

Effect of Thermal Treatment on Functional Groups and Degree of Cellulose Crystallinity of Eucalyptus Wood (*Eucalyptus grandis* × *Eucalyptus urophylla*)

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Abstract

In this study, eucalyptus wood was subjected to thermal treatment with super-heated steam under various conditions. The effects of temperature and exposure time on the functional groups and degree of cellulose crystallinity of the eucalyptus wood (*Eucalyptus grandis* × *Eucalyptus urophylla*) were analyzed. The results showed that the thermal treatment caused significant changes in the chemical composition and structure of wood. The Fourier transform infrared spectroscopy (FTIR) absorption peaks of the hydroxyl group decreased from 3,425 to 3,250 cm^{-1} for all thermally treated samples. Higher treatment temperature and extended exposure led to the lower methyl levels, with almost no methyl present at 220°C. The aromatic ring first increased and then decreased with increasing temperature and exposure time. The carbonyl group maintained the same intensity, with small fluctuations, across the different thermal treatment conditions. Compared with the untreated wood sample, the X-ray diffraction peak position of thermally treated samples shifted slightly left from 22.02° to 21.06°, and the distance between crystal planes increased. The degree of cellulose crystallinity of eucalyptus wood was affected significantly by treatment temperature with a confidence level of 99 percent.

To resolve the contradiction between supply and demand of timber and alleviate the ecological crisis on Earth, most countries around the world are devoted to developing industrial, fast-growing plantations to replace dwindling space for wood resources (Cheng and Song 2006, Liu et al. 2011). Eucalyptus (*Eucalyptus grandis* × *Eucalyptus urophylla*), one of the most important fast-growing trees worldwide, has been widely planted in more than 100 countries because of its strong adaptability, fast growth, and relatively high economic value (Huang 2010). However, defects including deformation, discoloration, cracking, and decay can easily occur in products made from eucalyptus, especially when these products are left outside. These defects originate from the intrinsic material properties of eucalyptus wood, such as high growth stress, low permeability, and poor weather and corrosion resistance. Shortages of eucalyptus wood limit its application and decrease the added value of eucalyptus products. Therefore, considering all of the above shortcomings of this fast-growing wood, there are still many challenges to be addressed on a global scale before the plantation wood can be used (De Moura and Brito 2011).

Wood modification is recognized as an effective method to improve the qualities of wood products. Wood modification can be classified into two main types: chemical modification and physical modification (Avramidis et al. 2011). Chemical modification involves impregnating wood with polymers, resins, or other chemicals to fill in voids within the wood, but it can be harmful to the environment

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and human health. The thermal treatment of wood, as a physical modification method, seems to be an eco-friendly and viable alternative to chemical modification (Li et al. 2008). In this method, wood is treated with steam at a temperature of at least 160°C (Tjeerdsma and Militz 2005, Inari et al. 2007). In recent years, researchers have studied aspects of thermal wood treatment (Yan et al. 2002, Xie et al. 2006), including equipment modification (Hatakeyama and Hatakeyama 2004), treatment-process optimization (Boonstra et al. 2007), and analysis of the thermal mechanism (Bekhta et al. 2003). During the process of thermal wood treatment, chemical changes occur within the wood (Windeisen et al. 2007). These changes include a lowering of the fiber saturation point and a decrease in the amount of hemicellulose and accessible hydroxyl groups (Michiel and Tjeerdsma 2006), which results in improved durability, dimensional stability, and weather resistance (Poncsak et al. 2006). Researchers have sought to determine why these changes take place. For example, Fourier transform infrared (FTIR) spectroscopy has been an extremely powerful tool used by researchers to examine the physical and chemical structure of materials and their chemical composition (Esteves and Marques 2013). The absorption observed in the FTIR spectra of wood materials was assigned to the mixture content of functional groups with C, H, and O elements in cellulose, hemicelluloses, lignin, extractives, and water (Rodrigues et al. 1998). These spectral characteristics were suitable for determining the density and chemical composition of cellulose and lignin (Fan et al. 2014). It is known that the crystalline regions in cellulose exhibit higher density and lower chemical reactivity than other less-ordered regions in the fiber. X-ray diffraction (XRD) is recognized as a universal method to analyze the special internal structure of crystalline and the degree of crystallinity of wood (Franceschi et al. 2008, Li et al. 2014).

As mentioned above, the combination of FTIR and XRD analysis is designed to monitor the changes of chemical composition and structure within the wood. The objective of this study was to evaluate the effect of thermal treatment on functional groups and the degree of cellulose crystallinity of eucalyptus wood, as well as to propose mechanisms whereby thermal treatment enhances the properties of the wood in our previous work (Cheng et al. 2015). It is hoped that the results obtained in this study can be referenced for the industrial implementation of the thermal treatment of eucalyptus wood.

Materials and Methods

Air-dried flat sawn lumber of a 10-year-old eucalyptus tree (*E. grandis* × *E. urophylla*) was obtained from the Guangxi Province in China. The average moisture content of the sawn lumber was approximately 12 percent. Test samples with dimensions of 300 mm (length) by 40 mm (width) by 20 mm (thickness) were prepared from the air-dried lumber. The specimens were free of knots, discoloration, mold, and other visible defects.

Thermal treatment was performed at four different temperature levels (160°C, 180°C, 200°C, and 220°C) for 4 hours, or at four different treatment times (1, 2, 3, and 4 h) with a set temperature of 200°C. All treatments involved heating with steam at atmospheric pressure in a laboratory thermal treatment oven (Model Type DN325×650, China), resulting in seven different treatment conditions. In the

experiment, the oven was first raised to 110°C at a rate of 20°C/h and then was held at 110°C for 60 minutes to reduce the moisture content of wood samples. Finally, the oven was heated to the set temperature and kept there for a designated amount of time until the thermal treatment experiment was finished. During the thermal treatment, steam was generated within the oven to make sure that the specimens did not burst into flames. Following treatment, the treated samples were left in the oven until the oven cooled down to room temperature.

After treatment, samples were conditioned indoors for 1 month prior to characterization, and all samples were later split, crushed, sieved (200 mesh), oven-dried at 103°C for over 24 hours, and ground. Afterward, an FTIR spectrometer (Impact type 410, USA) and X-ray diffractometer (XRD-TTR3, Japan) were used to analyze the functional groups and the degree of cellulose crystallinity, respectively, of the treated and untreated samples. For the FTIR analyses, the wood powder was mixed with KBr at a ratio of 1 to 100, and the FTIR scan wave number that was used ranged from 4,000 to 400 cm⁻¹. For the XRD measurements, the instrument parameters were set as follows: sample stack trace ranged from 5° to 50°; scanning speed was 2°/min; sampling step width was 0.01°; copper metal target for irradiation (with radiation emission of λ = 0.154 nm); radiant tube voltage was 36 kV; and radiant tube current was 20 mA. The spectra results are the average values from 10 replications for two replicated samples in each treatment condition.

Segal et al. (1959) found a simplified method for the measurement of crystallinity with the probability of error around 6.5 percent. The relative degree of cellulose crystallinity was calculated using Equation 1,

$$C_rI = \frac{I_{002} - I_{am}}{I_{002}} \times 100\% \quad (1)$$

where C_rI is the percentage of relative crystallinity (%), I_{002} is the maximum intensity of diffraction in 002 interference, and I_{am} is the intensity of diffraction for amorphous scatter at $2\theta = 18^\circ$. When conducting the calculations, 10 replications were tested for each treatment.

Results and Discussion

Effect of thermal treatment on functional groups of eucalyptus wood

The infrared (IR) absorption spectra of the thermal treated and untreated samples from 4,000 to 400 cm⁻¹ are shown in Figures 1 and 2. The characteristic frequencies in this study were mostly located in the range of 4,000 to 1,300 cm⁻¹, as most functional groups showed their characteristic absorptions in this range (Li 2003). In order to show more than one spectrum in each figure, a shift in the y axis was necessary.

It can be seen from these FTIR spectra that the characteristic absorptions for the untreated sample included the stretching vibration of free hydroxy groups (–OH) derived from cellulose, hemicelluloses, and lignin at 3,425 cm⁻¹; the stretching vibration of methyne (–CH), methylene (–CH₂), and methyl (–CH₃) from cellulose, hemicelluloses, and lignin at 2,880 cm⁻¹; the stretching vibration of carbonyl (C=O), attributed to the acetyl groups of xylose from hemicelluloses at 1,735 cm⁻¹; the vibration of aromatic phenyl group from lignin at 1,435 cm⁻¹; and the

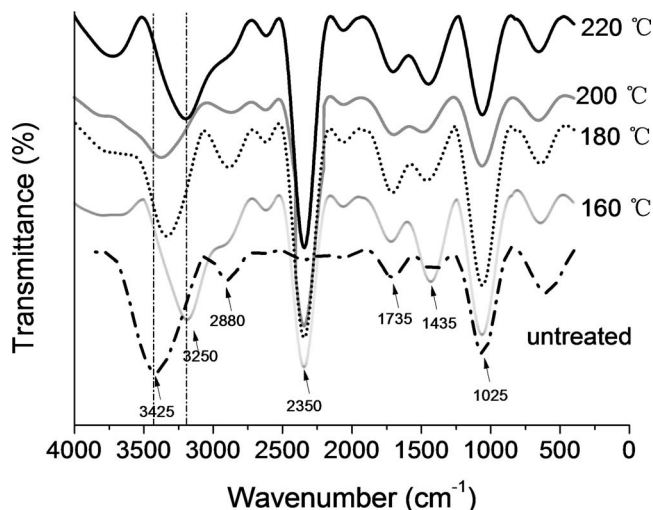


Figure 1.—Fourier transform infrared spectra of eucalyptus wood thermally treated at different temperatures for 4 hours.

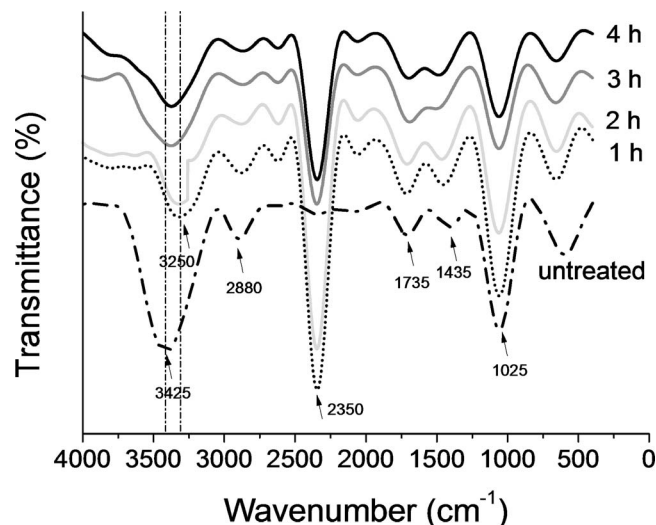


Figure 2.—Fourier transform infrared spectra of eucalyptus wood thermally treated at different times at 200°C.

vibration of the polysaccharide carbon from the hemicelluloses at $1,025\text{ cm}^{-1}$ (Table 1).

The FTIR spectra of treated samples showed a migration to lower wavenumbers of the group at $3,425\text{ cm}^{-1}$ corresponding to the O–H stretching vibration from alcohols ($3,600$ to $3,300\text{ cm}^{-1}$) and carboxylic acids ($3,300$ to $2,500\text{ cm}^{-1}$). This migration might be because of the increase in carboxylic acids owing to primary OH oxidation and/or hydrolysis of acetyl groups from hemicelluloses; the changed cellulose crystallinity also influenced this process by dehydration (Spiridon et al. 2011). The smallest peak in O–H group appeared at 200°C , which can be clearly seen in Figure 2. This finding is owing to carbohydrate degradation, which reduces accessible OH (Weiland and Guyonnet 2003); moreover, the cross-linking of free hydroxyl groups removes a molecule of water and generates an ether bond, resulting in a decrease in the amount of hydroxyl groups (Inari et al. 2007).

The three groups at $2,970$ to $2,900\text{ cm}^{-1}$ are composed of methyl ($-\text{CH}_3$), methylene ($-\text{CH}_2$), and methyne ($-\text{CH}$). The difference in absorption peak intensity between the treatment and control samples is due to structural and relative composition changes, namely, changes at the cellulose crystallinity level, which influence the C–H and O–H stretch frequencies (Spiridon et al. 2011). With higher treatment temperatures and longer durations, the lower

intensity at $2,880\text{ cm}^{-1}$ appeared (shown in both Figs. 1 and 2), but almost disappeared when the temperature was increased to 220°C .

A new and obvious strong absorption peak at $2,350\text{ cm}^{-1}$ for all thermal-treated samples was observed, which is always defined as the stretching vibration of CO_2 . Considering the process was the same for the control and treated samples, the difference shown here cannot be ignored, and the reason this difference occurs will need to be explained in future research. The absorption peak at $1,735\text{ cm}^{-1}$ was C=O. According to Michell and Higgins (2002), the group around $1,730\text{ cm}^{-1}$ is almost exclusively due to the carbonyl groups of acetoxy groups in xylan. The decrease of the $1,735\text{ cm}^{-1}$ peak could be attributed to the cleavage of acetyl or acetoxy groups in xylan, and the increase derived from carbohydrate loss (Li et al. 2002). The carbonyl content change could be explained by the early research on chemical changes of thermal-treated wood by Tjeerdma and Militz (2005): when wood samples were exposed in an environment with high temperature and humidity, acetic acid was generated from the hydrolysis of ethanoyl within polysaccharide molecular chains of hemicellulose, and the content of carbonyl was decreased. Apart from that, according to Inari et al. (2007), an esterification reaction occurs within the lignin of the wood cell wall under acidic conditions, also resulting in an increase in the amount of carbonyl. Based on the combined effect of these factors, it can be concluded that the amount of carbonyl groups reached a dynamic balance and present a constant intensity value of the absorption peak.

The group at $1,435\text{ cm}^{-1}$ corresponds to vibrations in the aromatic ring, which first increased and then decreased with higher temperatures and longer treatment times. This group increased when the temperature was lower than 180°C and the treatment time was less than 2 hours, which was caused by an increase in the percentage of lignin in the thermal-treated wood (Li et al. 2002). The decrease in content of this group may be due to a decrease in the methoxyl groups in lignin, leading to the loss of a monomer. Previous research indicates that a shift occurs in this group, which can be

Table 1.—Main groups of infrared spectra of wood and their assignment to functionality.

Wave no. (cm^{-1})	Functionality	Vibrating type ^a
3,450–3,400	–OH of alcohols, phenols and acids	O–H stretching
2,970–2,900	–CH, CH_2 and CH_3	C–H stretching
1,750–1,720	C=O of esters, ketones, aldehydes and acids	C=O stretching
1,700–1,350	C=O and C=C	C=O and C=C stretching
1,455–1,420	Aromatic ring	Benzene ring stretching
1,035–1,025	C–O–C	Deformation

^a From Rodrigues et al. (1998) and Michell and Higgins (2002).

attributed to the breaking of aliphatic side-chains in lignin and/or condensation reactions (Windeisen et al. 2007).

After exploring possible reasons for the improved dimensional stability and wettability of wood following thermal treatment, it could be concluded based on the chemical analysis of FTIR that there were two possible reasons: (1) it reduced the amount of hydroxyl groups, which are hydrophilic and can facilitate water absorption, and (2) it increased the amount of unsaturated bonds and aromatic groups, which are known as stable groups that contribute to the stability of wood. Moreover, the changes in the content of chromophoric groups (e.g., carbonyl, alkene, and phenyl) and auxochromophoric groups (e.g., hydroxyl and carboxyl groups) likely explain the dark color of the treated wood.

Effect of thermal treatment on the degree of cellulose crystallinity of eucalyptus wood

Figures 3 and 4 show the effect of thermal treatment temperature and time on the XRD spectrum of wood. Compared with the untreated sample, the diffraction peak position of the treated samples shifted from 22.02° (2θ diffraction angle). Based on the Bragg equation (Flessa et al. 1987), the incident angle, θ , was reduced and the distance between crystal planes was increased when the incident wavelength of the X-ray was held constant. This observation could be explained by considering that the cellulose polymer chains swell when exposed to high temperature and humidity, thus resulting in increased distance between the different crystal planes. In addition, the reduced internal residual tensile stress could have played a role in increasing the distance between the crystal planes, as referenced in wood chemistry by Sjoström (1981). Apart from the migration of the diffraction peak position, the influence of thermal treatment on the diffraction peak intensity was also prominently observed.

For the control sample and the samples treated at 160°C, 180°C, 200°C, and 220°C for 4 hours, the diffraction peak intensity values were 933, 1,092, 768, 635, and 918, respectively. Therefore, the peak intensity values initially decreased from 1,092 to 635 and then increased to 918 when the thermal treatment temperature increased from 160°C to 220°C. The differences in peak intensities between the

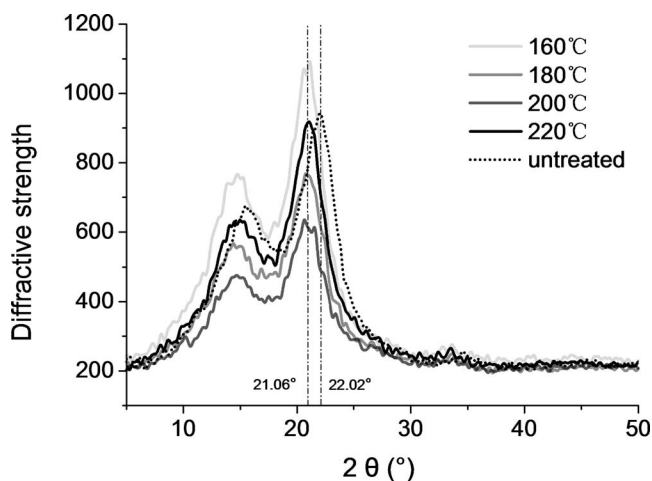


Figure 3.—X-ray diffraction of eucalyptus wood thermally treated at various temperatures for 4 hours.

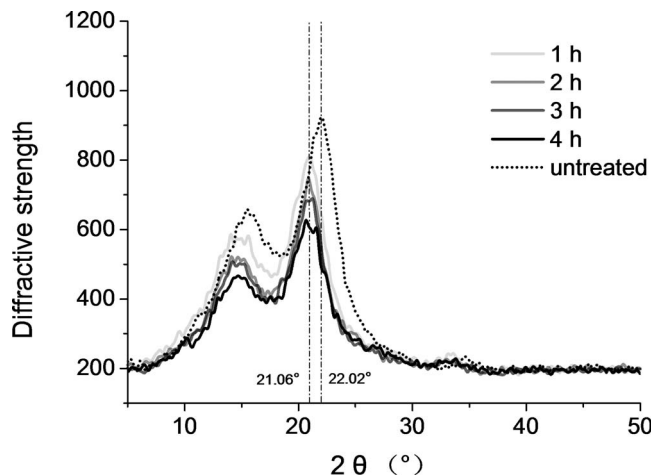


Figure 4.—X-ray diffraction of eucalyptus wood thermally treated for various amounts of time at 200°C.

untreated and the four treated samples were 159, –165, –298, and –15, respectively. From Figure 4, it can be seen that increased treatment time resulted in a reduced peak intensity of the treated wood samples. When compared with the untreated sample, the intensity of the peak from the treated wood samples decreased by 123, 186, 244, and 306 for thermal treatment at 200°C for times of 1, 2, 3, and 4 hours, respectively.

The degree of crystallinity of the cellulose from the thermally treated and untreated wood samples is shown in Figures 5 and 6. According to the Segal method, the relative degree of crystallinity for cellulose was calculated to be 46.78, 39.31, 37.46, and 45.09 percent when wood samples were treated at 160°C, 180°C, 200°C, and 220°C, respectively, for 4 hours. With this increase in treatment temperature, the degree of crystallinity first increased at 160°C, then decreased, and finally increased again at 220°C, which verified decreased mechanical strength resulting from thermal treatment (Cheng et al. 2015). While different results were found by Ding et al. (2012) showing a continuously increasing degree of crystallinity when

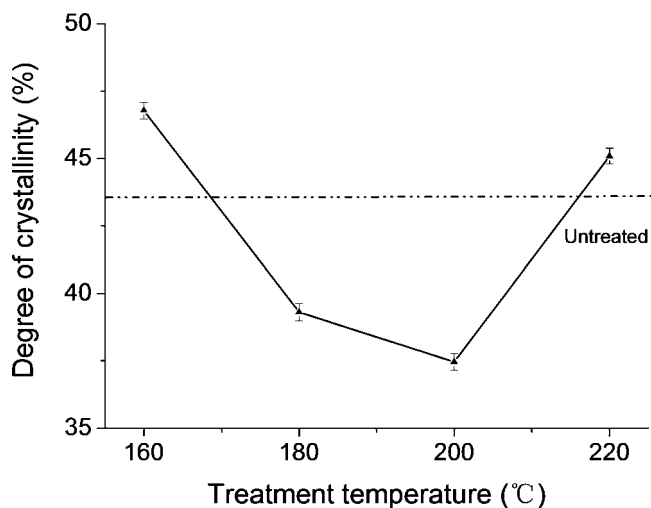


Figure 5.—Degree of crystallinity of samples at various treatment temperatures.

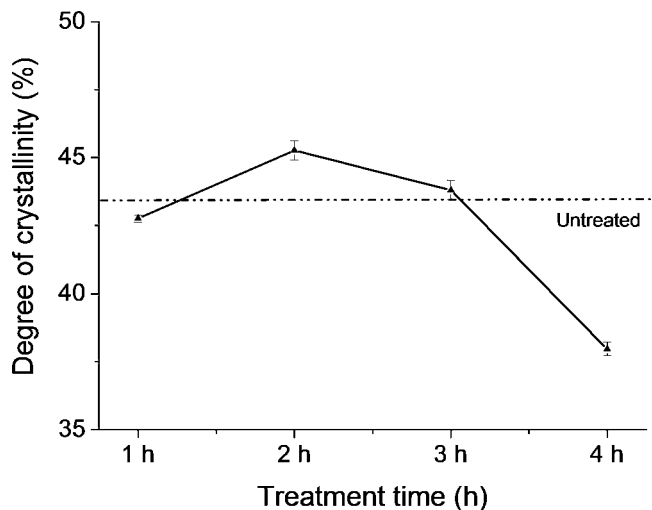


Figure 6.—Degree of crystallinity of samples at various treatment times.

temperature increased, this finding could be attributed to the different chemical composition and structure between the Chinese fir used in that study and the eucalyptus used in this study. At 220°C the degree of crystallinity was 42.76, 45.26, 43.80, and 37.97 percent for treatment times ranging from 1 to 4 hours. Analysis of variance revealed that both the treatment temperature and time significantly affected the degree of crystallinity and the significant level of treatment temperature with a confidence level of 99 percent.

When investigating the effect of thermal treatment conditions on degree of cellulose crystallinity, there are some factors that should be taken into consideration. In an environment with high temperature and high humidity, chemical polycondensation of free hydroxyls might occur within cellulose molecular chains in the amorphous region. Michiel and Tjeerdsma (2006) found that water molecules can be eliminated from the reaction, and ether bonds can be formed during the thermal treatment process. Their results indicated that the original internal structure of wood would be split and then reorganized. Moreover, the microfibrils would be arrayed in a more orderly fashion in the amorphous region of cellulose, and the above combined reactions resulted in the increase in the degree of crystallinity in the wood during thermal treatment.

Additionally, the acetic acid generated from the hydrolysis of ethanoyl within the polysaccharide molecular chains of hemicellulose results in an acidic atmosphere within the wood treated in a high-temperature and humidity environment. The newly generated acetic acid partly degrades the microfibril in the amorphous region of cellulose and even in the crystalline region, leading to a decreased degree of cellulose crystallinity. Apart from that, the pyrolysis and crystallization in the xylan and mannosan of the hemicelluloses may also play effective roles in the change in the degree of crystallinity (Akgül et al. 2007). To improve the effectiveness of the thermal treatment process and thereby improve the properties of thermal-treated wood, additional thermal experiments should be performed for suitable treatment conditions for various species of wood and for adopting precise

methods for tracking the thermal variation process with quantitative analysis.

Conclusions

1. Wood specimens were subjected to thermal treatment at different temperatures and amounts of time; the impact of thermal treatment on the functional groups and degree of crystallinity of eucalyptus wood was investigated. Thermal treatment significantly changed the chemical composition and structure of wood. The FTIR absorption peaks of hydroxyl group migrated from 3,425 cm^{-1} to lower wavenumbers at 3,250 cm^{-1} for all thermal-treated samples. With higher treatment temperatures and longer periods of time, a lower intensity of methyl was observed and almost disappeared at 220°C, while the aromatic ring first increased and then decreased. The carbonyl group maintained the same intensity with tiny fluctuations associated with different thermal treatment conditions.
2. Compared with the untreated wood sample, the X-ray diffraction peak position of the thermally treated eucalyptus wood migrated slightly to the left; the distance between the crystal plane might have increased. In general, the degree of crystallinity of cellulose increased after thermal treatment; however, the fluctuations in crystallinity we observed may be attributed to the different chemical composition and structure among various species of wood. The degree of cellulose crystallinity of eucalyptus wood was significantly affected by the treatment temperature with a confidence level of 99 percent.

Acknowledgments

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