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Color and surface chemistry changes of extracted wood flour after heating at 120 °C

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Abstract To investigate the effect of heat on color and surface chemistry of wood flour (WF), unextracted, extracted and delignified samples of commercial WF were heated at 120 °C for 24 h and analyzed by colorimetry, diffuse reflectance visible (DRV), attenuated total reflectance Fourier transform infrared (ATR-FTIR) and Fourier transform Raman (FT-Raman) spectroscopies. Unextracted samples showed a slight increase in CIEL*a*b* color coordinates, *a** and *b**. Compared with unextracted samples, color changes of the extracted samples varied with composition of the extraction solvents with generally smaller increases in *a** and larger decreases in *b** values. Delignified samples were marked by even larger increases in both *a** and *b** values. The color changes could be explained by analysis of the respective DRV, FTIR and FT-Raman spectra of the samples before and after heating. Heating of the extracted WF at 120 °C resulted in a red shift of the absorption at 430 nm and increase in absorption in the violet-blue spectral region of visible light. Delignified samples showed even more pronounced absorption in this spectral region after heating.

Introduction

Wood fiber is the most widely used lignocellulosic fiber for reinforcing plastics. The major chemical constituents of the wood cell wall contain hydroxyl and other

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oxygen-containing groups that attract moisture through hydrogen bonding (Peyer and Wolcott 2000). This hygroscopicity can cause problems both in composite fabrication and in the performance of the end product. When wood flour is used to extrude wood plastic composites (WPCs), it is usually subjected to heat to remove moisture. For this purpose, the flour is usually dried at 100–120°C. In addition to releasing moisture at these temperatures, the wood flour can also release some extractives (Tsoumis 1991). Loss of extractives can lead to both color and surface chemistry changes in the wood flour. Shafizadeh (1985) and LeVan (1989) reported that between 100 and 200 °C, wood releases water, carbon oxides and traces of organic degradation products. Degradation of the wood cell wall at elevated temperatures used in the process of manufacturing wood plastic composites can lead to undesirable properties, such as odor, discoloration and loss of mechanical strength (Nunez et al. 2002). Understanding the performance of WPCs requires knowledge of heat-induced changes in the surface chemistry and color of the wood fiber.

Heat treatment of wood flour can determine the final color of the WPC. The color of wood products has a significant influence on consumer preferences for cabinet and flooring woods (Fell 2002). The role of extractives on wood fiber color change during heat treatment is not well defined. The intrinsic color of wood fiber can be attributed to compounds that possess functional chromophoric groups such as phenoxy groups, double bonds and carbonyl groups that absorb UV-Vis light. Color change of wood as a result of exposure to heat can be related to the type and content of extractives. In thermally treated wood, phenolic extractives, such as stilbenes in radiata pine *(Pinus radiata)*, can contribute to color change (McDonald et al. 1997).

At 120–160°C, migration of phenolic extractives and wood resins to the wood surface was observed (Nuopponen et al. 2003). Degradation products from hemicelluloses and lignin resulting from thermal treatment, for example, can also contribute to color change. Increased extractive content is believed to be a result of hemicelluloses degradation during heat treatment (McGinnes and Rosen 1984). The presence of arabinose in extracts of kiln-dried radiata pine is believed to be the result of hemicelluloses degradation (Kreber et al. 1998). The liberation of acetic acid in thermally treated beech and Scots pine *(Pinus sylvestris)* is a result of degradation of hemicelluloses, which catalyzes further lignin degradation (Tjeerdsma et al. 1998). Low molecular weight compounds such as sugars and amino acids have been observed to redistribute toward the wood surface during thermal treatment, accumulating 0.5–1.5mm below the surface to produce discoloration. This phenomenon has been known for decades (Millet 1952; King et al. 1976; Theander et al. 1993). The redistribution has been investigated in Scots pine (Terziev 1995), and the discoloration during heat treatment has been investigated in radiata pine (Kreber et al. 1998). McDonald et al. (1997) have speculated that the discoloration is the result of the Amadori-Maillard reaction of fructose, sucrose or glucose with glutamic acid. In addition, oxidative and hydrolytic reactions are considered to be the main cause for production of chromophores during heat treatment of wood, where hydrolytic reactions generally are the dominant process when moisture is present (Fengel and Wegener 1989).

Because WF used in manufacturing WPCs is usually dried for 24 h at 120 °C to get rid of the moisture, it is essential to investigate the effect of such heat treatment on the surface chemistry and color of the wood fiber. The objective of the current study is to understand the relationship between surface chemistry and color changes of unextracted, extracted and delignified pine WF after heating at 120 °C.

Materials and methods

Wood flour samples

Pine WF was selected because it is the most common WF used in commercial WPC decking. The pine WF supplied by American Wood Fibers (AWF 4020; Schofield, Wisconsin, USA) was derived from scrap wood consisting primarily of ponderosa pine *(P. ponderosa)*. WF was sieved through a 40-mesh screen (0.425 mm) to remove the larger particles and through a 60-mesh screen (0.250 mm) to remove the fine particles. The WF was stored in a sealed polyethylene bag until required for further experimentation.

Wood flour extraction

The WF was extracted with various combinations of three different solvents: toluene/ethanol (TE), acetone/water (AW) and hot water (HW). Lignin was removed by sodium chlorite acidified with acetic acid (DL). TE solvent was prepared based on volume ratio (2:1) of toluene and ethanol. AW solvent was prepared based on volume ratio (9:1) of acetone and distilled water. In addition to extraction with single solvent system, TE, AW, HW or DL, samples of WF were also subjected to one or more extraction steps designated, TE-AW, TE-HW, TE-DL, TE-AW-HW, TE-AW-DL, TE-HW-DL, TE-AW-HW-DL, AW-HW, AW-DL, AW-HW-DL and HW-DL. For example, the designation TE-AW means the sample was subjected to two successive extraction steps, TE followed by AW, and TE-AW-HW-DL means that the sample was subjected to four successive extraction steps, TE followed by AW, HW and DL in that order. Each extraction step was performed for 24 h in a Soxhlet apparatus to ensure exhaustive removal of extractives. The delignification step (DL) was performed according to the method developed by Wise et al. (1946) with slight modifications.

Heat treatment of WF

Extracted and the unextracted WF samples were heated for 24 h at 120 °C in an Isotemp programmable forced-draft furnace (Fisher Scientific, Pittsburg, Pennsylvania, USA). After heating, the WF samples were pressed into pellets under a pressure of 10 MPa using a 30-mm-diameter die in a hydraulic press. The samples were stored in separate plastic bags in a desiccator until required for experimentation.

Characterization of WF

The surface chemistry of the wood fiber was characterized by ATR-FTIR and FT-Raman spectroscopy. ATR-FTIR was conducted on a Nicolet iZ10 module (Thermo Fisher Scientific, Fitchburg, Wisconsin, USA) using a Smart iTR Basic accessory. A single bounce, diamond crystal with 45° incident angle was used, and spectra were detected using a room temperature deuterated triglycine sulfate (DTGS) detector. Sixty-four scans at 4 cm^{-1} resolution were accumulated in the spectral region 4,000–600 cm^{-1}. The peaks were analyzed without smoothing the data. The spectra were baseline corrected and normalized on the band at 1,030 cm^{-1} as the internal standard. The peak at 1,030 cm^{-1} is attributed to C–O in cellulose and is stable to both solvent extraction and delignification.

FT-Raman spectroscopy was conducted on a Bruker MultiRam spectrometer (Bruker Instruments, Inc., Billerica, Massachusetts, USA). This Raman system is equipped with a 1,064 nm 1,000 mW continuous wave (CW) diode-pumped Nd: YAG laser. The laser power used for sample excitation was 300 mW, and 512 scans were accumulated in the spectral region 4,000–600 cm^{-1}. Bruker OPUS software program was used to find peak positions and carry out baseline corrections. The peak at 1,096 cm^{-1}, which is due mainly to stretching vibrations of C–C and C–O bonds in cellulose and hemicelluloses, was used as an internal standard (Agarwal and Ralph 1997). This peak is stable to solvent extraction or delignification. Five replicates were used for ATR-FTIR and FT-Raman spectroscopy.

Diffuse reflectance visible (DRV) spectroscopic analysis

Spectral measurements in the range 400–700nm were performed with a Miniscan EZ spectrophotometer (HunterLab, Reston, Virginia, USA), using cellulose filter paper (Whatman 42) as the standard. The reflectance spectra were converted into *K/S* spectra using the Kubelka–Munk equation:

$$K/S = \frac{(1 - R_{\infty})^2}{2R_{\infty}}$$

where R_{∞} (between 0 and 1) is the measured reflectance of the sample divided by the measured reflectance of the standard (∞ means the layer is so thick that further increase in thickness does not change the reflectance); and *K* and *S* are the absorption and scattering coefficients, respectively. The *K/S* spectra as a function of wavelength is used to identify the apparent absorption maxima. Typically, based on the Kubelka–Munk theory, it can be assumed that *K/S* is approximately linear to the variation of chromophores in this range of absorption values. Spectral changes due to heating at 120 °C were characterized by the difference *K/S* spectra, $\Delta K/S$.

Color measurement

Color was measured at three locations on each sample, and five replicates were used. A Minolta CR-400 Chroma Meter (Minolta Corporation, Ramsey, New

Jersey, USA) was used to measure color parameters with a C standard illuminant according to the CIELAB color system. CIELAB color system is a three-dimensional color space measuring the lightness *(L^*)* of the sample and color coordinates *(a^** and *b^*).* L^* represents reflectance of a sample that ranges between 0 for black and 100 for white. An L^* of 0 means the sample does not reflect light; an L^* of 100 means the sample reflects the entire incident light. An increase in L^* means the color has faded or has become lighter ($+\Delta L^*$ = lightening); and a decrease in L^* means the color has darkened ($-\Delta L^*$ = darkening). The color coordinates a^* and b^* do not have a specific range. a^* is defined as the red/green ($+a^*/-a^*$) coordinate; b^* is defined as the yellow/blue ($+b^*/-b^*$) coordinate (Papadakis et al. 2000; Segnini et al. 1999; Yam and Papadakis 2004). In this study ΔL^*, Δa^* and Δb^* were calculated according to Eqs. (2-4).

$$\Delta L^* = L^*_{heated} - L^*_{non-heated} \tag{2}$$

$$\Delta a^* = a^*_{heated} - a^*_{non-heated} \tag{3}$$

$$\Delta b^* = b^*_{heated} - b^*_{non-heated} \tag{4}$$

The corresponding total color change (ΔE^*) was calculated using Eq. (5).

$$\Delta E^* = \sqrt{\Delta L^{*2} + \Delta a^{*2} + \Delta b^{*2}} \tag{5}$$

Results and discussion

Changes in color parameters after heat treatment

Changes in color parameters, ΔL^*, Δa^*, Δb^* and ΔE^*, after heating at 120 °C are presented in Table 1. Positive values denote an increase and negative values denote a decrease in the color parameters.

Changes in L^* values

Samples that were subjected to solvent extraction only generally showed a larger increase ($+\Delta L^*$) in lightness compared with the control after heating. Exceptions were those samples that were subjected to AW or HW extraction. They, along with the delignified samples, showed a decrease ($-\Delta L^*$) in lightness after heating. Darkening was more pronounced for samples that had been subjected to solvent extraction followed by delignification. For these samples, it is reasonable to conclude that the darkening resulted mainly from dehydration reactions of hemicelluloses at 120 °C, which can form furfurals and their condensation products (Burtscher et al. 1987; Ellis and Paszner 1994; Kaar et al. 1991).

Changes in a^* values

The a^* values shifted toward red after heat treatment. This red shift was particularly more pronounced for delignified samples compared with those samples that had been subjected only to solvent extraction. Formation of red-colored structures on the

Table 1 Changes in color parameters of WF after heating at 120 °C

WF sample	ΔL^*	Δa^*	Δb^*	ΔE^*
Control (unextracted)	0.16[a] (±0.01)[b]	0.39 (±0.05)	0.62 (±0.04)	0.75 (±0.04)
TE	0.94 (±0.05)	0.02 (±0.04)	−0.33 (±0.04)	1.01 (±0.05)
AW	−0.28 (±0.33)	0.73 (±0.10)	−0.76 (±0.05)	1.09 (±0.04)
HW	−1.50 (±0.04)	0.63 (±0.04)	0.02 (±0.11)	1.63 (±0.01)
TE-AW	1.34 (±0.02)	0.22 (±0.05)	−1.11 (±0.10)	1.75 (±0.10)
TE-HW	0.71 (±0.07)	0.07 (±0.03)	−1.10 (±0.06)	1.31 (±0.07)
AW-HW	1.64 (±0.24)	0.03 (±0.09)	−0.18 (±0.12)	1.66 (±0.01)
TE-AW-HW	0.29 (±0.33)	0.08 (±0.07)	−0.34 (±0.16)	0.46 (±0.01)
DL	−1.10 (±0.06)	1.05 (±0.03)	3.99 (±0.18)	4.27 (±0.02)
TE-DL	−1.66 (±0.02)	1.34 (±0.01)	3.94 (±0.01)	4.48 (±0.01)
AW-DL	−2.59 (±0.02)	0.96 (±0.01)	4.82 (±0.11)	5.41 (±0.01)
HW-DL	−1.45 (±0.03)	1.18 (±0.01)	0.47 (±0.02)	1.93 (±0.02)
TE-AW-DL	−2.22 (±0.04)	1.78 (±0.03)	3.13 (±0.21)	4.23 (±0.02)
TE-HW-DL	−1.51 (±0.04)	0.81 (±0.01)	4.29 (±0.06)	4.62 (±0.06)
AW-HW-DL	−1.21 (±0.02)	0.13 (±0.00)	5.27 (±0.02)	5.56 (±0.02)
TE-AW-HW-DL	−2.33 (±0.03)	1.93 (±0.00)	0.73 (±0.06)	3.11 (±0.06)

[a] Average value of five replicates

[b] Numbers in parentheses represent the standard deviation of five replicates

wood fiber surface can result from air-oxidation of water-insoluble polyaromatic structures. Proanthocyanidins are one example of compounds that could form such colored structures by oxidation or polymerization at elevated temperatures (Luostarinen and Möttönen 2004a, b).

Changes in b values*

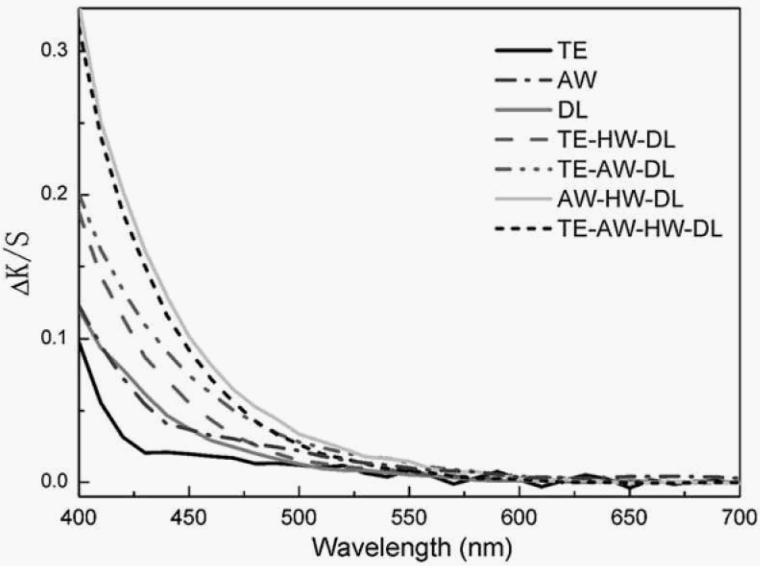
The delignified samples generally showed a strong shift toward yellow *(+Δb*)*. By comparison, samples that were subjected to solvent extractions only showed moderate-to-strong shifts toward blue *(−Δb*)*.Samples subjected to extraction with TE-AW and TE-HW showed particularly stronger shifts toward blue. The yellow color faded presumably because of loss of extractives by volatilization or thermal degradation.

Changes in total color, ΔE^*

Heat treatment resulted in little color change for the unextracted control and solvent-extracted samples (i.e., small ΔE^* values). However, heat treatment of delignified samples resulted in more pronounced color change. It is well known that changes in wood color upon heating can be an indication of chemical changes in wood (Burtin et al. 2000; Bekhta and Niemz 2003; Sundqvist 2004). During heat treatment, wood components are degraded or modified through reactions such as dehydration, hydrolysis, oxidation, decarboxylation and transglycosylation (Kocaefe et al. 2008). Although it is mostly extractives that contribute to the heat-induced changes in wood color by formation of various types of conjugated double bonds, carbonyl functionalities and quinoid structures, both lignin and hemicelluloses can also play an important role in the formation of color substances during heat treatment. Thus, it is reasonable to expect considerable heat-induced changes in the surface chemistry and color of the WF, because in addition to the major components hemicelluloses and lignin, such color changes can also originate from residual extractives.

Diffuse reflectance visible (DRV) spectroscopy

It is clear that heating at 120 °C resulted in two types of spectral responses, with low-absorption and high-absorption characteristics in the range 400–700 nm. The high-absorption spectra (Fig. 1) showed relatively higher absorption in the range 400-450 nm compared with the control. The control (Fig. 2) showed a peak at 420 nm and shoulders at 440, 465 and 490 nm upon heating. The increased absorption in these at these wavelengths resulted in yellow color of the sample. Absorption bands were also observed at 530–550, 640 and 670 nm, which resulted in red color of the sample. It is reasonable to conclude that the increased absorption was due to the formation of components with conjugated double-bond structures, including quinoid structures. This would cause the color of WF to shift toward yellow and red. HW-extracted samples showed relatively higher broadband absorption centered at 430 nm, with a shoulder at 490 nm. These absorption bands are far too broad to represent a single chromophore. Hence, they can be ascribed to



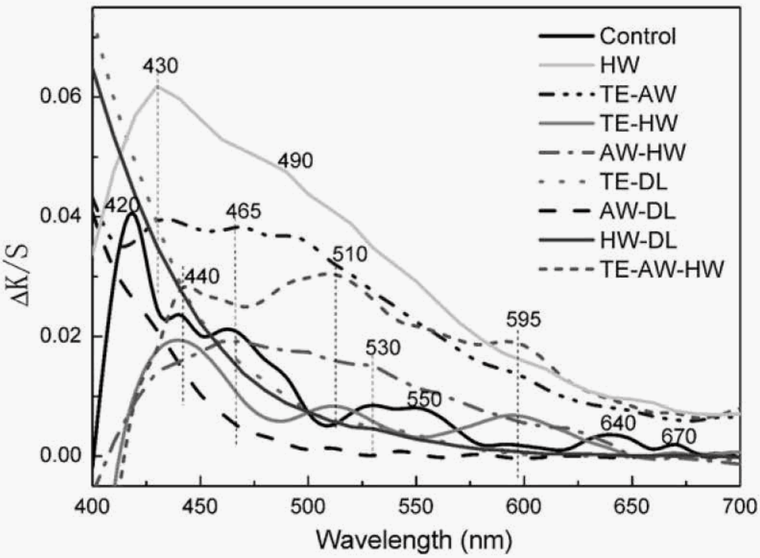


Fig. 2 Low-absorption $\Delta K/S$ spectra of WF samples heated at 120 °C

the extensive types of conjugated double bonds, carbonyl functionalities and quinoid structures (Hiltunen et al. 2006) formed from the heat-induced breakdown products of lignin and residual extractives. The high-absorption intensity in the spectral region of 400-600 nm for samples that had been subjected to HW extraction can be attributed to the oxidation of water-soluble extractives that may have migrated and accumulated on the surface of the wood fiber, resulting in darkening $(-\Delta L^{*})$ and higher ΔE^{*} value compared to control samples. Heat treatment of TE-AW-extracted WF also resulted in a red shift to 430 nm with shoulders around 465 and 480 nm. The red shift appeared at 440 nm for TE-HW- and TE-AW-HW-extracted WF and higher wavelength at 465 nm for AW-HW-extracted WF. TE-AW-extracted WF absorbed more in the region of 450-550 nm, with an absorption maximum around 470-480 nm. This is consistent with the higher Δa^{*} values observed for TE-AW-extracted WF. Broad shoulder peaks at 510 and 595 nm were observed for samples that had been subjected to extraction with TE-HW and TE-AW-HW. This suggests the formation of substances that absorb in the violet-green spectral region of visible light, which resulted in increase in Δa^{*} value. The increased absorption peak of solvent-extracted WF at higher wavelength was presumed to be the result of breakdown products of lignin and some hemicelluloses. Samples that had been subjected to extraction followed by delignification showed higher absorption at short wavelengths, 400-430 nm, whereas no absorption peak was observed for these samples at longer wavelengths. The absorption in the region 500–700nm was particularly more pronounced for samples that had been subjected to solvent extraction only, and thus suggesting heat degradation of lignin-derived products to form red substances.

ATR-Fourier transform infrared spectroscopy

ATR-FTIR spectroscopy was used to study the effect of heat treatment on the surface chemistry of the extracted or delignified samples (Fig. 3) and of samples that were subjected to multiple extraction steps (Fig. 4).

In the case of unextracted control, it can be seen that heat treatment did not result in any noticeable change in the intensity of the peaks at 1,505, 1,455, 1,421 and 1,330 cm^{-1}. The samples with single or multiple extractions also presented the same trends. The peaks at 1,505, 1,455 and 1,421 cm^{-1}, which correspond to the aromatic skeletal vibrations in lignin, almost disappeared with delignification. The peak at 1330 cm^{-1}, which was assigned to ring breathing with C-O stretching, is stable upon heating for all the extracted or delignified samples. More pronounced changes were observed in the region of 1,800–1,550 cm^{-1} and 1,300–1,200 cm^{-1}. The peak at 1,733 cm^{-1}, which was assigned to non-conjugated C=O stretching vibration (Rosu et al. 2010), and the peak at 1,265 cm^{-1}, which is attributed to absorption by C-O stretching in acetyl groups in hemicelluloses, increased considerably after heat treatment. The phenomenon was also observed for AW- or HW-extracted WF and also those samples that were subjected to multiple-step extraction. This suggests that a considerable amount of carboxylic groups was produced. A possible explanation for this is thermal dehydration of hemicelluloses to form furfurals and their condensation products (Norton 1947; Harris et al. 1960). The increase in the intensity of the peak at 1,733 cm^{-1} was consistent with the

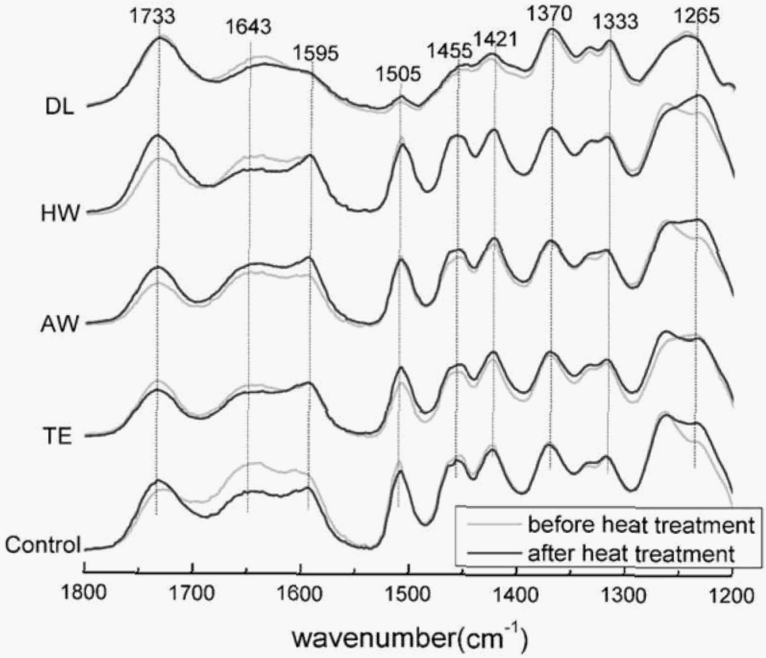


Fig. 3 ATR-FTIR spectra of control WF and single-step extracted WF before and after heat treatment at 120°C

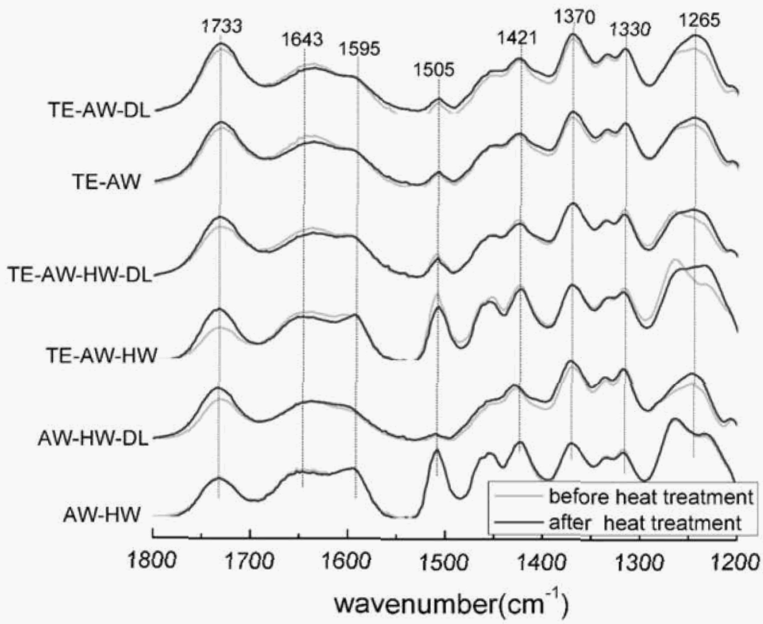


Fig. 4 ATR-FTIR spectra of multiple-step extracted WF before and after heat treatment at 120 °C

positive changes in Δa^*. However, in the case of AW-HW-extracted WF and the delignified samples, both of the peaks increased slightly or did not change. Another possible mechanism would be the formation of muconic acid type structures upon lignin oxidation during the heat treatment. On the contrary, TE-extracted WF showed a decrease in both of the peaks. It can be speculated that TE- or AW-HW-insoluble extractives, which contained non-conjugated C=O groups, were lost by volatilization or thermal degradation during heat treatment. The decrease in the surface concentration of these groups appeared to be associated with the smallest changes of Δa^* and Δb^*color coordinates after heating at 120 °C.

It can be observed from the control that the conjugated C=O groups and C=C unsaturated linkages at 1,643 cm^{-1} decreased significantly after heating. Samples that were subjected to one-step extractions also showed a decrease in this peak with the exception of AW-extracted WF. For samples that had been subjected to multiple-step extractions, this peak did not show any measurable change after heating. It is possible that the decrease in intensity of 1,643 cm^{-1} could be attributed to thermal rearrangement reactions of lignin molecules. Conjugated C=O and ethylenic C=C in coniferyl alcohol unit corresponding to the peak at 1,643 cm^{-1} are located in the a-position of side chains of aromatic rings in lignin. This position is a reactive site and readily reacts with other phenyl nuclei above 120 °C to form diphenylmethane type structures (Funaoka et al. 1990). However, the peak at 1,643 cm^{-1} increased for the AW-extracted WF after heating. This is consistent with the greater Δa^* values of AW-extracted WF after heat treatment. The increase in conjugated C=O groups and red color presumably is due to oxidative coupling of compounds related to tannins, which can be readily extracted by acetone/water and oxidized into insoluble polyphenols and redeposited on the WF surface. Hydrolysis and oxidative transformation of polyphenols to dark color polymers can be considered as a possible contributor to form quinoids structures and color changes of the WF.

Fourier transform Raman spectroscopy

Figure 5 shows the Raman intensity at 1,660 and 1,600 cm^{-1} for samples that were either extracted or delignified before and after heat treatment. The peaks at 1,660 and 1,600 cm^{-1} are the most informative scattering characteristics of lignin in Raman spectra and the most informative ones for explaining the heat-induced color changes of wood flour. The peak at 1,660 cm^{-1} is assigned to contributions of ethylenic C=C bond in coniferyl alcohol units and to the γ-C=O in coniferaldehyde units in lignin (Yamauchi et al. 2005). Aromatic ring stretch modes of lignin are detected at 1,600 cm^{-1}. The intensity of these two peaks increased after heating the unextracted control at 120 °C. This is indicative of formation of structures containing C=C and C=O groups such as quinone or substituted aromatic acetophenone type moieties on the wood fiber (Agarwal and Atalla 2000). However, there was no apparent change in the intensity of these two peaks when the

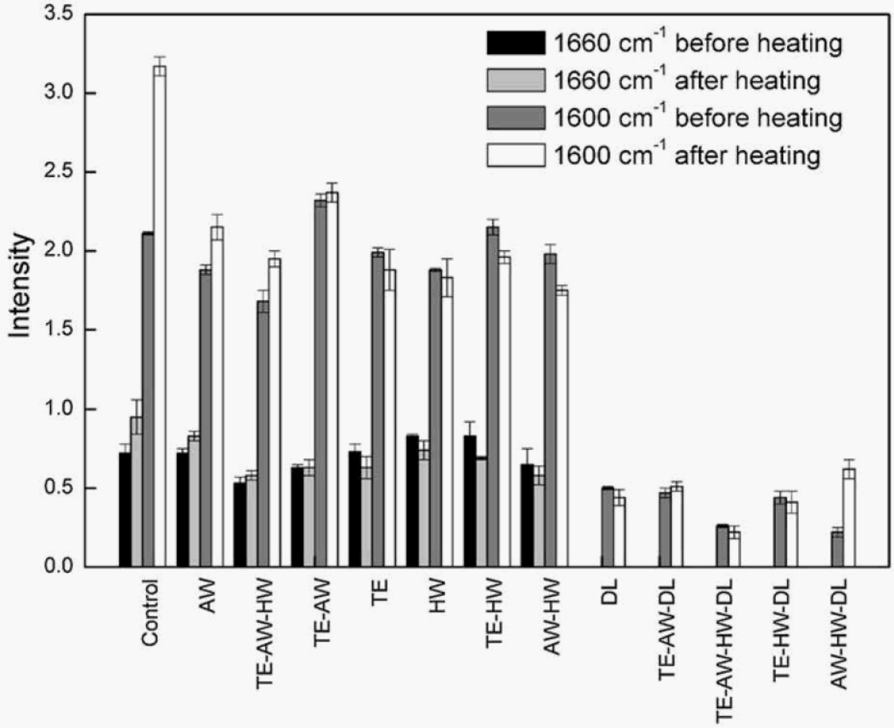


Fig. 5 Raman intensity at 1,660 cm^{-1} and 1,600 cm^{-1} before and after heat treatment at 120 °C

extracted or delignified samples were subjected to heat treatment. In the case of TE-extracted WF, the Raman intensity of both peaks at 1,600 and 1,660 cm^{-1} decreased after heat treatment, while the AW-extracted WF increased after heat treatment. In the case of HW-extracted WF, the peak at 1,660 cm^{-1} decreased, while the peak at 1,600 cm^{-1} increased slightly after heating. Heat treatment of TE-extracted WF resulted in the least amount of C=C or C=O groups compared with AW- or HW-extracted WF. This appears to be consistent with little or no change in the color parameters a^* and b^* for the TE-extracted WF (See Table 1). For the delignified sample, the peak at 1,660 cm^{-1} was not detectable, and there was no significant change in the peak at 1,600 cm^{-1} after heat treatment.

In the case of the samples treated with multistep extraction after heating, it can be seen that no apparent change in the intensity of these two peaks was observed. However, the intensity of the peak at 1,600 cm^{-1} increased when TE-AW-HW- or AW-HW-DL-extracted WF was subjected to heating. In the case of TE-HW or AW-HW extraction, the intensity at 1,660 and 1,600 cm^{-1} decreased. This is consistent with the lower absorption of visible light and as a result of yellow color fading when TE-AW- or AW-HW-extracted WF was subjected to heating.

Conclusion

Compared with unextracted control, samples of solvent-extracted WF showed an increase in total color change, ΔE^*, after heating at 120 °C. The exception to this trend was WF that had been subjected to multistep extraction with TE followed by AW, followed by HW. This sample was characterized by a relatively small increase in lightness $(+\Delta L^*)$, accompanied by a small increase in the red/green coordinate $(+\Delta a^*)$ and a relatively small decrease in the yellow/blue coordinate $(-\Delta b^*)$. Samples that had been subjected to a delignification step alone or in combination with solvent extraction steps showed large increases in total color change, ΔE^*, characterized by large decreases in lightness $(-\Delta L^*)$ and large increases in both the red/green $(+\Delta a^*)$ and yellow/blue $(+\Delta b^*)$ coordinates. These color changes appear to be consistent with the stronger absorption at 400-500 nm observed for these samples after heating. Raman and infrared spectra of these samples suggest that these color changes may be related to the wood fiber surface distribution of conjugated and non-conjugated carboxyl groups and C=C linkages.

Samples that were subjected to the multistep extraction procedure, AW-HW-DL, showed the greatest total color change after heating. This is consistent with the higher absorption in short wavelength of visible light due possibly to increased surface carboxylic groups after heating. The least total color change after heating was shown by WF samples that had been subjected to the multiple solvent extraction procedure, TE-AW-HW. Thus, to minimize color change after heating at 120 °C, the wood flour should be subjected to the multistep extraction procedure, TE-AW-HW. However, if, on the other hand, large color changes are desired, then the WF should be subjected to the multi-step extraction procedure, AW-HW-DL.

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