

Changes of Chemical Composition, Crystallinity, and Fourier Transform Infrared Spectra of *Eucalypt pellita* Wood under Different Vacuum Heat Treatment Temperatures

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Abstract

Heat treatment is one effective method of improving dimensional stability and durability of wood. However, it usually decreases the bending strength of wood. Vacuum heat treatment, which is one way that does not use a thermal medium, had little influence on wood elasticity or bending strength in our previous study. But until now, few studies have reported on the changing mechanism of vacuum heat-treated wood. In this article, chemical composition and crystallinity of untreated and treated *Eucalypt pellita* wood were investigated under vacuum heat treatment temperatures from 80°C to 280°C for 4 hours. The results showed that lignin content increased and that holocellulose and α -cellulose decreased when the temperature was raised. Alcohol benzene extractive increased at the initial stage and then decreased when the temperature was above 240°C. Crystallinity of wood increased with a temperature increase from 80°C to 200°C and reached a maximum value of 52.57 percent. Fourier transform infrared spectroscopy analysis indicated that absorption intensity of a benzene skeleton at 1,600 cm^{-1} increased and that the hydrogen bond absorption band at 3,423 cm^{-1} shifted to a lower wave number as temperature increased.

Heat treatment is an effective method for wood modification, especially to improve the dimensional stability and durability of wood. By heat treatment, wood color can be darkened, making it look like a rare species (Hakkou et al. 2006, Esteves et al. 2008, Gu et al. 2010, Ding et al. 2011). Steam, hot oil, nitrogen, and molten metallic are often used as media for transferring heat and separating wood from oxygen in the air to prevent the weakening of wood (Rapp and Sailer 2000, Vernois 2001, Bruno et al. 2007); in vacuum heat treatment when no thermal medium is used, air is pumped out to maintain a vacuum in the treatment box during the process.

There are many reports on heat-treated wood properties. Although effective in improving the dimensional stability, durability, and color of wood, heat treatment has also shown some negative effects. Many studies have shown that mechanical properties of heat-treated wood decrease (Santos 2000, Unsal and Nadir 2004, Yildiz et al. 2006, Shi et al. 2007, Cao 2008, Wu and Huang 2012), while others have shown that bending strength decreases but that modulus of elasticity of wood is improved under certain treatment combinations (Li et al. 2009). Research on vacuum heat

treatment has shown that the bending strength and modulus of elasticity of wood can be enhanced when it is heat treated at about 200°C (Li et al. 2009, Liu et al. 2010, Wang et al. 2011). Such vacuum heat treatment might be one way to

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modify wood to improve its dimensional stability and physical-mechanical properties.

Changes in chemical composition and microstructure by thermal treatment have been regarded as a cause of dimensional stability and the mechanical properties of heat-treated wood (Hakkou et al. 2005). Studies on the changes in chemical composition and crystallinity when a heat medium is used have shown that cellulose and holocellulose decrease while lignin and benzene-alcohol extractive increase during the whole thermal procedure (Brito et al. 2008, Olov et al. 2011, Kévin et al. 2013); crystallinity changed as the treatment time was extended (Bhuiyan et al. 2000, Ding et al. 2012), and the changing trend of crystallinity increased at first, decreased, and increased again as the temperature rose (Sun and Li 2010). Compared with heat treatment with a thermal medium, few studies have addressed changes in chemical composition and crystallinity during vacuum heat treatment.

To find an optimal method for improving dimensional stability and also retaining the bending strength of wood, *Eucalypt pellita* wood was treated under vacuum conditions, showing that mechanical properties can be kept or even improved slightly by vacuum heat treatment under certain temperatures (Wang et al. 2014). The objective of this study was to explain how vacuum heat treatment affects wood by investigating the changes in chemical composition and crystallinity and the Fourier transform infrared (FTIR) spectra of heat-treated *E. pellita* wood at different temperatures.

Materials and Methods

Materials

Six-year-old *E. pellita* wood was obtained from the Eucalyptus Research Center in Guangdong Province, China. *E. pellita* wood samples were oven-dried below 80°C before vacuum heat treatment, and specimens measuring 330 by 40 by 40 mm (length by tangential by radial) were prepared from the oven-dried wood. Specimens were divided into two groups. One group was heat treated at 80°C, 120°C, 160°C, 200°C, 240°C, and 280°C for 4 hours (time for temperature increasing not included) under pressures of -0.08 to -0.09 MPa, and the other group was used as a control.

Vacuum heat treatment equipment (HJ-ZK60; Guangzhou Hengjun Company), with a minimum pressure of -0.09 MPa and a maximum temperature of 350°C, was used to treat wood. The equipment was composed of two parts: a processing box and a vacuum pump. During treatment, the vacuum condition was realized by a vacuum pump, and a high-temperature environment was realized by a heating cord around the processing box. There were three layers for the vacuum box, heating cords were located between the two layers of wall, and when the cords were heating, the wood in the vacuum box was also heating.

Wood powder was used to test the chemical composition, crystallinity, and FTIR spectra. First, samples were divided into two groups: one as control and the other prepared for vacuum heat treatment at 80°C, 120°C, 160°C, 200°C, 240°C, and 280°C, respectively, for 4 hours. Second, all of the wood, including both treated and untreated, was chopped into small strips and then smashed into powder in a grinder. Finally, wood powder was graded by sieves; powder between 40 and 60 mesh was used for chemical composition testing and that between 80 and 100 mesh was

used for crystallinity testing. Wood powder tested for FTIR spectra should be shaved from the wood by a sharp blade (the smaller the better). The moisture content of all the powder was dried to 0 percent.

Testing methods

Chemical composition.—Chemical composition tested in this experiment and its corresponding testing methods are as follows:

Lignin: Tested by Chinese Standard GB/T 2677.8-94 (Standardization Administration of the People's Republic of China [SAC] 1994b), which equals the American standard TAPPI T222 om-11.

Holocellulose: Tested by Chinese Standard GB/T 2677.10-1995 (SAC 1995) were as follows: (1) Weighed 2 g of powder and recorded the weight as m_0 . (2) Bagged the powder in filter paper and then extracted it with a benzene and alcohol solution; after extracting, the power bag was air-dried. (3) Removed the powder from the filter paper bag, put it in a 250-mL container, and then added 65 mL of distilled water, 0.5 mL of glacial acetic acid, and 0.6 g of sodium chlorite, in that order, and heated the mixture for 1 hour at 75°C. (4) Added 0.5 mL of glacial acetic acid and 0.6 g of sodium chlorite in the mixture and heated them for 1 hour at 75°C again. (5) Repeated Step 4 until the powder turned white. (6) Washed the residue with distilled water until the residue showed no acid, and then washed it with acetone three times. (7) Removed the solvent and dried the residue at 105°C to a constant weight (labeled as m_1). The content of the holocellulose was calculated by the following equation:

$$X_1 = \frac{m_1}{m_0} \quad (1)$$

where

X_1 = content of holocellulose in wood (%),

m_1 = weight of holocellulose after drying (g), and

m_0 = weight of wood powder sample under oven-dried condition (g).

α -Cellulose: Tested by the nitric acid-ethanol method according to the following steps: (1) Prepared 500 mL of the 68 percent nitric acid and absolute alcohol mixture with a ratio of 1:4. (2) Weighed 1 g of powder (labeled as m_0). (3) Put the powder in the prepared 25-mL mixed solution. (4) Heated the 68 percent nitric acid and absolute alcohol mixture in boiling water for 1 hour. (5) Removed the solvent and kept the residue. (6) Repeated Steps 3, 4, and 5 until the residue turned white. (7) Washed the whitened residue with the 68 percent nitric acid and absolute alcohol mixture, hot water, and absolute alcohol, in that order. (8) Dried the residue at 105°C until the moisture content was reduced to 0 and then weighed it (labeled as m_1). (9) Treated the residue at 500°C and then weighed it (labeled as m_2). The content of the α -cellulose was calculated by the following equation:

$$C = \frac{m_1 - m_2}{m_0} \times 100\% \quad (2)$$

where

C = content of α -cellulose in wood (%),

m_0 = weight of the wood powder sample (g),

m_1 = weight of residue after drying at 105°C (g), and

m_2 = weight of residue after treating at 500°C (g).

Benzene-alcohol extractive: Tested by Chinese Standard GB 2677.6-94 (SAC 1994a), which equals the American standard TAPPI T204-om97.

Crystallinity.—The crystallinity of wood was determined by an X-ray diffractometer (Philips X'Pert PRO) with a tube voltage of 40 kV and a current of 40 mA. Cu targets were used as the X-ray tube, a nickel layer was used to eliminate radiation from CuK α , a $\theta/2\theta$ link scan was used as a measuring method, and the emission slit was 0.76 mm.

Before testing, wood powder was pressed into a thin flat used for the 2θ scan. The scan range was from 5° to 50° , and the scan speed was 8° min^{-1} .

Crystallinity was calculated by the Segal formula as follows:

$$C_r I = \frac{I_{002} - I_{am}}{I_{002}} \times 100\% \quad (3)$$

where

$C_r I$ = relative crystalline degree, namely, crystallinity (%);

I_{002} = maximum intensity of (002) crystal lattice diffraction angle (arbitrary unit), appearing in scan curve when 2θ was near 22° ; and

I_{am} = scattering intensity of background diffraction in amorphous region when 2θ was near 18° and whose unit was the same with that of I_{002} .

FTIR spectroscopy

For these tests, KBr powder was mixed with the powder of wood samples using a weight ratio of 100:1. Then this mixture was pressed into a small disc with a diameter of 13 mm under a pressure of 75 kN cm^{-2} . The IR spectra of every sample were analyzed with a PerkinElmer Spectrum One FTIR spectrometer. Averaged curves from 32 repeated scans were obtained between wave numbers of 4,000 and 400 cm^{-1} . Precision of the spectrometer was 4 cm^{-1} .

Results and Discussion

Chemical composition

Thermal decomposition degrees of chemical components in wood depend on different treatment temperature. Relative content of each component showed different trends with an increase of temperature from 80°C to 280°C (Fig. 1). As shown in Figure 1, compared with untreated samples, content of holocellulose and α -cellulose of treated wood decreased gradually as temperature increased and dropped markedly above 200°C . Content of holocellulose and α -cellulose decreased from 74.25 and 45.43 percent, respectively, under the untreated condition to 49.36 and 12.97 percent, respectively, when treated at 280°C . Lignin content increased as temperature rose and changed from 34.1 percent at 200°C to 54.98 percent at 280°C . Alcohol benzene extractive increased as temperature rose; the maximum value was 4.54 percent at 240°C .

The changing trends of these several chemical compositions were consistent with many studies on heat-treated wood. Deng et al. (2009) treated *Cunninghamia lanceolata* thinning wood in hot air for 4 hours at 200°C , finding that holocellulose, cellulose, Klason lignin, and benzene-alcohol extractive changed from 63.83, 45.73, 32.12, and 3.18 percent to 55.12, 43.89, 39.96, and 3.37 percent, respec-

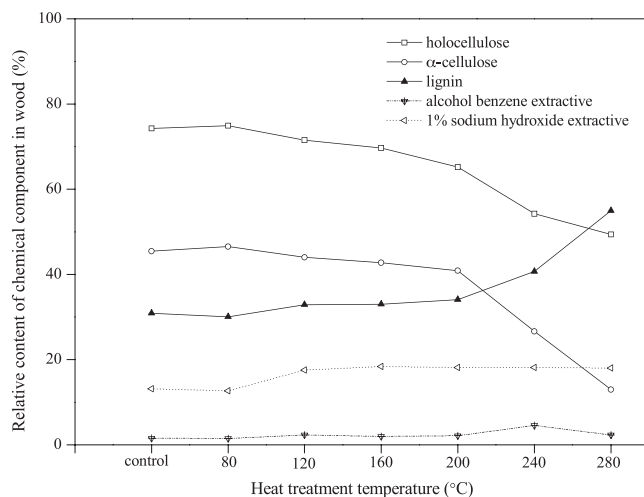


Figure 1.—Relative content of chemical components under different heat treatment temperatures.

tively. Among the three main chemical components of wood, hemicellulose is the first structural compound to be thermally affected, even at low temperatures. The degradation starts by deacetylation, and the released acetic acid acts as a depolymerization catalyst that further increases polysaccharide decomposition (Tjeerdma et al. 1998, Sivonen et al. 2002). Cellulose is less affected by heat treatment below 200°C , probably because of its crystalline nature (Bourgois and Guyonnet 1988), but as temperature increases, the structure of crystalline is destroyed, and the degree of polymerization is reduced at about 240°C (Yin 1996).

Lignin increased as treatment temperature rose, according to many researchers. Lignin content of *Pinus pinaster* increased from 28 percent to 41, 54, and 84 percent, respectively, at 260°C for 0.5, 1, and 4 hours (Bourgois and Guyonnet 1988); for Scots pine and birch treated between 205°C and 230°C during 4 and 8 hours, the lignin content increased from 24.5 to 38.7 percent and from 21.8 to 35.8 percent, respectively (Zaman et al. 2000). The increase of lignin content as temperature rises might be caused by the following reasons. First, the small molecule detached from hemicellulose or cellulose enclosed in the lignin was hard to detach because of the three-dimensional network structure of lignin. Second, as an amorphous polymer, lignin has a character of glass transition; when heated at about 200°C or above, lignin steps into the glass transition stage, becomes sticky, works as an adhesive, and glues the degradation product detached from hemicellulose or cellulose to lignin molecules, enhancing lignin content as temperature rises (Yin 1996). Finally, lignin determined using the standard methods in heat-treated wood samples is probably not a pure lignin. Several authors have suggested that polycondensation reactions with other cell wall components results in further cross-linking and contributes to the increase in apparent lignin content (Tjeerdma and Militz 2005, Boonstra and Tjeerdma 2006).

Crystallinity

Crystallinity of wood exerts a significant effect on the physical-mechanical properties of wood or fiber, such as tensile strength, modulus of elasticity, hygroscopicity,

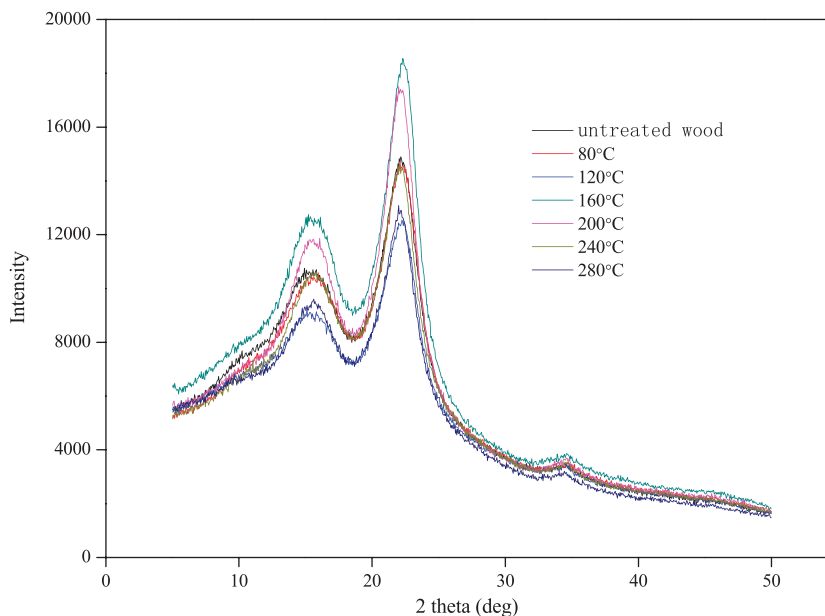


Figure 2.—X-ray diffraction spectra under different temperatures. (Color version is available online.)

moistening, and so on. Besides age and position, heat treatment also had an effect on the extent of crystallization. Changes of wood X-ray diffraction spectra treated under different vacuum heat treatment temperatures can be seen in Figure 2.

As shown in Figure 2, the X-ray diffraction intensity of wood treated at 160°C and 200°C was larger than the intensities at other temperatures. Crystallinity changes at different temperatures are shown in Figure 3. Crystallinity mainly increased as temperature increased from 80°C to 200°C, and then decreased once the temperature was above 200°C (Fig. 3). As the temperature rose from 240°C to 280°C, crystallinity increased again from 43.76 to 45.86 percent. The maximum crystallinity was 52.57 percent at 200°C, an improvement of 16 percent compared with the untreated samples. Crystallinity is an important factor affecting the physical, mechanical, and chemical properties of wood. The larger the crystallinity, the higher the tensile

strength, bending strength, and dimensional stability of wood (Yin 1996). In a previous study, when we heated *E. pellita* wood under vacuum, dimensional stability increased by about 30 percent, and modulus of elasticity increased by 25.2 percent at 200°C (Wang et al. 2014).

A similar changing tendency of crystallinity was also found in other studies; for example, the crystallinity of heat-treated *Larix* spp. first increased, then decreased, and finally increased, and the value of crystallinity was 50.77, 56.46, 43.59, and 64.15 percent when treated at 180°C, 200°C, 220°C, and 240°C, respectively, for 4 hours in air (Sun and Li 2010).

Cellulose is composed of two phase structures: a crystalline region and an amorphous region. The increase in crystallinity as temperature rises might be caused by two reasons. First, degradation of amorphous cellulose, as reported earlier, results in a decreased accessibility of hydroxyl groups to water molecules (Wikberg and Liisa

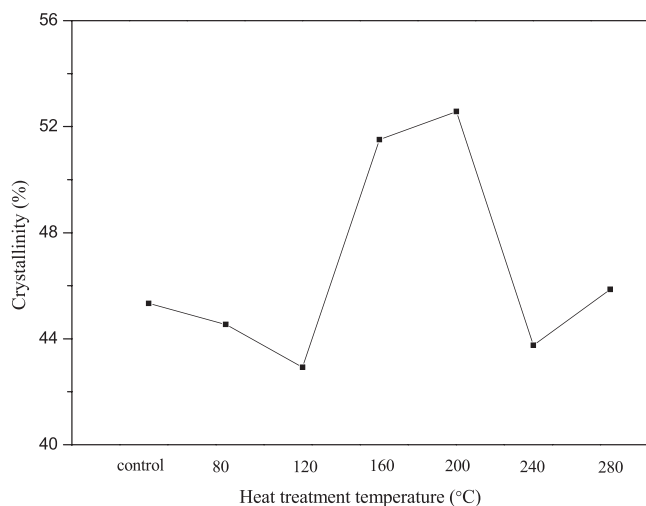


Figure 3.—Crystallinity changes under different vacuum heat treatment temperatures.

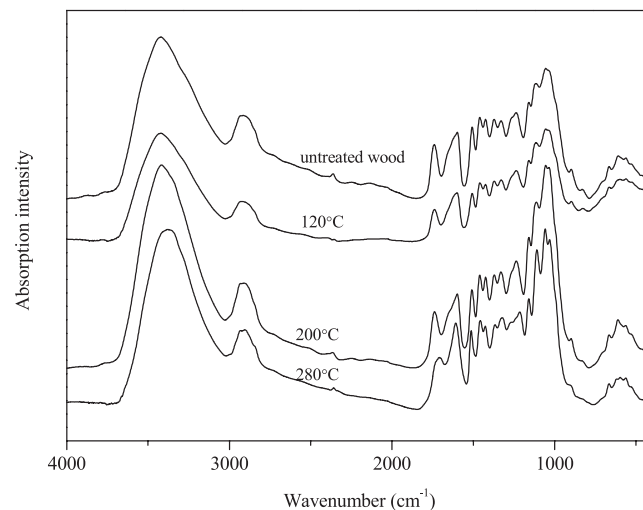


Figure 4.—Fourier transform infrared spectra of untreated and treated wood.

Table 1.—Absorption band in Fourier transform infrared spectra under different temperatures.

Main absorption peak (cm ⁻¹)				Assignments
Control	120°C	200°C	280°C	
3,423	3,419	3,416	3,379	O–H, N–H stretch
2,917	2,917	2,917	2,903	C–H stretch vibration in –CH ₃ , –CH ₂ –, R ₃ C–H, –CH=O
1,739	1,739	1,739	1,713	C=O stretch
1,595	1,597	1,597	1,608	Aromatic skeletal vibration
1,505	1,505	1,505	1,514	Aromatic skeletal vibration
1,463	1,455	1,455	1,455	C–H deformation vibration in lignin or –CH ₂ – of glycan; aromatic skeletal vibration in lignin
1,424	1,425	1,425	1,427	H–C–H scissoring in cellulose; H–C–H deformation vibration in lignin
1,372	1,372	1,372	1,368	C–H plane deformation vibration in cellulose or hemicellulose
1,329	1,329	1,330	1,321	O–H plane deformation vibration
1,235	1,235	1,235	1,215	–OCOR stretch vibration in –OCOCH ₃ of hemicellulose; benzene oxygen bond stretch vibration in lignin
1,159	1,159	1,159	1,158	C–O–C stretch vibration in cellulose and hemicellulose
1,118	1,112	1,115	1,109	OH bond association absorption band
1,054	1,055	1,055	1,057	C–O stretch vibration in cellulose and hemicellulose; alkyl bond stretch vibration in acetyl
1,029	1,034	1,029	1,032	C–O stretch in cellulose, hemicellulose, or lignin

2004, Bhuiyan and Nobuyuki 2005). Second, crystallization in quasi-crystalline amorphous regions is due to the rearrangement or reorientation of cellulose molecules inside these regions (Akgül et al. 2007). In crystalline regions, as the temperature increases, the segment motion of cellulose chain is enhanced, causing part of the space between the cellulose chain to decrease and a new hydrogen bond to form so that wood crystallinity is enhanced. In this study, when heated at over 240°C, crystallinity of wood decreased again; when the temperature increased to over 240°C, segment motion was so acute that the hydrogen bond was destroyed, the intermolecular force between the cellulose chain was reduced, wood crystallinity decreased, and in the macroscopic view, the mechanical property dropped.

FTIR analysis

Figure 4 shows FTIR spectra of untreated and heat-treated *E. pellita* wood at 120°C, 200°C, and 280°C. Assignments of main absorption are listed in Table 1.

Figure 4 and Table 1 show that absorption peaks of some bands shifted and that intensity changed in the FTIR spectra when heated. The absorption peak band of the benzene skeleton at 1,595 and 1,505 cm⁻¹ for the untreated samples shifted to 1,608 and 1,514 cm⁻¹ at 280°C. The stretching vibration peaks of O–H and N–H at 3,423 cm⁻¹, C–H at 2,917 cm⁻¹, C=O at 2,917 cm⁻¹, and –OCOR at 1,235 cm⁻¹ for untreated samples shifted to 3,379, 2,903, 1,713, and 1,215 cm⁻¹, respectively, when treated at 280°C.

The shift to lower wave numbers of C–H and C=O at 2,917 cm⁻¹ and –OCOR at 1,235 cm⁻¹ for heat treatment samples was probably caused by the intramolecular π - π conjugator effect (Li 2003). When wood was heated at high temperatures, new double or triple bonds formed because of an active group reaction under the thermal effect; if the new double or triple bonds interval linked with double bonds, such as acetyl groups on xylan or –OCOR groups by single bonds, then characteristics of double or triple bonds would decrease (Liu et al. 2000), reducing the absorption peak to a lower wave number.

Absorption intensity of C=O stretching vibration at 1,739 cm⁻¹ decreased, consistent with hemicellulose decomposition. When hemicellulose began to decompose under the thermal effect, the number of carboxyl and carbonyl groups, located mainly in hemicellulose (Yin 1996), and the

absorption peak, which was at 1,739 cm⁻¹ in the infrared spectra (Faix 1991), were reduced, further causing the absorption intensity of C=O stretching vibration at 1,739 cm⁻¹ to decrease.

The band absorption intensity at 1,595 and 1,505 cm⁻¹, assigned to benzene skeleton vibration (Faix 1991), increased as the heat treatment temperature rose, indicating that lignin content rose as temperature increased. Absorption peak position at 3,423 cm⁻¹ assigned to O–H stretching vibration (Faix 1991, Schwanninger et al. 2004) shifted to lower wave numbers, and the band broadened as temperature increased, probably caused by hydrogen bond association (Li 2003) under the thermal effect, reducing numbers of hydrophilic hydroxyl groups in wood and increasing crystallinity, hygroscopicity, and dimensional stability.

Conclusions

Vacuum heat treatment exerted obvious effects on the chemical composition of *E. pellita* wood, especially when the treatment temperature was above 200°C: lignin relative content increased, holocellulose and α -cellulose content decreased as temperature rose, and alcohol benzene extractive content increased at the initial stage and then decreased when the treatment temperature was over 240°C. The content of lignin, holocellulose, α -cellulose, and alcohol benzene extractive changed from 30.88, 74.25, 45.43, and 1.59 percent for untreated samples to 34.1, 65.17, 40.9, and 2.18 percent, respectively, at 200°C.

Changing trends of wood crystallinity generally increased at first and then decreased when the temperature was above 200°C. The maximum crystallinity value of 52.57 percent was realized at 200°C and improved by 16 percent compared with untreated samples. Absorption intensity of the benzene skeleton at 1,600 cm⁻¹ on the FTIR spectra increased as temperature rose, consistent with the phenomenon that lignin content increases as temperature rises. The hydrogen bond absorption band at 3,423 cm⁻¹ was shifted to a lower wave number, which might cause the wood crystallinity to increase as temperature increases.

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