Effect of Wax and Dimethyl Silicone Oil Pretreatment on Wood Hygroscopicity, Chemical Components, and Dimensional Stability

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Wood is a renewable and environmental friendly material, but its low dimensional stability characteristics limit its applications. In this study, wax mixed with dimethyl silicone oil was used to enhance the dimensional stability under heat treatment. Samples were heated at 120 °C under 3 impregnation conditions (wax, wax + 20% dimethyl silicone oil, and wax + 40% dimethyl silicone oil) for 3 and 6 h respectively. After treatment the effects of combination pretreatment on wood weight gain percentage (WPG), tangential, radial and volume swelling coefficients (TS, RS, VS), distribution of impregnation liquid, and the types of functional groups of African Padauk (*Pterocarpus soyauxii*) were evaluated. The results showed that impregnation improved the dimensional stability of wood to a certain extent; moreover, the addition of dimethyl silicone oil improved the modification effect. Furthermore, the VS reduced to 0.66 (±0.28)% in the treatment of wax + 20% dimethyl silicone oil for 6 h. The impregnation liquid mainly adhered to the walls of vessels and ray cells. The hydroxyl absorption intensity of the impregnated groups was lower than that of the control group.

Keywords: Dimensional stability; FTIR; SEM; WPG; Wood modification

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INTRODUCTION

Wood is a renewable and environmentally friendly material. It provides numerous advantages, rendering it applicable in many aspects of human life. Specifically, many hardwood materials are judged as satisfactory due to their excellent properties (such as color, texture, *etc.*). However, low dimensional stability limits their wide application. Since wood shrinks and swells with changes of temperature and humidity, wood modification is a key step before wood products are put into use (Sun *et al.* 2010; Li *et al.* 2012). Wood modification alters the material to overcome or ameliorate its disadvantages. Hill (2006) further subdivided wood modification methods based on Norimoto and Gril (1993) into four types: chemical modification, thermal modification, surface modification, and impregnation modification.

Wax modification is an impregnation method. Wax has many advantages, such as cost-effectiveness, abundance, and low toxicity (Chau *et al.* 2015). The wax impregnation of wood is thought to have originated from the Chinese Ming dynasty, while the use of wax has a history spanning more than 3,000 years in China.

Wax impregnation is usually conducted at elevated temperature and concentration, either in a vacuum or under pressure. Based on existing research, the wax application of wood can be divided into two categories: wax impregnation (Scholz *et al.* 2010b; Chau *et al.* 2015; Li *et al.* 2015; Wang *et al.* 2015; Yang *et al.* 2017) and wax impregnation combined with other treatments (Partansky 1959; Wang *et al.* 2016; Humar *et al.* 2016; Liao *et al.* 2016). Wax was used as a water repellent to reduce hygroscopicity and water absorption (Feist and Mraz 1978; Ghosh *et al.* 2009; Xie *et al.* 2013), thereby improving the dimensional stability of wood for long-term use. The rate of hygroscopicity and water absorption could be reduced after wax fills the cell cavity (Papadopoulos and Pougioula 2010).

Wax impregnation could also improve the termite resistance of wood; moreover, the effect was found to be related to the type and the proportion of wax (Scholz *et al.* 2010b). Wax once was applied to the surface of wood to improve the resistant of wood decay fungi, although it could not improve resistance to blue stain fungi (Lesar and Humar 2011). In addition, different from chemical modifications (Dunningham *et al.* 1992), all the evaluated mechanical properties of the wax-treated wood in previous studies were improved (Hill 2006; Scholz *et al.* 2010a; Möttönen *et al.* 2015). As for combined treatments, they had a synergistic effect that can better improve the properties of the wood. The combination of wax impregnation and thermal modification could not only improve the hydrophobicity, dimensional stability, and the resistance against fungal decay of wood considerably, but also reduce the uptake of liquid water and water vapor (Wang *et al.* 2015; Humar *et al.* 2016). The wax and copper azole emulsion compound systems could lower the relaxation, reduce the water absorption, and improve the shrinkage and swelling as well (Liao *et al.* 2016; Wang *et al.* 2016). The cutting qualities were improved by impregnating the wood with wax and polyethylene glycol (Partansky 1959).

Moreover, the wood quality could be improved by heat treatment with a temperature below 260 °C (Sidorova 2008). Oil heat treatment (OHT) was shown to transfer heat in the wood effectively and uniformly and had already in use in Germany (Boskou 2011). Vegetable oil is one of the heat transfer media because of their boiling point exceeding 260 °C. Under anaerobic conditions, a certain amount of vegetable oil was absorbed into wood, which can improve wood performance (Cheng *et al.* 2013). Besides, dimethyl silicone oil was applied to heat treatment as the most common and well-studied silicone oil (Noll 1968; Weigenand *et al.* 2007). Okon *et al.* (2017) was the first to use dimethyl silicone oil for heat treatment of Chinese fir. Analysis of the physical and chemical properties of Chinese fir samples showed that its dimensional stability was enhanced. In addition, dimethyl silicone oil treated wood had greater resistance to soft rot and showed lower weight loss and loss of dynamic MOE than untreated wood (Ghosh *et al.* 2008; Weigenand *et al.* 2008; Ghosh 2009).

As previously stated, few impregnation tests have been performed directly with melted wax at 120 °C under atmospheric pressure. No previous studies have used dimethyl silicone oil in the combined treatment with wax to modify *P. soyauxii*. This study evaluated the improvement in the dimensional stability of *P. soyauxii* with different dimethyl silicone oil to wax ratios. The distribution of the impregnation liquid was observed by scanning electron microscope (SEM). Moreover, Fourier-transform infrared spectroscopy (FTIR) analysis of wood samples was carried out before and after the impregnation modification.

EXPERIMENTAL

Materials

African Padauk (*Pterocarpus soyauxii*) (4.80% moisture content) with an air-dry density of 0.63 g/cm^3 were collected from YiJiuXuan company, Xianyou, China. Heartwood was selected as the test material and cut into specimens of 20 mm (L) × 20 mm (R) × 20 mm (T). The specimens were free of knots and lacked visible evidence of infection by mold, stain, or fungi. 85# microcrystalline wax with a melting point of 82 °C to 87 °C was used. Dimethyl silicone oil, a colorless (or light yellow), tasteless, and high-transparency liquid with a thermal conductivity of 0.134 to 0.159 W/(M*K), can be used long-term at -50 °C to 200 °C.

Methods

Determination of initial moisture content

The air-dry samples were numbered, weighed, and recorded in accordance with GB/T 1931 (2009) (China) to determine moisture content.

Wax impregnation

A total of 105 specimens were selected before they were randomly classified into seven groups (A, B, C, D, E, F, and G), with 15 specimens numbered 1 to 15 in each group. The experimental design is presented in Table 1. A certain amount of dimethyl silicone oil was added into the fluid wax obtained by melting the solid wax in a steel tank (32 cm × 16 cm × 16 cm) at 120 °C. The test material was immediately dipped into the stirring-well mixture to prevent the liquid from curding. After impregnation, the wood samples were wiped to remove residual impregnation liquid and then cooled in a silica gel desiccator balanced at room temperature.

Table 1. Experimental Design of the Processes Conducted at 120 °C

Group Information	A	B	C	D	E	F	G
Dimethyl Silicone Oil Proportion (%)	0	0	20	20	40	40	_
Impregnation Time (h)	3	6	3	6	3	6	_
Impregnation Temperature (°C)	120	120	120	120	120	120	_

Characterization experiments

Weight gain rate (WPG) was conducted on an AR124CN electronic balance (Ohaus Instruments Co., Shanghai, China) and calculated using Eq. 1,

$$\Delta G = \frac{G_1 - G_0}{G_0} \times 100\% \quad (1)$$

where ΔG represents the WPG (%) of the specimen after wax impregnation relative to before treatment; G_0 and G_1 denote the weight of oven-dry samples before and after impregnation treatments, respectively.

All specimens underwent hygroscopicity testing at a constant temperature and in a humidity chamber (DHS-500, Beijing Yashilin Test Equipment Co., Beijing, China) at 20 °C and a relative humidity of 65% in accordance with the GB/T 1934.2 (2009) standard (China). After moisture absorption, the changes of tangential, radial, and volume (which is the product of tangential dimension, radial dimension and longitudinal dimension) swelling coefficients (marked by TS, RS, and VS, respectively) were determined by the following equation,

$$S=\frac{S_1-S_0}{S_0}\times 100\ \% \quad (2)$$

where S_0 and S_1 are the tangential dimensions (radial dimensions or volumes) of wood samples before and after impregnation, respectively.

At the same time, in order to observe the internal wax distribution, seven pieces of wood from six experimental groups and the control group were randomly selected. The tangential section was observed by scanning electron microscopy (SEM) (Hitachi S－3400N, Techcomp (China) Ltd, Beijing, China).

The Fourier transform infrared (FTIR) spectra of 4 samples selected randomly from group B, D, F, and G were obtained by infrared spectrometry (TENSOR27, Tianjin Optical Instrument Factory, Tianjin, China). Measurements of 4000 to 500 cm^{-1} were recorded. Prior to testing, all samples were prepared as 100 to 120 purpose wood flour and then dried at 103 ± 2 °C.

RESULTS AND DISCUSSION

Weight Gain Percentage

The WPG reflects the quantity of the impregnation liquid entering the wood. Figure 1 shows the WPG of *P. soyauxii* impregnated under different conditions. The WPG of wood treated for 6 h was higher than that treated for 3 h under all impregnation conditions. However, the growth rates in the latter 3 h of the 6 h impregnation groups were slower than that in the previous 3 h period. According to the experimental data, the calculated WPG growth rates of the latter 3 h period were 8.18% (group A, B), 29.9% (group C, D), and 42.16% (group E, F) of the growth rates obtained in the previous 3 h period. This may be due to the fact that the impregnation was completed basically at a certain point during the latter 3 h period, so that, the impregnation liquid could not further fill the cell pores as time went on.

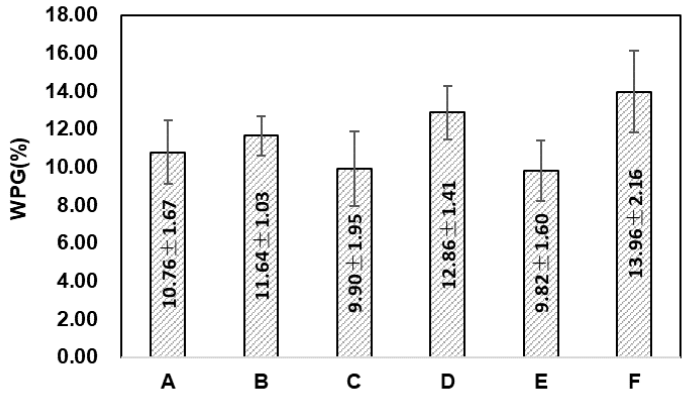

Fig. 1. Weight gain rates of the impregnated groups. (A) wax 3 h, (B) wax 6 h, (C) wax+20% dimethyl silicone oil 3 h, (D) wax+20% dimethyl silicone oil 6 h, (E) wax+40% dimethyl silicone oil 3 h, (F) wax+40% dimethyl silicone oil 6 h

WPG showed a decreasing trend with an increase in the proportion of dimethyl silicone oil after impregnation for 3 h, while it increased along with the increasing

proportion of dimethyl silicone oil when the impregnation time was 6 h. The WPG of group A without dimethyl silicone oil was 11.64(±1.67)%, which was increased to 13.96(±2.16)% in group F with 40% dimethyl silicone oil.

Dimensional stability

Figure 2 shows the tangential swelling coefficients (TS) of *P. soyauxii* in seven groups. The TS of six impregnated groups were less than that of the control group. Moreover, the TS was lower when the impregnation time was extended to 6 h than 3 h. This finding indicates that the tangential dimensional stability increased with time. When the impregnation time was 3 h, TS decreased to 2.28 (±0.3)% in group C with 20% dimethyl silicone oil, which was 0.26% less than that of the control group. The TS of the group B impregnated in wax for 6 h was reduced to 1.45 (±0.19)% which was nearly half of the control group. When adding 40% dimethyl silicone oil, tangential dimensional stability was significantly improved, as the TS values were 1.91 (±0.16)% for 3h and 1.77 (±0.27)% for 6 h. When impregnating for 3 h, the TS value became smaller as the proportion of dimethyl silicone oil increased. The slight tangential dimensional stability enhancement may be attributed to the good lubrication of dimethyl silicone oil so that wax can more easily enter into wood. The experiment shows that the lowest TS was obtained in group B rather than other groups with added dimethyl silicone oil. The possible reason is that the wood had a limited volume to accommodate impregnation liquid, part of the volume of wax was replaced by dimethyl silicone oil.

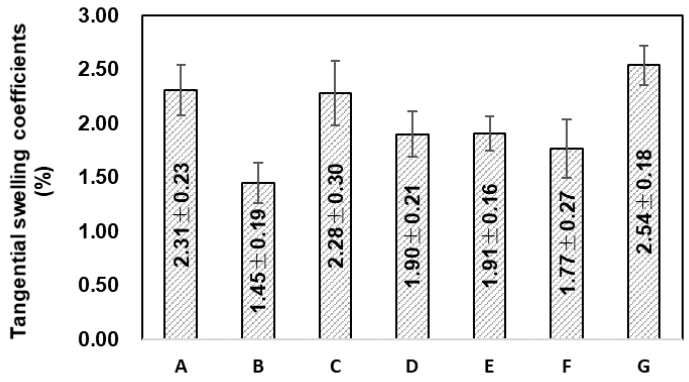

Fig. 2. The tangential swelling coefficients of the control group and the impregnated groups. (A) wax 3 h, (B) wax 6 h, (C) wax+20% dimethyl silicone oil 3 h, (D) wax+20% dimethyl silicone oil 6 h, (E) wax+40% dimethyl silicone oil 3 h, (F) wax+40% dimethyl silicone oil 6 h, (G) control group

Figure 3 shows seven groups' radial swelling coefficients (RS) of *P. soyauxii*. The figure shows that the RS obtained by 6 h impregnation was lower than that for 3 h. In addition, the radial dimensional stability of the impregnated groups improved markedly. Radial dimensional stability increased mostly when the dimethyl silicone oil ratio was 20% impregnated for 6 h, and it reduced from 1.85 (±0.45)% to 1.23 (±0.14)% in accordance with the specific RS value.

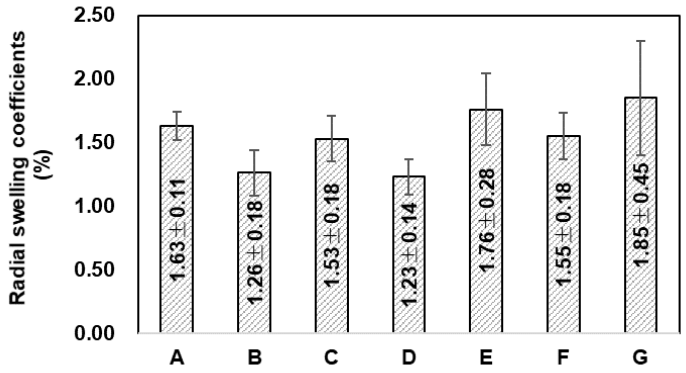

Fig. 3. The radial swelling coefficients of the control group and the impregnated groups. (A) wax 3 h, (B) wax 6 h, (C) wax+20% dimethyl silicone oil 3 h, (D) wax+20% dimethyl silicone oil 6 h, (E) wax+40% dimethyl silicone oil 3 h, (F) wax+40% dimethyl silicone oil 6 h, (G) control group

Figure 4 shows that the volume swelling coefficients (VS) of the impregnated groups were reduced compared with the control group. When the impregnation time was 3 h, the VS of the groups increased gradually with the increase in the proportion of dimethyl silicone oil but decreased with the increase in impregnation time. Moreover, the VS was 1.65 (±0.52)% after impregnating with wax for 3h, which was the lowest of the 3 impregnation conditions. Under the treatment with wax + 20% dimethyl silicone oil for 6 h, the VS was as low as 0.66 (±0.28)%, which was 20% of the control group (3.85± 0.26%).

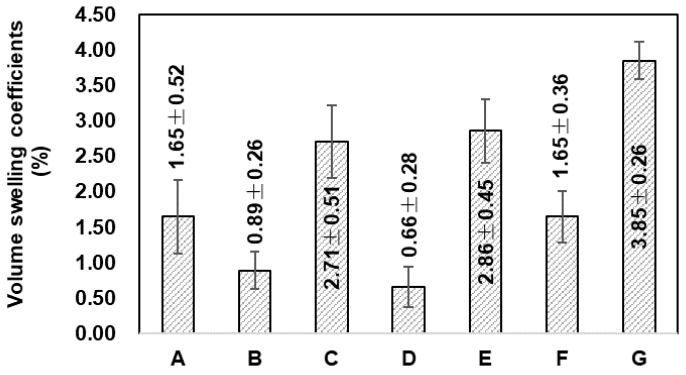

Fig. 4. The volume swelling coefficients of the control group and the impregnated groups. (A) wax 3h, (B) wax 6h, (C) wax+20% dimethyl silicone oil 3h, (D) wax+20% dimethyl silicone oil 6h, (E) wax+40% dimethyl silicone oil 3h, (F) wax+40% dimethyl silicone oil 6h, (G) control group

Therefore, the WPG was the largest under the treatment with an impregnation time of 6 h in wax + 40% dimethyl silicone oil, the TS was the lowest under the wax treatment for 6 h, the RS and the VS were the lowest under the wax + 20% dimethyl silicone oil treatment for 6h. The likely reason is that dimethyl silicone oil has better lubricity than wax, thereby making wax enter into wood more easily. However, the ratio of dimethyl silicone oil to wax has a critical value, if the proportion of dimethyl silicone oil continues to increase upon extending the critical value, the proportion of the wax that enters the wood will be reduced even though the WPG increases.

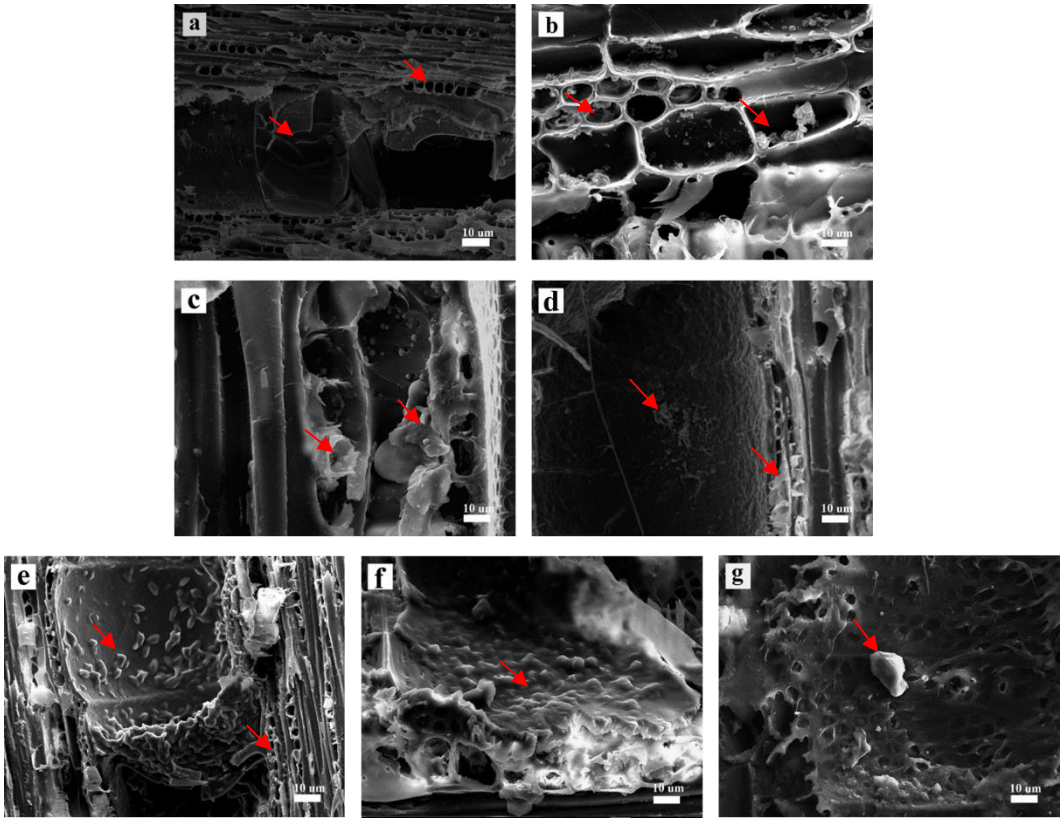

Fig. 5. The SEM of the control group and the impregnated groups. (a) control group, (b) wax impregnated 3 h, (c) wax impregnated 6 h, (d) wax + 20% dimethyl silicone oil impregnated 3 h, (e) wax + 20% dimethyl silicone oil impregnated 6 h, (f) wax + 40% dimethyl silicone oil impregnated 3 h, (g) wax + 40% dimethyl silicone oil impregnated 6 h

Scanning Electron Microscopy

The distribution of the impregnation liquid within the wood was observed by SEM. The control specimen imaging was darker and gloss-free, which was contradictory to the impregnated specimens. In Fig. 5(a), small cracks on the vessel wall were visible, and no obvious attachments inside the vessel and wood ray cells could be clearly seen. Figures 5(b) to 5(g) represent the internal distribution of the impregnation solution under different treatments in the tangential section; the arrows added point to part of the internal impregnation liquid distributed in the vessels and wood rays, respectively. Figures 5(e) - 5(f) suggest that the perforated plate between the two vessels can accumulate more impregnation liquid, compared with the vessel wall. Figures 5(b) - 5(c) show the specimens tested with pure wax impregnated for 3 h and 6 h separately, and wax mostly showed a massive distribution and distinctly shaped droplets on the surface; the overall distribution was uneven. Figures 5(d) - 5(e) show the internal view when 20% dimethyl silicone oil is added. Figure 5(d) shows a thin layer of impregnation liquid on the surface of the vessel with a uniform distribution. When the impregnation liquid turns into small, numerous droplets, they become more evenly distributed on the internal surface of the vessel, as shown in Figure 5(e). Meanwhile, figures 5(f) - 5(g) present the wood after adding dimethyl silicone oil up to 40%, and most of the impregnated liquid was uniformly deposited on the perforated plate.

Fourier Transform Infrared Spectroscopy

The wax is intended to improve the dimensional stability of the sample with the hydrophobic properties of wax. After they are absorbed into the wood, the wax particles wrap free hydroxyl groups with hygroscopicity, which considerably reduces deformation. In this study, *P. soyauxii* showed apparent changes. FTIR spectroscopy was used to analyze changes in the internal groups. The chemical composition of the cell wall can be determined by the peaks in the 4000 to 500 cm^{-1} wavelength range of *P. soyauxii*.

Table 2 presents the chemical composition of the characteristic peaks. The dotted lines were added in Figs. 6 to 10 to identify peaks; from left to right, the first peak at 3400 cm^{-1} represents -OH, which affects the moisture absorption of the wood and on behalf of cellulose. The wavelength of the second peak, which is 2900 cm^{-1}, denotes the aliphatic C-H vibration peak, which is the characteristic peak of cellulose. The wavelength of the third peak is 1735 cm^{-1}, representing the C=O stretching vibration, which is related to lignin. The wavelengths of the fourth and fifth peaks, 1600 and 1508 cm^{-1}, respectively, are associated with the characteristic peak of lignin (Tjeerdsma and Militz 2005; Huang *et al.* 2012; Luo *et al.* 2013; Gu *et al.* 2014), where 1266 cm^{-1} is the wavelength of the guaiac. This analysis mainly considers the changes in -OH. The intensity of the -OH absorption peak at 3410 cm^{-1} of *P. soyauxii* decreased slightly, as determined when the results for the treated groups were compared with those for the control group.

Table 2. FTIR Analysis

Wave Number (cm^{-1})	Group Attribution	Corresponding Chemical Composition
3410	O-H Stretching Vibration	Cellulose, Phenol, Alcohol, Carboxylic Acid Compounds
2900	C-H Stretching Vibration	Cellulose
1735	C=O Stretching Vibration	Hemicellulose, Lipids, Ketones
1600	Aromatic Carbon Skeleton Vibration	Lignin
1508	Aromatic Carbon Skeleton Vibration	Lignin, Aromatic Hydrocarbons
1463	C-H Stretching Vibration, CH3, CH2 Asymmetric Bending Vibration	Lignin, Ether Compounds
1427	Aromatic Carbon Skeleton Vibration and C - H Bending Vibration	Lignin, Cellulose
1266	G Ring and Acyloxy C-O Stretching Vibration	Lignin
1160	C-O-C Stretching Vibration	Cellulose, Hemicellulose
895	Different Carbon (C) Vibration Frequency	Cellulose

Figure 6 shows the infrared spectra of the wax impregnated groups and the control group. At the wave number of 3400 cm^{-1}, the curve from the top to the bottom shows the wax treatment for 6 h and 3 h as well as the control group. The highest to lowest absorption intensities of -OH, from highest to lowest, were obtained as follows: the control group > the group impregnated for 3 h > the group impregnated for 6 h.

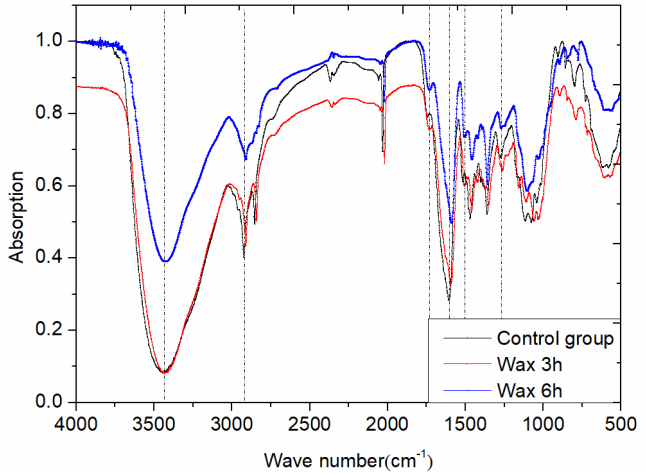

Fig. 6. Infrared spectra of the control group and the wax impregnated groups

Figure 7 shows the FTIR curves of the wood impregnated with wax + 20% dimethyl silicone oil and the control group. The figure illustrates that the hydroxyl absorption intensity of the specimen treated for 6 h is lower than that treated for 3 h.

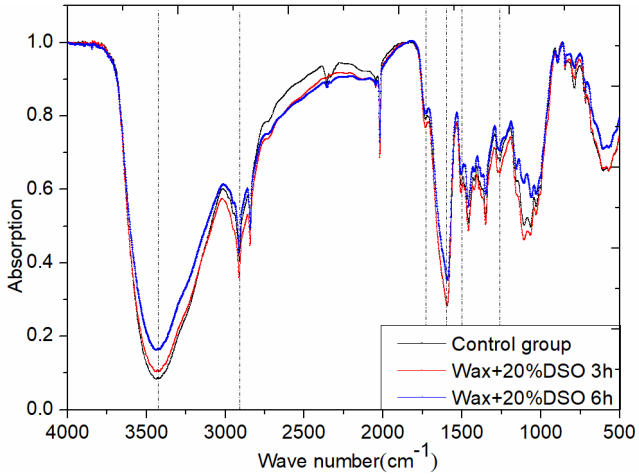

Fig. 7. Infrared spectra of the control group and the wax + 20% DSO impregnated groups. (DSO) represents dimethyl silicone oil

Figure 8 presents the infrared spectra of groups impregnated with wax + 40% dimethyl silicone oil and the control group. The group impregnated for 3 h had the smallest number of free hydroxyl groups, while the group impregnated for 6 h had lower hydroxyl absorption intensity than the control group.

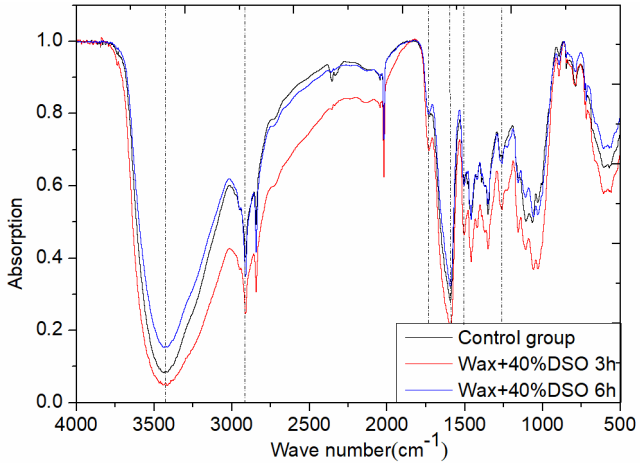

Fig. 8. Infrared spectra of the control group and the wax + 40% DSO impregnated groups. (DSO) represents dimethyl silicone oil

Figures 9 and 10 present a comparison of infrared spectra between the treated wood under three different impregnation conditions at the same treatment time and the control group. Figure 9 shows that the impregnation time was 3 h. The wax impregnated group and the 20% dimethyl silicone oil group had the same moisture absorption intensity, which was lower than that of the control group. The 40% dimethyl silicone oil group obtained the lowest moisture absorption intensity. As shown in Fig. 10, the 40% dimethyl silicone oil group and the 20% dimethyl silicone oil group had almost the same moisture absorption intensity, which was lower than that of the control group after treatment for 6 h. Meanwhile, the moisture absorption intensity of the wax impregnated group was considerably lower than that of the control group.

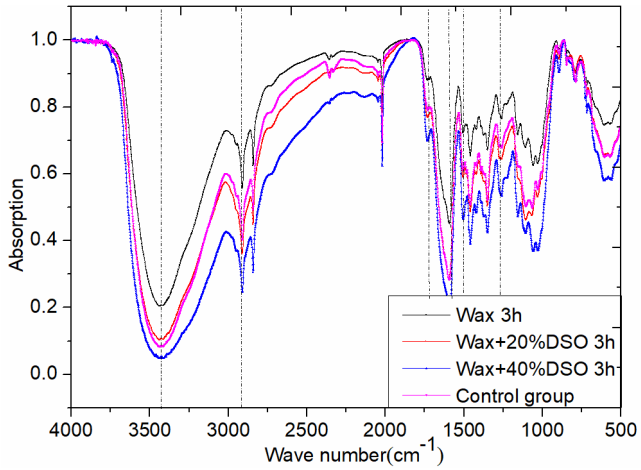

Fig. 9. Infrared spectra of the control group and the 3 h impregnated groups. (DSO) represents dimethyl silicone oil

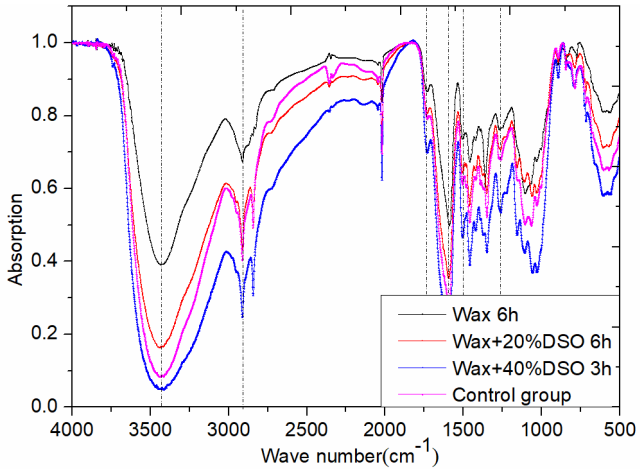

Fig. 10. Infrared spectra of the control group and the 6 h impregnated groups. (DSO) represents dimethyl silicone oil

Combined analysis of the VS and FTIR of *P. soyauxii* indicated that when the impregnation time was 3 h, the wood was not completely impregnated. The VS, from highest to lowest was obtained as follows: the 40% dimethyl silicone oil group > the 20% dimethyl silicone oil group > the wax group. In addition, the infrared measurements of the hydroxyl group strength, from highest to lowest, were as follows: wax group > 20% dimethyl silicone oil group > 40% dimethyl silicone oil group. The lowest VS was obtained when the moisture absorption intensity of the free hydroxyl group was the smallest after the impregnation treatment. This result could be attributed to either the free hydroxyl group of the wood itself or to the addition of dimethyl silicone oil. In this experiment, dimethyl silicone oil increased the flowability of wax and facilitated the absorption of wax into the wood. Although the free hydroxyl could be wrapped around the wax to reduce the moisture absorption to a certain extent, dimethyl silicone oil, despite its insolubility in water, could easily absorb moisture with an increase in humidity. With 40% dimethyl silicone oil added to the treatment, the total proportion of dimethyl silicone oil was larger because of the extremely high concentration of dimethyl silicone oil in the environment and the greater ease of entry into the wood, compared with other materials. The VS of the 40% dimethyl silicone oil group was greater than the VS of the wax group and less than the VS of the untreated group. After 6 h, impregnation was basically completed, and the hydroxyl intensities were as follows: 40% dimethyl silicone oil group > 20% dimethyl silicone oil group> wax group. The VS, from highest to lowest, were obtained, as follows: 40% dimethyl silicone oil group > wax group > 20% dimethyl silicone oil group. This finding could be attributed to the fact that as the dimethyl silicone oil concentration increased, the amount of wax entering the wood also increased because of increased lubricity, and the number of free hydroxyl groups was reduced. On one hand, the decrease in the number of hydroxyl groups could increase the dimensional stability; on the other hand, the increase in dimethyl silicone oil concentration enhanced the moisture absorption capacity of the wood. Therefore, with an increase in dimethyl silicone oil concentration after impregnation for 6 h, the amount of wax absorbed into the wood was increased, the number of free hydroxyl groups exposed was decreased, and the intensity of the hydroxyl was decreased. As for the VS of wood, more wax could be absorbed into the wood to improve the dimensional stability of the material with an increase in dimethyl silicone oil concentration. However, the dimethyl silicone oil concentration was increased, thereby increasing the

moisture absorption of wood. Therefore, the increase in the dimensional stability of the final wood was related to the ratio of wax to Dimethyl silicone oil. In this experiment, 20% dimethyl silicone oil was added into the solution to achieve the highest dimensional stability of *P. soyauxii*.

CONCLUSIONS

1. The wax and dimethyl silicone oil pretreatment could improve the dimensional stability of *P. soyauxii*. The TS, RS, and VS decreased with the increase of time. However, the modification of dimensional stability was not doubled with time. Therefore, impregnation time should be considered as a factor in manufacturing to obtain optimal results.
2. The effect of modification was related to the dimethyl silicone oil concentration and the optimal property of *P. soyauxii* was obtained by the impregnation of wax + 20% dimethyl silicone oil in this experiment. The TS was decreased to as low as 1.9(± 0.21)%, the RS and the VS was reduced to 1.23(±0.14)% and 0.66(±0.28)%, which was 66.6% and 17% of the control group after impregnating for 6 h.
3. The impregnation liquid mainly adhered to the vessel wall and the ray cell, as observed by SEM. Meanwhile, under different impregnation conditions, the distribution of the impregnation liquid exhibited a distinct shape. The absorption intensity of treated wood was lower than that of the control group. When impregnated in wax + 40% dimethyl silicone oil for 3 h, the treated wood obtained the lowest strength. The overall change in the hydroxyl absorption intensity of the 7 specimens was not significant, since wax impregnation modification is physical modification. In the future, researchers can continue to study the mechanical properties, termite resistance and the resistance against fungal decay of impregnated wood.

ACKNOWLEDGMENTS

This paper was supported by Major Scientific and Technological Achievements Incubation Projects in Beijing Forestry University – The Key Technology of High Efficiency Pretreatment of Solid Wood Furniture (2017CGP014), the Fundamental Research Funds for the Central Universities of China (2015ZCQ-CL-01), and the Hot Tracking Project in Beijing Forestry University-Wood Solar Synergistic Energy-Saving Drying Technology (2017BLRD04).

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Article submitted: April 19, 2018; Peer review completed: June 15, 2018; Revisions accepted June 27, 2018; Published: June 28, 2018.
DOI: 10.15376/biores.13.3.6265-6279