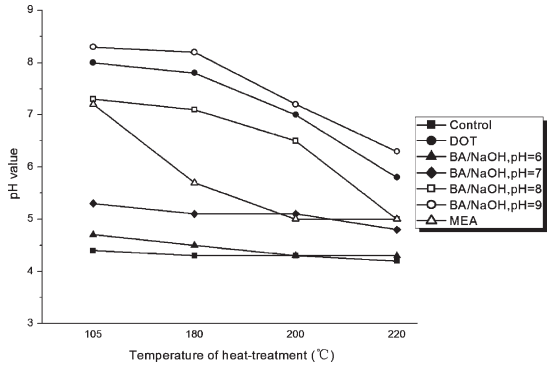

Figure 1. pH values of thermally modified wood samples pretreated with different solutions.

Chemical Component Analysis

Table 2 lists changes in wood chemical components after different pretreatments with various solutions and after thermal treatments at

Table 2. Chemical analysis of primary components of wood samples after different combinations of pretreatment with pH buffering solutions and thermal treatment.

Labeling of samples[a]	Main components (%)			
	Holocellulose	α-cellulose	Hemicelluloses	AIL[b]
C-C	80.70	49.24	31.46	19.86
C-D	79.47	48.62	30.86	19.40
C-BN6	79.91	50.50	29.41	19.54
C-BN7	78.42	49.64	28.77	19.10
C-BN8	78.26	50.70	27.56	19.90
C-BN9	77.84	50.57	27.26	19.94
C-M	74.21	50.00	24.21	19.47
180C	75.98	49.80	26.18	19.73
180D	72.58	48.29	24.29	19.72
180BN6	73.80	48.91	24.90	19.63
180BN7	72.97	48.95	24.02	19.26
180BN8	73.07	49.34	23.73	19.32
180BN9	71.35	47.74	23.61	19.87
180M	71.29	50.11	21.17	19.78
200C	72.54	50.74	21.80	20.70
200D	70.52	47.50	23.03	19.57
200BN6	72.50	48.61	23.89	19.43
200BN7	71.69	48.71	22.97	19.49
200BN8	71.11	48.71	22.40	19.64
200BN9	70.96	48.56	22.40	19.83
200M	68.06	49.77	18.28	20.00
220C	66.13	52.28	13.85	22.74
220D	64.88	49.59	15.29	20.41
220BN6	66.34	50.65	15.68	20.35
220BN7	65.38	48.37	17.01	20.02
220BN8	63.37	48.48	14.89	20.68
220BN9	63.25	48.59	14.66	20.30
220M	66.83	51.02	15.81	20.66

[a] See Table 1 for definition of labeling.
[b] AIL, acid insoluble lignin.

different temperatures. Because the temperature of thermal degradation for cellulose is very high, almost no change was found in the amount of α-cellulose after different treatments. Similar results were reported by Yildiz et al (2006). Therefore, the significant decline of holocellulose content was mainly caused by degradation of hemicelluloses.

Heat treatment at 180, 200, and 220°C for 4 h caused remarkable decreases in hemicelluloses, as Fig 2 clearly shows. Degradation of hemicelluloses was inhibited by DOT and BA/NaOH pretreatments within the temperature range 200-220°C, whereas alleviation of the degradation was not remarkable at 180°C. Results of this study agree well with Kartal et al (2008) who found that the buffering action of BA and DOT solutions did not affect degradation of hemicelluloses such as arabinan, glactan, xylan, rhamnan, and mannan at 180°C but inhibited degradation of hemicelluloses at 220°C. There was no significant difference between DOT and the other four BA/NaOH solutions. Although the remaining hemicelluloses of MEA-treated wood samples showed a similar trend to DOT- and BA/NaOH-treated wood samples (Fig 2), the absolute value of hemicellulose content of MEA-pretreated wood was obviously lower than other pretreatments below 200°C (Table 2). This was considered to be caused by severe degradation of hemicelluloses under the high alkaline condition.

A significant increase in the amount of AIL was found in nonpretreated specimens after TM, whereas the pretreated and then TM specimens showed comparable AIL content with unheated specimens (Fig 3). No significant difference was found among pretreated specimens. Changes in AIL content were caused by hemicellulose removal during heat treatment (Boonstra and Tjeerdsma 2006), hence showing related trends to the hemicelluloses.

Fourier Transform IR Analysis

To understand the changes in wood components at the molecular level, samples after different pretreatments and different thermal treatments were analyzed using FTIR. Thermal degradation started from deacetylation. During this process, acetyl groups were broken and ester groups were formed (Tjeerdsma et al 1998), and thereafter, acid-catalyzed hydrolysis of the ester occurred, which finally resulted in decreases of carbonyl groups. Figure 4 shows FTIR spectra of control, 220°C heat-treated samples without pretreatment, and 220°C heat-treated samples with DOT pretreatment. The peak observed at about 1740 cm^{-1} was assigned to absorption of carbonyl stretching of ester (Tjeerdsma and Militz 2005). The diminution of the peak at 1740 cm^{-1} confirmed that cleavage of acetyl groups occurred at high temperatures, whereas decrease of 220°C heat-treated samples with DOT pretreatment

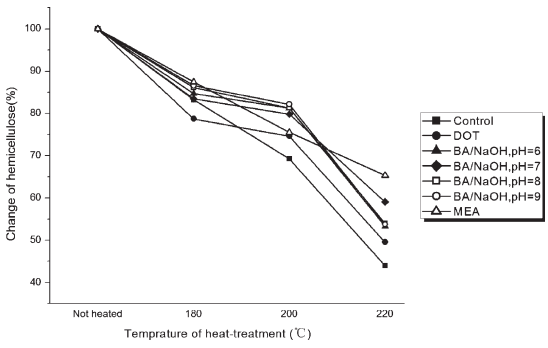

Figure 2. Change of hemicelluloses content in wood samples after different pretreatments with various solutions and thermal treatments at different temperatures.

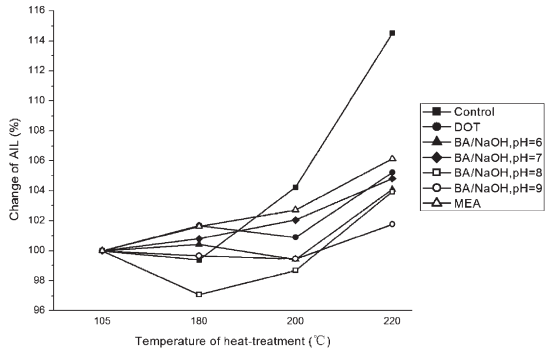

Figure 3. Change of acid insoluble lignin (AIL) content in wood samples after different pretreatments with various solutions and thermal treatments at different temperatures.

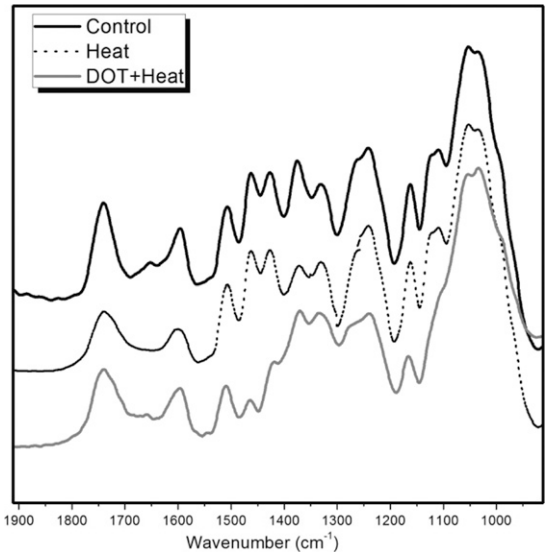

Figure 4. Fourier transform IR spectra of control sample, 220°C heat-treated sample without pretreatment and 220°C heat-treated sample with disodium octoborate tetrahydrate (DOT) pretreatment.

was less than that of 220°C heat-treated samples without pretreatment, which indicated that DOT pretreatment alleviated hemicellulose degradation. The peak at 1650 cm^{-1} also decreased during heat treatment, which was caused by formation of diphenylmethane structures from condensation of lignin during heating (Funaoka et al 1990). The different intensity of this peak shown in three spectra also indicated the assuasive effect of DOT pretreatment. FTIR spectrum of BA/NaOH-pretreated samples was similar to the spectrum of DOT-pretreated samples (not displayed in Fig 4).

Mass Loss

Mass losses in samples after various treatments were calculated after heat treatment. Results showed that mass losses increased remarkably with elevating heating temperature, whereas DOT and buffering solutions decreased mass loss of thermally treated wood under all temperature conditions, especially at higher temperatures (Fig 5). MEA-treated samples were different from other pretreated samples because

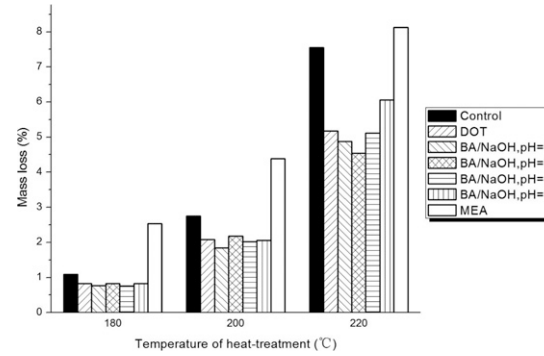

Figure 5. Mass loss of wood samples after different pretreatments with various solutions and thermal treatments at different temperatures.

they showed increased mass loss compared with nonpretreated controls, which was attributed to severe degradation of wood components under a high alkaline condition.

Mechanical Properties

Table 3 lists average values of bending MOR and MOE of all samples. Results indicated that bending MOR and MOE decreased significantly after thermal treatment, and the decrease was positively correlated with heating temperature. According to chemical analysis data, hemicellulose content in samples decreased with increasing heating temperature. Therefore, the decrease in bending MOR and MOE was highly correlated with the thermal degradation of hemicelluloses. This result agreed with previous studies. Boonstra et al (2007) suggested that loss of mechanical strength, especially bending strength, during TM was mainly caused by hemicellulose degradation.

Figures 6 and 7 compared percentage decreases in mechanical properties of thermally treated wood with or without pretreatment, which showed that decreases in bending MOR and MOE during heat treatment were lower for DOT- and BA/NaOH-pretreated samples than for nonpretreated samples. This is consistent with results of mass loss shown in Fig 5. This was also reported in previous studies. For example, Kaygın

Table 3. Bending MOR and MOE of wood samples after different combinations of pretreatment with pH buffering solutions and thermal treatment.

	MOR (MPa)		MOE (MPa)	
Labeling of samples[a]	Average	SD[b]	Average	SD
C-C	74.8	8.9	5722	263
C-D	75.7	5.7	6137	638
C-BN6	79.1	13.2	5925	557
C-BN7	79.6	7.5	6059	715
C-BN8	78.7	6.9	6012	468
C-BN9	77.9	4.3	6030	713
C-M	72.7	11.0	5326	724
180C	67.5	4.8	5383	649
180D	74.2	5.1	5841	666
180BN6	68.8	3.5	5698	998
180BN7	71.7	5.3	5665	617
180BN8	71.5	4.4	5776	468
180BN9	70.7	7.0	5810	541
180M	67.5	10.6	5243	667
200C	59.4	2.7	5136	712
200D	64.8	1.5	5705	448
200BN6	64.4	3.5	5582	683
200BN7	64.8	2.8	5512	255
200BN8	64.0	4.3	5668	437
200BN9	69.5	3.4	5790	586
200M	59.0	8.8	5074	1062
220C	40.1	5.5	4487	156
220D	50.4	12.9	5265	414
220BN6	51.2	14.8	5464	470
220BN7	53.5	8.0	5455	992
220BN8	53.9	13.4	5505	966
220BN9	51.8	8.9	5468	365
220M	45.2	5.4	4703	381

[a] See Table 1 for definition of labeling.
[b] SD, standard deviation.

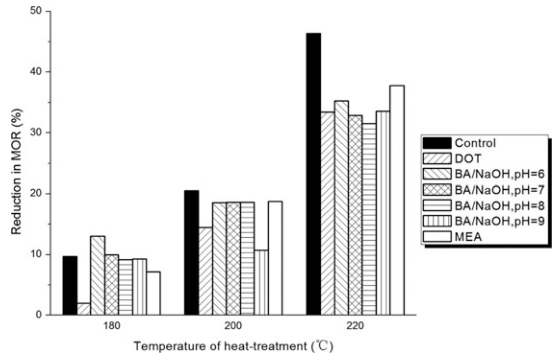

Figure 6. Percentage decreases in bending MOR of wood samples after different pretreatments with various solutions and thermal treatments at different temperatures.

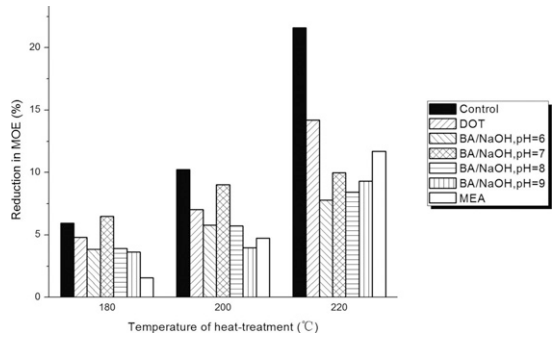

Figure 7. Percentage decreases in bending MOE of wood samples after different pretreatments with various solutions and thermal treatments at different temperatures.

et al (2009) reported that heat treatment of wood results in a decrease in mass and the decrease was related to mechanical properties. This is reasonable because they are both tightly related to thermal degradation of hemicelluloses. DOT and BA/NaOH pretreatments could mitigate the degree of degradation of wood subjected to thermal modification. Awoyemi and Westermark (2005) studied effects of disodium tetraborate impregnation on response of wood strength to heat treatment and investigated the similar result that pretreatment mitigated the degree of degradation during heat treatment. Results also indicated that the mitigating effect of DOT and BA/NaOH pretreatments on strength loss increased as temperature increased. According to independent-samples t-test, all p values of the test of differences in MOR and MOE between DOT and BA/NaOH pretreatments and nonpretreated samples were less than 0.05, suggesting that improvement of bending MOR and MOE by DOT and BA/NaOH pretreatments was statistically significant. Figures 6 and 7 and Table 3 show that MEA-pretreated wood had comparable or even lower bending MOR and MOE than the nonpretreated control before or after thermal treatments. However, no significant differences in bending MOR and MOE were found among the various buffering solutions ($p > 0.05$).

CONCLUSIONS

Impregnation of wood with DOT and BA/NaOH buffering solutions before thermal treatment could mitigate the decrease in mass loss and

mechanical properties of thermally modified wood by alleviating the thermal degradation of hemicelluloses. As demonstrated by FTIR analysis results, changes were undoubtedly attributed to the neutralizing effect of these alkaline solutions on the acidity of wood. Whereas the MEA pretreatment was different, it increased mass loss and showed comparable or even lower bending MOR and MOE than the nonpretreated control, which was considered to be related to its volatility. However, differences among various buffering solutions were negligible within the experimental conditions of this study.

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