Effect of a Combination of Moderate-Temperature Heat Treatment and Subsequent Wax Impregnation on Wood Hygroscopicity, Dimensional Stability, and Mechanical Properties

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Received: 2 August 2020; Accepted: 20 August 2020; Published: 23 August 2020

Abstract: Wood is an environmentally friendly material, but some natural properties limit its wide application. To study the effect of a combination of heat treatment (HT) and wax impregnation (WI) on wood hygroscopicity, dimensional stability, and mechanical properties, samples of Pterocarpus macrocarpus Kurz wood were subjected to HT at a moderate temperature of 120 °C and a high temperature of 180 °C, for a 4 h duration. Subsequently, half of the 120 °C HT samples were treated with WI at 90 °C. The results showed that 180 °C HT and WI decreased the capacity of adsorption and liquid water uptake and swelled the wood significantly, while WI had the biggest reduction. The effect of 120 °C HT was significant only on decreasing the capacity of adsorption and the swelling of liquid water uptake. The bending strength (MOR) of wood decreased only after 180 °C HT, and 120 °C/4h HT and WI had no significant influence on MOR. The bending stiffness (MOE) increased significantly after 180 °C HT and WI, while 120 °C/4h HT had no significant influence on MOE. Therefore, the combination of moderate-temperature HT can act synergistically in the improvement of certain aspects of wood properties such as capacity of water adsorption and liquid water uptake. WI effectively improved wood hygroscopicity, dimensional stability, and mechanical properties.

Keywords: heat treatment; wax impregnation; hygroscopicity; dimensional stability; mechanical properties

1. Introduction

Wood is widely used in buildings and wood products due to its special properties such as high strength-to-weight ratio, environmental sustainability, low production energy, and renewability. However, there are also undesirable properties such as poor durability and low dimensional stability which limit its utilization and reduce its service life and value [1,2]. These disadvantages of wood are all associated with the water present in wood cells [3,4]. In order to overcome wood’s natural shortcomings, modifications are performed to improve its comprehensive quality [5].

The hygroscopicity is the capacity of wood to react to the moisture content of the air by absorbing or releasing water vapor. The hygroscopicity of wood is mainly attributed to the hemicelluloses, which are amorphous and readily hydrolyzed by hydroxyl groups [6,7]. The durability, dimensional stability, and hydrophobicity of wood therefore can be improved by reducing wood hygroscopicity.
The reduction of hygroscopicity can be obtained by modifications, such as chemical modification [8–11], heat treatment (HT) [12–15], surface modification [16], and impregnation [17]. Chemical modification can obtain good results in dimensional stability and hydrophobicity; however, it can be harmful to people and the environment [17]. Above all, HT is an eco-friendly and effective approach to increase durability and dimensional stability [18,19] and to reduce wood wettability [20]. However, its disadvantages are obvious in effects such as color change, lowered of mechanical strength, and higher energy consumption [12,21].

Waxes are environmentally friendly water repellents that are helpful for wood protection [22]. Wood is usually treated through wax impregnation (WI) and WI combined with other treatments [23,24]. The rate of hygroscopicity and water uptake may be reduced after WI [25,26]. In addition, WI may increase the mechanical strength of wood [27,28] and maintain wood color and texture [29].

HT and WI are both environmentally friendly wood modification methods. In combination, the two may complement each other’s strengths and improve the durability, dimensional stability, hydrophobicity, and mechanical strength of wood, in addition to reducing energy consumption. Wood was impregnated using wax and subsequently subjected to HT as the general procedure of this combination in previous studies. Although studies [30,31] were carried out using the opposite procedure of HT first, subsequently followed by WI, wood was subjected to high-temperature HT, which may affect mechanical properties and wood color and improve energy consumption.

In this study, *Pterocarpus macrocarpus* Kurz wood was treated by HT at moderate and high temperatures, and half of the moderate HT samples were subsequently subjected to WI at a low temperature under atmospheric conditions. The object of this study was to systematically investigate the effect of moderate-temperature HT and low-temperature WI on hygroscopicity, dimensional stability, and mechanical properties of wood. The results of this study represent a contribution to the academic understanding of wood HT combined with WI treatment.

2. Materials and Methods

2.1. Raw Materials

Air-dried *P. macrocarpus* Kurz boards measuring 1000L mm × 100T mm × 25R mm were collected from Degoo Furniture Co., Ltd., Xianyou, China. They were all heartwood boards with an average moisture content (MC) of 10 ± 1% (GB/T 1931–2009 national standard) [32]. The dimensions of the test specimens were 300L mm × 20T mm × 20R mm and 20L mm × 20T mm × 20R mm. Each dimension contained 28 specimens that were free of knots and other defects. Commercial microcrystalline wax was used for the impregnation test with a melting point of about 60 °C, molecular weight of 500–800 g, refractive index of 1.435–1.445, kinematic viscosity (99 °C) of 9.2–25.0 mm²/s, and density of 0.80–0.92 g/mL.

2.2. Heat Treatment and Wax Impregnation

All test specimens (300L mm and 20L mm) were first oven-dried at 103 ± 2 °C to constant mass and then were randomly divided into two HT groups and one control group. For 120 °C HT, there were 14 specimens for each dimension; for 180 °C HT and the control group, there were 7 specimens for each dimension. HT under vacuum pressure has some advantages, as reported in a previous study [33]. In this study, HT was therefore subsequently applied at two temperature levels (120 and 180 °C) under 13.4 kPa in a vacuum heating chamber (HJ-ZK60, Dongguan Hengjun Instrument Equipment Co., Ltd., Dongguan, China). As shown in Table 1, the HT processes were composed of preheating, HT, and cooling phases. After cooling, half of the 120 °C HT specimens were fully impregnated in a steel tank using liquefied wax at 90 °C until the weight became constant after 48 h. After impregnation, the remaining wax was wiped and the specimens were put into sealed bags and cooled at a constant temperature of 30 °C for 1 h.
| Treatment | Process   | Temperature (°C) | Time (h) | Pressure (kPa) |
|-----------|-----------|------------------|----------|----------------|
| HT        | Preheating| 100              | 1        | 13.4           |
|           | HT        | 120/180          | 4        | 13.4           |
|           | Cooling   | 100              | 1        | 13.4           |
| WI        | Impregnation| 90              | 48       | 101.3          |
|           | Cooling   | 30               | 1        | 101.3          |

2.3. Mass Loss and Weight Percentage Gain

The specimens of 20L mm × 20T mm × 20R mm were used for the measurement of mass loss (ML) owing to HT and weight percentage gain (WPG) due to WI. They were calculated using Equations (1) and (2), and the value is the average of seven replicates. The mass was measured using an electronic balance (JA21002, Shanghai Liangping Instrument and Meter Co., Ltd., Shanghai, China; 1200 g/1 mg).

\[
ML = 100\% \times \frac{(M_o - M_h)}{M_o}
\]

(1)

\[
WPG = 100\% \times \frac{(M_w - M_h)}{M_h}
\]

(2)

where \(M_o\) is the oven-dried mass of specimens before HT, \(M_h\) is the oven-dried mass of specimens after HT, and \(M_w\) is the mass of specimens after WI.

2.4. Moisture Adsorption and Liquid Water Uptake

A total of 28 specimens measuring 20L mm × 20T mm × 20R mm were used for moisture adsorption and liquid water uptake tests. For the measurement of 120 °C HT, 120 °C HT + WI, 180 °C HT, and control, each group contained seven replicates. The specimens were first oven-dried at 103 ± 2 °C until constant mass and then conditioned in a climate chamber at 20 °C and 65% RH to reach the equilibrium moisture content (EMC) according to GB/T 1931-2009 national standard. After the moisture adsorption test, the same specimens were oven-dried at 103 ± 2 °C to constant, weighed again, and then were immersed in distilled water for liquid water uptake tests to constant weight. The results of moisture adsorption and liquid water uptake are presented as moisture content which was calculated using Equation (3), which is used to assess the capacity of wood adsorption and liquid water uptake.

\[
MC_e = 100\% \times \frac{(M_e - M_o)}{M_o}
\]

(3)

where \(MC_e\) is the moisture content of the specimens after conditioning or water uptake tests, \(M_e\) represents the mass of the specimens after conditioning or water uptake, and \(M_o\) denotes the mass of the oven-dried specimens.

2.5. Dimensional Stability Measuring

The dimensional stability was estimated based on swelling tests according to GB/T 1931-2009 national standard. The swelling data of specimens of 20L mm × 20T mm × 20R mm was measured using a digital caliper (CD-20CPX, Mltutoyo, Japan, 0–200 mm/0.01 mm) during the moisture adsorption and liquid water uptake test. For the measurement of 120 °C HT, 120 °C HT + WI, 180 °C HT, and control, each group contained seven replicates. The dimensions and weights of the treated and control groups were measured before and after tests. The swelling was calculated using Equation (4):

\[
S = 100\% \times \frac{(L_w - L_o)}{L_o}
\]

(4)

where \(S\) is the swelling in tangential or radial directions, \(L_w\) is dimension after moisture adsorption and liquid water uptake, and \(L_o\) represents the dimension in oven-dry state.
2.6. Mechanical Property Testing Bending Strength and Modulus of Elasticity

Mechanical properties of treated and controlled wood were determined according to GB/T 1936.1-2009 national standard [34]. Seven replicates of each group were tested for bending strength or modulus of rupture (MOR) and modulus of elasticity (MOE) using a 3-point bending test machine (Shimadzu, Japan). Prior to the test, all specimens of 300L mm × 20T mm × 20R mm were conditioned to a constant weight in a climate chamber at 20 °C and 65% RH. The average value of the seven replicates was used for comparison.

2.7. Statistical Analysis

Data were analyzed using analysis of variance (ANOVA) by SPSS to assess the effects of various treatments on the hygroscopicity, dimensional stability, and mechanical properties of treated wood. The differences between mean values of each treatment level were further separated using Duncan’s multiple range test at \( p < 0.05 \).

3. Results and Discussion

3.1. Mass Loss, Weight Percentage Gain, and Moisture Content of Conditioned Wood

The \( ML \) of specimens after heat treatment, along with the WPG of the 120 °C HT group after wax impregnation, are shown in Table 2. The \( ML \) was slight after moderate-temperature HT and became severe when temperature rose to 180 °C. The \( ML \) of 180 °C HT group was 2.7 times that of 120 °C HT group, which indicates that the \( ML \) was influenced significantly by temperature. After 48 h wax impregnation, the weight of the 120 °C HT group increased by 8.82%, which demonstrates that wax was impregnated into wood successfully.

| Treatment  | \( ML \) (%) | WPG (%) | \( MC_e \) (%) |
|------------|-------------|---------|----------------|
|            |             |         | Adsorption     | Liquid Water Uptake |
| Control    | -           | -       | 7.91 (0.31)    | 56.19 (3.87)         |
| 120/4HT    | 0.96 (0.27) |         | 6.77 (0.40)    | 52.77 (3.52)         |
| 180/4HT    | 2.62 (0.44) | 8.82 (1.01)| 5.52 (0.53)    | 48.55 (3.58)         |
| 120/4HT + WI| 8.82 (1.01)|         | 2.82 (0.34)    | 43.95 (3.34)         |

3.2. Moisture Adsorption and Liquid Water Uptake

To present the effects of HT and WI on the hygroscopicity of wood, the \( MC_e \) of specimens after long-term water vapor sorption at 20 °C and 65% RH was determined, which demonstrates the EMC of wood. Meanwhile, the \( MC_e \) of specimens after long-term water immersion was also determined. The \( MC_e \)'s of the control and treated groups are shown in Figure 1 and Table 2. Figure 1a indicates that the \( MC_e \) of samples after long-term exposure decreased significantly after HT and HT + WI. Compared with the control group, \( MC_e \) of samples after 120 °C/4h HT, 180 °C/4h HT, and 120 °C/4h HT + WI decreased by 14.3%, 30.2%, and 64.1%, respectively. HT decreased the \( MC_e \), and the reductions in \( MC_e \) were greater for the more severe heat treatment, which is similar to previous reports [35]. However, the 120 °C/4h HT + WI samples exhibited the lowest \( MC_e \), which decreased by almost half as compared to the samples after 180 °C/4h HT. This indicates that wax impregnation could further decrease the moisture adsorption capacity. In contrast to the control group, the contribution ratios of 120 °C/4h HT and WI to the reduction of \( MC_e \) were 14.3% and 49.8%, respectively. The effect on moisture adsorption capacity reduction by wax impregnation was 3.5 times that of 120 °C/4h HT. This shows that WI played a significant role in further decreasing the moisture adsorption capacity of wood. The reduction of \( MC_e \) in the current work was clearly larger than that of a previous report [19]. The main reason is quite likely due to the impregnation of a large quantity of wax into the wood.
After water immersion, the moisture content of the treated group showed a similar tendency. MC,e decreased significantly, except for in the 120 °C/4h HT group. Compared to the control group, the MC,e of 180 °C/4h HT and 120 °C/4h HT + W decreased by 13.6% and 21.8%, respectively. Although the MC,e after 120 °C/4h HT decreased slightly, the 120 °C/4h HT + W group showed remarkably lower moisture content than that of the control group and the 120 °C/4h HT group (Figure 1b). These findings suggest that wax impregnation further decreased the capacity of liquid water uptake of wood. The results may be attributed to the impregnation of wax. On the one hand, wax solution firstly fills the cell lumens fully or partly, which results in a reduced space in the impregnated wood structure for water uptake. On the other hand, wax impregnates into wood and forms wax coating attached to wood cell walls, which blocks the migration path of the free water due to the hydrophobic property of wax [30].

**Figure 1.** Moisture content of the control and treated wood: adsorption (a) and liquid water uptake (b). Bars with different letters present significant difference (p < 0.05) in accordance with Duncan’s multiple range tests. Note: There are no significant differences between groups containing the same letters. The bar with ab means that there are no significant differences between this group and groups labeled with a or b.

### 3.3. Effect of Heat Treatment and Wax Impregnation on Swelling of Wood

The swelling of tangential and radial dimensions after adsorption and liquid water uptake are shown in Figure 2. After water vapor sorption and immersion, tangential and radial swelling of the treated groups was less than that of the control group and presented a similar tendency. The adsorption swelling (Figure 2a) of the treated group presented a significant difference (p < 0.05) compared to the control group, except for the 120 °C/4h HT group. These results suggest that moderate-temperature HT has little improvement on adsorption dimensional stability of wood, however, high temperature or WI improves wood adsorption dimensional stability greatly. The adsorption swelling of wood after WI was the smallest in all treated groups, indicating that this group has the best dimensional stability. This shows that the effect of WI on dimensional stability improvement was greater than the effect of 180 °C/4h HT and indicates that WI is a suitable treatment for dimensional stability modification. Wood has obvious anisotropy in swelling properties, and tangential swelling is generally twice as great as radial swelling. The ratio of adsorption swelling between tangential (T) and radial (R) directions presents a decreasing trend after 180 °C/4h HT and 120 °C/4h HT + WI. The T and R swelling became almost the same for the wood after 120 °C/4h HT + WI. These results indicate that WI extremely impacts the swelling properties, resulting in an improved dimensional stability.

In contrast to the adsorption swelling, all water uptake (immersion) swelling of treated groups decreased significantly (p < 0.05) compared with the control group. The immersion swelling of the 120 °C/4h HT + WI group was the lowest in all treated groups. This suggests that both HT and WI improved wood immersion dimensional stability. Comparing the swelling of 120 °C/4h HT and 120 °C/4h HT + WI in Figure 2b, the swelling was greatly reduced after WI, showing that WI has a significant effect on dimensional stability. The reduction in swelling after WI was closely associated with the long hydrophobic chains of the wax and the bulking effect of wax [35]. The water uptake
capacity of wood was weakened both after HT and WI treatment, resulting in an improved deformation resistance property. However, the ratios of immersion swelling between T and R directions did not change after HT and WI treatment, indicating that the effect on reduction of immersion swelling was the same in both tangential and radial directions.

**Figure 2.** Tangential and radial swelling of the control and treated wood: (a) water vapor sorption and (b) water immersion. Bars with different letters indicate significant difference ($p < 0.05$) in accordance with Duncan’s multiple range tests. Note: There are no significant differences between groups containing the same letters. The bar with ab means that there are no significant differences between this group and groups labeled with a or b, while the bar with AB means that there are no significant differences between this group and groups labeled with A or B.

### 3.4. Effect of Heat Treatment and Wax Impregnation on Bending Strength and Bending Stiffness

The bending strength (MOR) and bending stiffness (MOE) of the control and treated groups are shown in Figure 3. No significant difference ($p < 0.05$) among control, 120 °C/4h HT, and 120 °C/4h HT + WI specimens was determined. Only high-temperature (180 °C) HT decreased the MOR in current work, which is in agreement with previous studies [18,27,31,33]. These results demonstrate that moderate-temperature HT and moderate-temperature HT + WI had little influence on wood bending strength. However, the MOE of treated groups increased significantly, except for in the 120 °C/4h HT group. Several previous studies [36,37] showed that MOE of heat-treated wood under lower treatment severity was higher than control, which is in agreement with the current study. The reduction of MOR could be attributed to the degradation of hemicellulose and evaporation of extractives during heat treatment [38], while MOE loss is highly dependent on the density of treated wood. The increase of MOE shown by the WI group may be attributed to the impregnation of wax. Wax fills the cell lumens fully or partly, improving the density of treated wood; therefore, the MOE of wood after wax impregnation was improved.

**Figure 3.** Mechanical properties of the control and treated wood: (a) MOR and (b) MOE. Bars with different letters indicate significant difference ($p < 0.05$) in accordance with Duncan’s multiple range tests. Note: There are no significant differences between groups containing the same letters. The bar with ac means that there are no significant differences between this group and groups containing a or c.
4. Conclusions

Certain properties of wood were increased via the synergistic combination of moderate-temperature HT and WI. WI effectively improved the hygroscopicity, dimensional stability, and mechanical properties as compared with the moderate- and high-temperature HT, as well as the combination of the two. Capacity and swelling of wood in adsorption and liquid water uptake conditions were decreased significantly after 180 °C HT and WI. The reductions were the lowest for WI wood. The 120 °C HT group exhibited significant decreases only for capacity of adsorption and the swelling of liquid water uptake. The ratio of adsorption swelling between tangential (T) and radial (R) directions decreased after 180 °C/4h HT and 120 °C/4h HT + WI, while the ratio of immersion swelling remained almost constant for all treated wood. There was no significant influence of 120 °C/4h HT and WI on wood bending strength (MOR), which decreased significantly only after 180 °C HT; 180 °C HT and WI improved bending stiffness (MOE) significantly, while no significant influence of 180 °C HT on MOE was observed. Therefore, WI played a significant role in improvement of wood hygroscopicity, dimensional stability, and mechanical properties. The combination of moderate temperature acted synergistically only in certain aspects of wood properties such as capacity of water adsorption and liquid water uptake. The combination of moderate-temperature heat treatment and wax impregnation is more suitable for the modification of indoor wood products.

Author Contributions: Writing—original draft preparation, L.Y.; writing—review and editing, H.-H.L. and L.Y. All authors have read and agreed to the published version of the manuscript.

Funding: This research was funded by the National Natural Science Foundation of China (Grant No. 31870545 and 31570558) and Key Laboratory of Bio-based Material Science & Technology (Northeast Forestry University), Ministry of Education (SWZ-MS201903).

Acknowledgments: Special acknowledgment is extended to the Xian You Degoo Furniture Company for their assistance.

Conflicts of Interest: The authors declare no conflict of interest.

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