Effects of Thermal Treatment on the Dimensional Stability and Chemical Constituents of New and Aged Camphorwood

Guang Rui Sun,[a,b,c] Yi Ran He,[d] and Zhi Hui Wu [b,c,*]

Green and naturally aged camphorwood (*Cinnamomum camphora*) samples were thermally treated at 120, 150, or 180 °C for 2.5 h to investigate how various thermal treatments affected dimensional stability and hygroscopity. As a result, mass loss showed an increasing trend after treatment from 0.88% to 2.44%, whereas equilibrium moisture content exhibited a decreasing trend (in the range of 10.88% to 7.96%) after treatment at 180 °C. Additionally, thermal treatment improved the dimensional stability of camphorwood samples. Fourier-transform infrared spectra for wood after thermal treatment revealed that treatment led to hemicellulose deacetylation.

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*Contact information: a: College of Art Design, Tianjin University of Science and Technology, Str. Dagu South Road No. 1038, Tianjin, China; b: Co-Innovation Center of Efficient Processing and Utilization of Forest Resources, Str. Longpan No. 159, Nanjing, China; c: College of Furnishing and Industrial Design, Nanjing Forestry University, Str. Longpan No. 159, Nanjing, China; Nanjing Polytechnic University, Str. Zhongshan East Road, No. 532-2; *Corresponding author: wzh550@njfu.edu.cn*

INTRODUCTION

Wood is an important renewable lignocellulosic natural resource with the features of low-processing energy, great strength/weight ratio, and resistance to natural aging and environmental changes in temperature and humidity, and environmental friendliness (Wu *et al.* 2016; Liu *et al.* 2019a,b, 2021a,d). Given these properties, wood has been extensively utilized in applications such as interior decoration, furniture, and architecture (Huang *et al.* 2021; Hu *et al.* 2021; Hu and Chen 2021; Hu and Zhang 2022). However, as a natural material, wood contains cellulose, hemicellulose, lignin (Liu *et al*. 2021b, 2021c), and other extracts that are anisotropic. With changes in temperature or humidity, wood will adjust its equilibrium moisture content (EMC) and shrink or swell, resulting in defects, including deformation, cracks, or decay, shortening wood product value or service life, thereby restricting its use (Liu *et al*. 2019c; Yang 2021; Yin and Liu 2021).

Many modification processes have been developed to improve wood's dimensional stability, including high-temperature thermal treatment, acetylation or using impregnation resin (Esteves *et al.* 2007a,b, 2008; Candan *et al.* 2013; Acosta *et al.* 2021). Of these, the first process has been a modification approach with environmental friendliness, which contributes to degrading lignin and hemicellulose. Though the thermal treatment could decrease the strength of wood (Hill 2006), such decrease couldn't affect the application of wood, also, the thermal treatment could reduce hydrophilic groups within wood (Bhuiyan and Hirai 2000; Bekhta and Niemz 2003; Boonstra *et al.* 2007; Gašparík *et al.* 2019; He

et al. 2019a,b). It also elevates the cellulose crystallinity and crystallization zone, thereby increasing dimensional stability while decreasing wood hygroscopicity (Esteves *et al.* 2008; Esteves and Pereira 2009; Gunduz *et al.* 2009). As a result of natural aging, old wood subjected to long-term use has higher dimensional stability; thus, old wood recycled from old buildings or large furniture items is often used for the construction of furniture and wooden crafts.

Cinnamomum camphora has long been planted along the southern Yangtze River in China, in Taiwan, Korea, southern Japan, Vietnam, and India; meanwhile, it has been introduced to numerous additional countries. Camphorwood is a popular furniture material, widely used in boxes, wardrobes, and as wedding furniture because of its good anti-degradation and insect-proof properties, while there has been a lack of studies on the dimensional stability of camphorwood. In this study, old used and new camphorwood samples were heat-treated at different temperatures in the range of 120 to 180 °C and then dimensional stability and hygroscopicity were measured. In addition, the dimensional stability was compared for used and new camphorwood samples, and the chemical and physical structures of treated and untreated camphorwood were analyzed.

EXPERIMENTAL

Materials

Green wood sample was cut from a camphorwood tree with 45 cm diameter at breast height. The initial moisture content of the sample was 65 ± 3%, and the content after drying was 12 ± 2%. The samples were measured according to the standard GB/T 1931 (2009). A sample of old camphorwood was taken from a large chest (made in the 1940s) obtained from a Chinese classic furniture collector in Tianjing City, China. The initial moisture content was 12 ± 2% (measured in line with standard GB/T 1931 (2009). Test samples of dimensions 20 × 20 × 20 ((length × width × thickness) mm^3 were prepared from old and green wood samples using sapwood. Each test sample was dried at 103 °C prior to thermal treatment.

Methods

Thermal treatment

In this study, thermal treatments were conducted for 2.5 h in an oven under diverse temperatures (120, 150, and 180 °C). Meanwhile, 40 untreated samples (20 green wood samples and 20 old wood samples, marked as 20 °C) were used as controls to compare properties with heat-treated samples.

Mass percentage loss

Mass percentage loss (MPL) could be measured using mass change values prior to and succeeding thermal treatments using Eq. 1,

$$MPL = \frac{m_0 - m_t}{m_0} \times 100\% \quad (1)$$

where m_0 (g) stands for sample weight after oven drying before treatment, and m_t (g) stands for sample weight following treatment.

Wood swelling coefficient (WSC) and moisture absorption (MA)

The WSC and MA values are two important indicators of wood dimensional stability, with the swelling coefficient specifically important for measuring swelling in the tangential direction. Swelling tests performed in this study followed the standard GB/T 1931 (2009). In brief, both untreated and treated samples were subject to oven-drying, followed by preservation within at 20 °C and 65% humidity conditions for reaching EMC. The authors determined sample weight and sample dimension prior to and following treatments. The WSC and MA values were calculated *via* Eqs. 2 and 3, respectively,

$$a = \frac{l_w - l_0}{l_0} \times 100\% \quad (2)$$

$$\mathrm{MA} = \frac{w_a - w_b}{w_b} \times 100\ \% \quad (3)$$

where a stands for swelling coefficient (tangential), l_0 indicates original sample dimension (mm), and l_w stands for post-treatment dimension (mm). In Eq. 3, w_b (w_a) stands for pre (post)-treatment sample weight (g) within the chamber.

Statistical analysis

Treatments were compared by subjecting mass percentage loss, wood swelling coefficient, and moisture absorption data to variance analysis using SPSS (IBM Corp., IBM SPSS Statistics for Windows, v. 25, Armonk, NY, USA), with the probability level being set at 0.05. This study did not detect experimental run through treatment interaction and combined data from diverse runs in subsequent analyses. Levene's test was conducted to analyze homogeneity of variance, whereas the Shapiro-Wilk test was applied in determining normality. Fisher's Protected least significant difference (LSD) test was employed to separate treatment averages at $\alpha = 0.05$.

Morphological characteristics

For investigating the possible changes of physical architectures of untreated and treated wood samples, this work adopted the environmental scanning electron microscope (SEM) (Quanta 200, FEI Company, Eindhoven, Netherlands) to observe the surface shapes of wood specimens by measuring electrical conductivity. Wood samples were prepared by applying a sputter gold coating (2 nm) using Gold Palladium SEM Annular Sputtering by adopting the target 2" ID × 3" OD × 0.1 mm Anatech (SC502-314; Quorum Technologies, Ltd., Watford, UK). The bombarding voltage used for SEM was 20.0 kV.

Chemical structure analysis

Fourier-transform infrared (FTIR) spectra were obtained for both treated and untreated ground wood samples under direct transmittance with the standard FTIR spectrometer (Tensor 27, Bruker, Ettlingen, Germany) within the 700 to 4000 cm^{-1} range at a 4.0 cm^{-1} resolution for altogether 32 scans. After aligning light equipment, the authors collected background spectra before measurement. The spectra presented are averages of six measurements for each treatment.

RESULTS AND DISCUSSION

Mass Percentage Loss

The MPL is the main metric to follow the quality of thermally treated wood, and it explains the degree of pyrolysis of small molecular components such as hemicellulose (Qu *et al.* 2019; Mania *et al.* 2020). The MPL values depend on heating medium, wood varieties, treatment duration, and temperature (Esteves and Pereira 2009). Table 1 reports the oven-dry weight and MPL of green and old wood samples. Compared with the weight of the green camphorwood samples, the old camphorwood samples were of lighter weight, consistent with the effects of decomposition due to the approximately 70 years of natural aging. As a result, mass loss showed an increasing trend as temperature was elevated. Notably, mass losses of green and old camphorwood samples were 2.438% and 2.095%, respectively, at 180 °C, which was greater than the losses at 120 and 140 °C treatments. The mass loss was significantly different between green and old camphorwood samples (p value < 0.001), indicating fewer easily degradable components in old wood. With increasing treatment temperature, the MPL values increased significantly, but the interaction factor between wood type and treatment temperature was not significant, with a p value of only 0.030.

Table 1. Oven-Dry Weight before Treatment and MPL of the Thermally Treated Wood

Wood Type	Oven-dry Weight Before Treatment	WPL After Treatment (%) at 2.5 h		
		120 °C	150 °C	180 °C
Green Camphorwood	4.179 (0.138)	1.49 (0.32)	2.13 (0.77)	2.44 (0.81)
Old Camphorwood	4.064 (0.103)	0.88 (0.25)	1.56 (0.49)	2.10 (0.60)

Mean values are presented and figures within parentheses stand for standard deviation (n = 20)

Wood Dimensional Stability (WDS)

The WDS significantly affects wood product use and quality. Wood with low dimensional stability is prone to the effects of ambient temperature and humidity, which can result in defects such as swelling, cracking, bending, and deformation. The tangential swelling coefficient plays a more important role than those in the radial and cross directions (Qiu *et al.* 2016). Therefore, in this study only tangential swelling coefficient was investigated. When wood moisture content is lower than fiber saturation point (FSP), the cell wall can absorb water, leading to expansion. Thus, the moisture content has a great influence on the size of wood. For better investigating how thermal treatment affects WDS, MA values were measured. The results presented in Table 2 show how thermal treatments affected WDS. Both untreated and treated wood swelled and absorbed moisture due to the presence of hydrophilic components. Due to natural aging effects, the swelling coefficient and MA values were 1.53% and 8.78% for the old wood sample, respectively, values that are significantly lower (p < 0.001) than those of green wood (1.88% and 9.60%, respectively). Treatment temperature exhibited a significant impact on the WDS values (p < 0.001). With increased treatment temperature, the swelling coefficient and MA gradually decreased. The swelling coefficients were 2.12% for the control group, 1.77% for the 120 °C treatment group, 1.59% for the 150 °C treatment group, and 1.34% for the 180 °C treatment group. The MA values were 10.88% for the control group, 9.41% for the 120 °C

treatment group, 8.51% for the 150 °C treatment group, and 7.96% for the 180 °C treatment group. However, the interaction factor between wood type and treatment temperature against swelling coefficient (p value = 0.196) and MA (p value = 0.351) is not significant.

Table 2. Role of Thermal Treatments in WDS

Wood Type	Swelling Coefficient (%)	Moisture Absorption (%)
Green Camphorwood	1.88(0.036)[a]	9.60(0.192)[a]
Old Camphorwood	1.53(0.032)[b]	8.78(0.171)[b]
Thermal Treatment		
Control	2.12(0.039)[a]	10.88(0.220)[a]
120 °C, 2.5 h	1.77(0.034)[b]	9.41(0.187)[b]
150 °C, 2.5 h	1.59(0.031)[b]	8.51(0.171)[c]
180 °C, 2.5 h	1.34(0.026)[c]	7.96(0.159)[c]
	p Values	
Wood Type	< 0.0001	< 0.0001
Thermal Treatment	< 0.0001	< 0.0001
Wood Type × Thermal Treatment	0.196	0.351

Note: p value indicates the significance of different influencing factors. The smaller the p value means the stronger the significance. It is generally considered that when the p value is less than 0.05, the influencing factors are significant, and when the p value is greater than 0.05, it is not significant. “Wood Type × Thermal treatment” is the influence of the interaction between wood type and thermal treatment.

As shown above, the changes in both the swelling coefficient and MA values indicated that all tested thermal treatments improved the dimensional stability of wood. Additionally, the data show an obvious correlation between swelling coefficient and MA. A simultaneous analysis of swelling coefficient and MA is presented in Fig. 1.

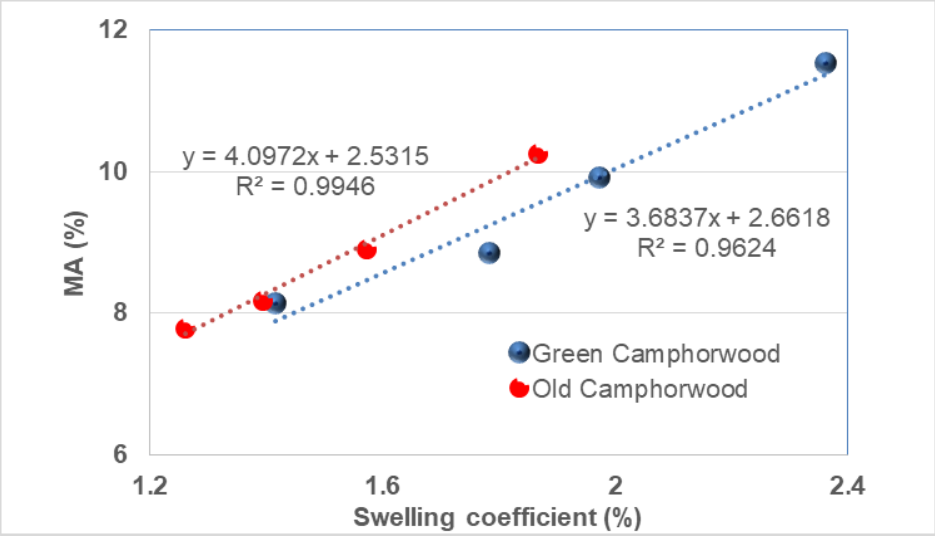


Fig. 1. Correlations between swelling coefficient and MA

The MA values of green and old camphorwood samples were plotted against the swelling coefficients, as shown in Fig. 1. There was a strong, statistically significant dependency of MA on the swelling coefficient. The linear correlation further supports the model that at WMC < FSP, which means that wood cell walls may absorb water resulting in swelling. Alternatively, it can desorb water and contract. These changes result in a change in wood dimension. Changes in the swelling coefficient and MA values of untreated and treated green and old camphorwood are due to changes in the content of hydrophilic components.

Chemical Structure Analysis Using FTIR Spectroscopy

Improvements to wood mass loss and dimensional stability resulting from thermal treatment are caused by various and specific chemical transformations occurring in the main wood components and extracts. FTIR spectroscopy is widely and effectively used to measure those changes of sample chemical architecture because of diverse treatment conditions (Chen *et al.* 2011; Basso *et al.* 2017; He *et al.* 2017). Comparison of the FTIR spectra reveals a noticeable decrease of IR-band intensities at approximately 3340 cm^{-1} that correspond to −OH stretching. Thermal treatment reduces the relative number of hydroxyl groups (He *et al.* 2019), thus improving the dimensional stability of the wood. Figure 2 displays FTIR spectra of treated and control samples within 800 to 1800 cm^{-1} for 2.5 h treatment at 120, 150, or 180 °C.

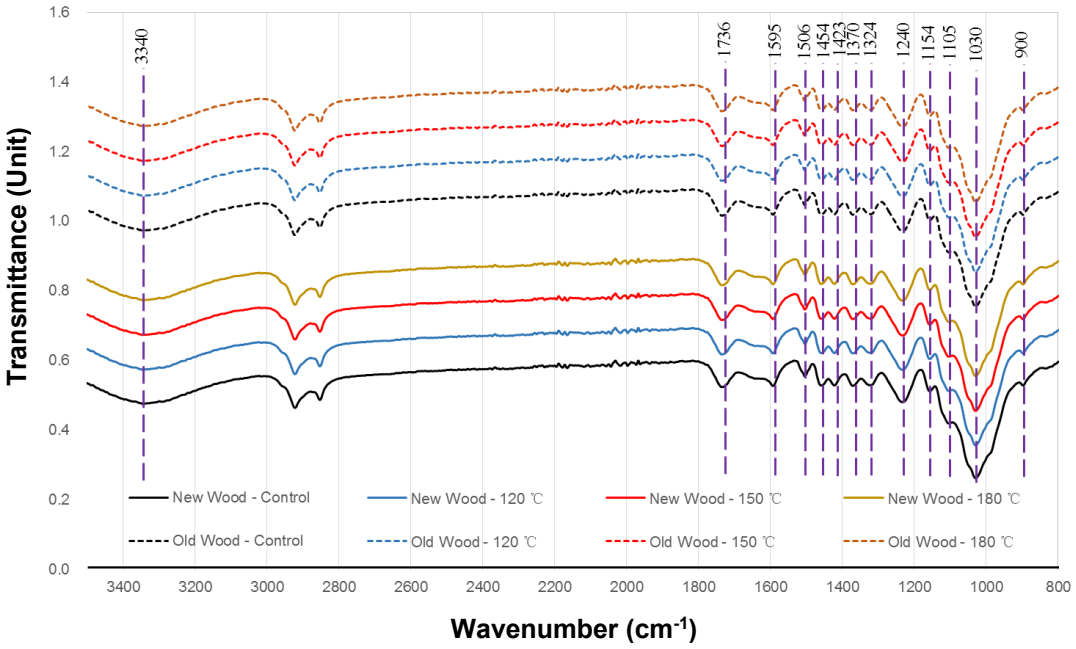


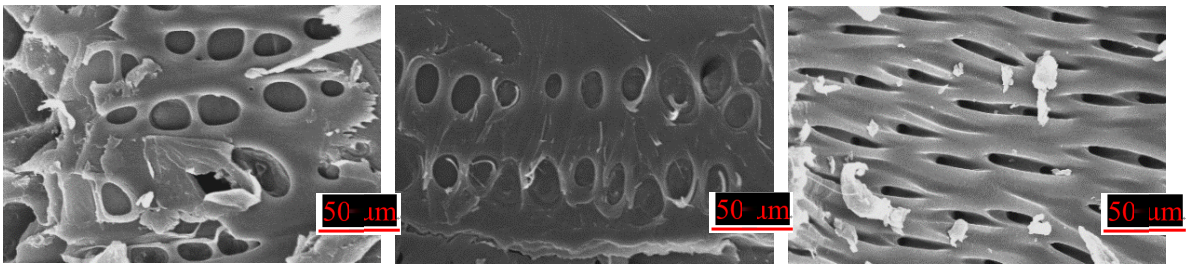
Fig. 2. FTIR spectra for treated and control samples

Comparison of the FTIR spectra between treated and control samples within 800 to 1800 cm^{-1} revealed a decrease in the intensity of the band at 1736 cm^{-1}, associated with acetyl carbonyl stretching within hemicellulose (Chien *et al.* 2018). This observation indicates that thermal treatment induces hemicellulose deacetylation by cleaving acetyl groups (Altgen *et al*. 2018). Additionally, two aromatic skeletal stretching peaks were detected at 1506 and 1593 cm^{-1}, respectively, for lignin. The peak intensities of these bands showed an increasing trend as temperature elevated, though these changes were minimal. The increase in intensities of these bands can be associated with the elevated lignin faction

because of decomposition of polysaccharides, especially hemicellulose. Furthermore, the peak at 1105 cm^{-1} was associated with CO stretching, which was more obvious in sample after 180 °C thermal treatment. It is possibly associated with ether bond generation based on hydroxyl groups from lignin and hemicellulose (Colom *et al.* 2003), or novel ester or alcohol formation during thermal treatment (Kocaefe *et al.* 2008).

Morphology

To evaluate potential changes in physical structures resulting from the heat treatments, a SEM micrograph was used to image the different wood samples, as shown in Fig. 3. Compared with old wood, smoother cell walls are evident for the green wood sample, and many wrinkles appeared on the cell wall of the old wood sample, and the pits of the wood were partially broken after 180 °C heat treatment, all the phenomenon might for the reason that under the initially moist and hot conditions, the lignin and hemicellulose within the wood cell wall were plasticized, and the capillary forces and pressure inner wood also could break the wood tissue.



a) New wood control **b)** Old wood control **c)** Green wood 180 °C treated

Fig. 3. SEM micrographs of control and treated samples at 1000X magnification

CONCLUSIONS

1. Compared with green camphorwood, old camphorwood was found to have a lower density and better dimensional stability due to long-term natural aging. Aging causes changes in the hydrophilic chemical composition and degradation of other chemical components.
2. Both green and old camphorwood samples experienced mass loss and improved dimensional stability as a result of thermal treatment at different temperatures. With increased treatment temperature, the wood dimensional stability (WDS) values gradually improved.
3. Differences in Fourier transform infrared (FTIR) spectroscopy indicated that chemical changes occurred during thermal treatment, likely the deacetylation of hemicellulose.
4. A comparison of the morphological characteristics of untreated and treated wood samples revealed that some cell wall components are degraded during heat treatment, which improves the dimensional stability. Thermal treatment could be useful for preparing various wood products that can be used in landscape architecture, furniture, and building.

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