Effect of Beeswax Impregnation on the Dimensional Stability, Surface Properties, and Thermal Characteristics of Wood

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Wood is both renewable and natural, which are qualities that make it both environmentally sustainable and useful in terms of development. However, wood also has certain inherent defects, such as its tendencies to shrink when dried and rot when wet. These defects restrict the use and popularity of lumber as a building material. In this paper, the effects of beeswax impregnation on the dimensional stability of wood were studied. Pieces of African padauk (*Pterocarpus soyauxii*) (20 mm × 20 mm × 20 mm) were used as the test material. The wood was treated at a temperature of 120 °C for periods of 3 h or 6 h. Measurements of weight gain rate, size expansion coefficient, and contact angle of the control samples were compared with samples treated with beeswax for 3 h and 6 h to explain macroscopic changes in wood. The effects of beeswax impregnation were compared using scanning electron microscopy, thermal weight loss characteristic analysis, and functional group analysis. The results indicated that, to some extent, beeswax impregnation improved the dimensional stability of wood and remarkably enhanced its surface hydrophobicity.

Keywords: Dimensional stability; Beeswax; Impregnated wood; WPG; TG; FTIR

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INTRODUCTION

Wood has been indispensable throughout human history. It is natural and endlessly reproducible. It is one of those rare materials that is both environmentally friendly and useful in terms of development (La Mantia and Morreale 2011). Wood possesses many natural advantages, such as its lack of electrical conductivity, its high strength-to-weight ratio, its insulating properties, its superior acoustic performance, and its temperature stability. In contrast, wood also has some major natural defects, such as dry shrinking, rotting in wet conditions, hot air deformation, and its susceptibility to fungi (Sablík *et al.* 2016). Due to the essential anisotropy and variability of wood, it is difficult to avoid encountering its various defects and restrictions when utilizing it as a material.

To improve the dimensional stability of wood, most current research aims to improve its performance through chemical, biological, or physical methods, which act on the wood through a range of media (Di Blasi *et al.* 2008; Clausen *et al.* 2010; Simsek *et al.* 2010; Qiu *et al.* 2016). These modification methods can be divided into active modification (change the chemical properties), passive modification (not change the chemical properties), and combination modification (which combines the other two). In this experiment, the impregnation modification method was used, which is a widely applied passive modification method (Scholz *et al.* 2010a,b). This method involves immersing the

wood in an impregnating agent so that the impregnation reagent achieves modification by entering the cell walls. Impregnation modification has the advantage of being both logistically simple and environmentally friendly.

In this experiment, beeswax was used as the impregnant. At present, petroleum wax is a more commonly used wax in wood wax impregnation. Petroleum wax having a high melting point is considered a more effective medium than low melting point petroleum wax (Chau *et al.* 2015; Li *et al.* 2015b; Humar *et al.* 2017; Jiang *et al.* 2018; Wang *et al.* 2018; Chen *et al.* 2019). Some types of wood impregnated with paraffin and dimethyl silicone oil are more effective than those impregnated with petroleum wax alone (Okon *et al.* 2017; Qian *et al.* 2018). However, the dewaxing process in the later stage of wax treatment is more complicated, because the wax cools and sticks to the wood surface, from which is difficult to remove. Beeswax is a complex organic compound. Esters, fatty acids, and sugars synthesized from higher fatty acids and mono-alcohols are the main components of beeswax, but there are also some differences in these components due to differences in bee species, honey powder source plants, and extraction methods. Beeswax is widely used in manufacturing, agriculture, animal husbandry, medicine, food, and other fields (Cavallaro *et al.* 2015; Németh *et al.* 2015). It has the characteristics of low melting point, low density, large yield, and lack of pollution (Regert *et al.* 2001; Bogdanov 2004). Currently, beeswax is mostly used to treat the surface properties of wood. In such applications, the color stability, hydrophobicity, and antibacterial properties of wood treated with beeswax have been greatly improved. However, there is still minimal research on the potential for improving the dimensional stability of wood *via* beeswax impregnation (Petric *et al.* 2004; Chen and Yan 2012).

EXPERIMENTAL

Materials

Samples of African padauk (*Pterocarpus soyauxii*), with a 4.90% moisture content and an air-dry density of 0.97 g/cm^3, were collected from the YiJiuXuan company (Xianyou, China). The dimensions of the wood samples were 20 mm × 20 mm × 20 mm (radial × tangential × longitudinal). The impregnation liquid was beeswax (with a melting point at the onset 42 °C) provided by JiaLeJie Company (Beijing, China).

Methods

Beeswax impregnation heated treatment- Material processing

First, wood samples without obvious defects were selected, any surface burrs were scraped off, and then their dimensions were numbered and measured. A total of three sets of samples were selected, with 15 in each group, of which one group served as a control.

Dry processing

The wood was dried according to the Chinese national standard GB/T 1931 (2009). The samples were placed into the oven according to batch number. The oven temperature was 103 ± 2 °C, and the samples were baked for 8 h. Two to three samples of the wood were selected for one test by sampling in five locations, and the selected samples were weighed once every 2 h. The test piece was considered completely dry when its weight difference was not more than 0.5% of its mass.

Impregnation processing

The immersion treatment temperature was 120 °C, and the immersion treatment times were 3 h and 6 h. The impregnation media was beeswax, which was placed in a dipping wax bath and was not diluted. The oven temperature was set to 120 °C, and the impregnation was completed in the dipping wax bath once the beeswax was completely melted. At this point, the African padauk samples were immersed in the dipping wax tank, which remained at a treatment temperature of 120 °C. When the impregnation was complete, the remaining beeswax was wiped away with a paper towel.

Characterization Experiments

Weight gain rate

Weight gain rate (WPG) was calculated using Eq. 1,

$$\triangle G\,(\%) = (G_1 - G_0)/G_0 \times 100 \quad (1)$$

where ΔG is the WPG of the specimen after wax impregnation relative to that before treatment (%), G_1 is the mass of the waxed material, and G_0 is the mass of the dry material (before impregnation).

Hygroscopicity test- Moisture treatment

After the impregnation was completed according to the Chinese national standard GB/T 1934.2 (2009), the treated material and the control group were placed in an environment with a temperature of 20 ± 2 °C and a relative humidity of 65 ± 3%. During the moisture absorption process, the tangential size was measured every 6 h for each sample group. The temperature and humidity adjustments were considered complete when the difference between two subsequent measurements did not exceed 0.02 mm.

Water absorption treatment

The samples were immersed in a container filled with distilled water until the moisture content stabilized. After being immersed for 20 d, two or three samples were selected to measure their tangential size. After this, they were measured every 3 d. If the difference between two measurements was not more than 0.02 mm, then their size was considered to be stable. The distilled water in the container was clean and was replaced every 4 d to 5 d.

Next, swelling rate of the samples between dry and air dry was calculated using Eq. 2,

$$a_w\,(\%) = (l_w - l_0) / l_0 \times 100 \quad (2)$$

where a_w is the swelling coefficient (radial, tangential, and volumetric) (%) from dry to air dry, l_0 denotes the initial dimensions of the samples (after impregnation) (mm), and l_w represents the dimensions after the samples have been equilibrated to 20°C and 65% RH (mm). Once they had stabilized after the water absorption process, the swelling rates of the samples were calculated using Eq. 3,

$$a_{max}\,(\%) = (l_{max} - l_0) / l_I \times 100 \quad (3)$$

where a_{max} is the swelling coefficient (radial, tangential, and volumetric) (%) once it has absorbed water to a stable size, and l_{max} represents the dimension at the end of the water absorption treatment (mm) once it has absorbed water to a stable size.

Contact angle

After dewaxing, select nine points on the wood surface to test the contact angle. The differences in liquid and solid contact angles between untreated and beeswax-treated wood samples were measured using the suspended drop method and a contact angle meter (Optical Contact Angle Meter, OCA 20; DataPhysics Instruments GmbH, Filderstadt, Germany).

The actual measurement time was more than 120 s. The curve was drawn by selecting the data from 0 s to 100 s, and each group was measured three times. The average value was calculated as the final test result.

Scanning electron microscopy (SEM)

Beeswax-treated wood samples of African padauk was randomly selected. The tangential surface layer and the tangential center layer were taken to prepare a sample sheet of 5 mm × 5 mm × 1 mm, and was observed by scanning electron microscopy (Gemini SEM 500, Beijing, China). The appropriate magnification was chosen, and the desired image on the screen was selected and saved.

Fourier transformed infrared (FTIR) spectroscopy

Pieces were randomly selected from the samples of African padauk immersed for 3 h and 6 h, as well as from the untreated samples. These were then ground into a wood powder, and the 100-mesh to 120-mesh parts were sieved. A powder sample of 1 g from each wood type was prepared.

The detection was performed using a Fourier transform infrared spectrometer Nicolet 6700 (Thermo Fisher Scientific, Waltham, MA, USA). Potassium bromide (KBr) was prepared by being dried at 120 °C for 48 h and then ground into a powder. The ratio of KBr powder to wood sample powder was in the range 70:1 to 100:1 (KBr 70 mg to 100 mg, sample 1 mg). Samples with uniform mixing were selected and then treated at a pressure of approximately 12 MPa for approximately 1 min.

Thermogravimetric analysis (TGA)

Pieces were randomly selected from the samples of African padauk immersed for 3 h and 6 h. A random selection was also taken from the untreated sample, placed in the oven, and dried. The dry test materials were then ground into wood powder, and the 40-mesh to 60-mesh portions were sieved into test sizes of 2 g.

Using a TGA Q5000 V3.17 Build 265 thermogravimetric analyzer (TA Instruments, New Castle, DE, USA), the samples were subjected to atmospheric pressure and thermogravimetric analysis.

The experimental carrier gas was N_2, with a flow rate of 50 mL/min, and the temperature was increased at a 10 °C/min rate. The ambient temperature (28 °C) was eventually raised to 900 °C. The mass change during the heating process was recorded. During the heating process, the mass fraction and mass change rate of the sample were calculated, and the thermogravimetric (TG) and derivative thermogravimetric (DTG) curves were plotted.

RESULTS AND DISCUSSION

Weight Gain

Figure 1 shows the weight changes in the test materials at different treatment times under the beeswax impregnation conditions. These results showed that the weight gain increased with the increase of wax immersion time, with the amount of beeswax in the wood continuously increasing. The weight gain of test material treated for 6 h was higher than that of the test material treated for 3 h, but the weight gain for the test material between the 3 h to 6 h period was much less than the weight gain between 0 h to 3 h. The weight gain of increase after 3 h was 44.54% of the increase that occurred in the first 3 h, so it could be inferred that at some time after 3 h, impregnation of the test material was essentially complete, and the immersion liquid could not fill the cell pores any further.

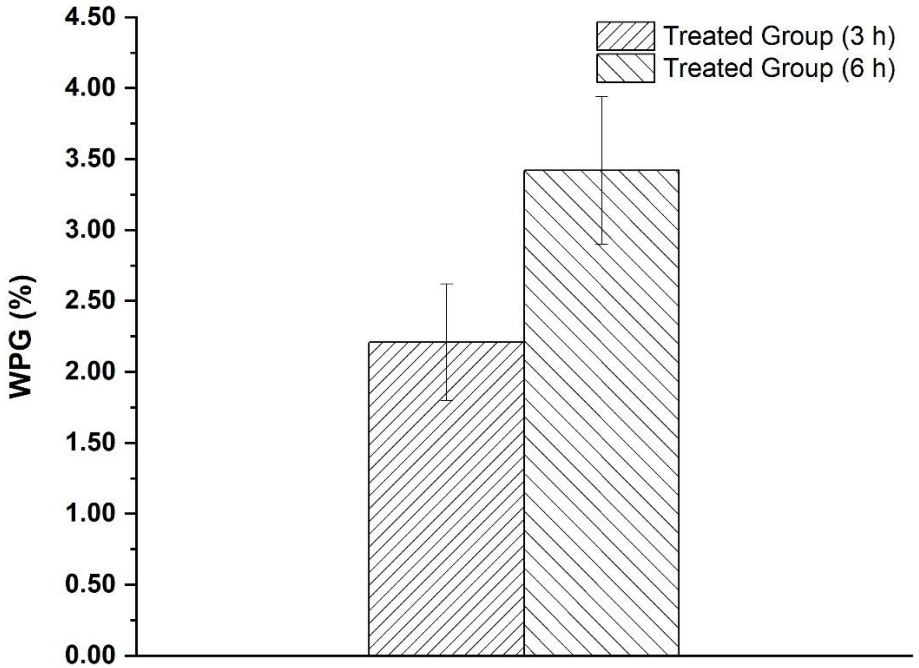

Fig. 1. Weight gain rates of the treated groups

Hygroscopicity Test

Figure 2 shows that the dimensional stability (a_w) of the beeswax-impregnated materials did improve remarkably. The tangential swelling ratio value for the control group was 3.52, whereas the tangential swelling ratio of the sample impregnated with beeswax for 6 h was 1.04, which meant that it decreased 70.5%. The value of the sample impregnated for 3 h was 1.39, which was a decrease of 52.8%. The radial swelling ratio of the control group was 2.31, and the radial swelling ratio of the sample impregnated with beeswax for 6 h was 0.81, which was a decrease of 64.9%. The value of the sample impregnated with beeswax for 3 h was 1.12, which was a decrease of 51.5%. The volumetric swelling ratio of the control group was 6.03, the volumetric swelling ratio of the sample impregnated with beeswax for 6 h was 2.17 (a 64.0% decrease), and the value of the sample impregnated with beeswax for 3 h was 2.54 (a 57.9% decrease). Based on these results, it was obvious that beeswax impregnation noticeably improved the

dimensional stability of wood, and the modification achieved following a 6 h impregnation period was more effective than after the 3 h impregnation period.

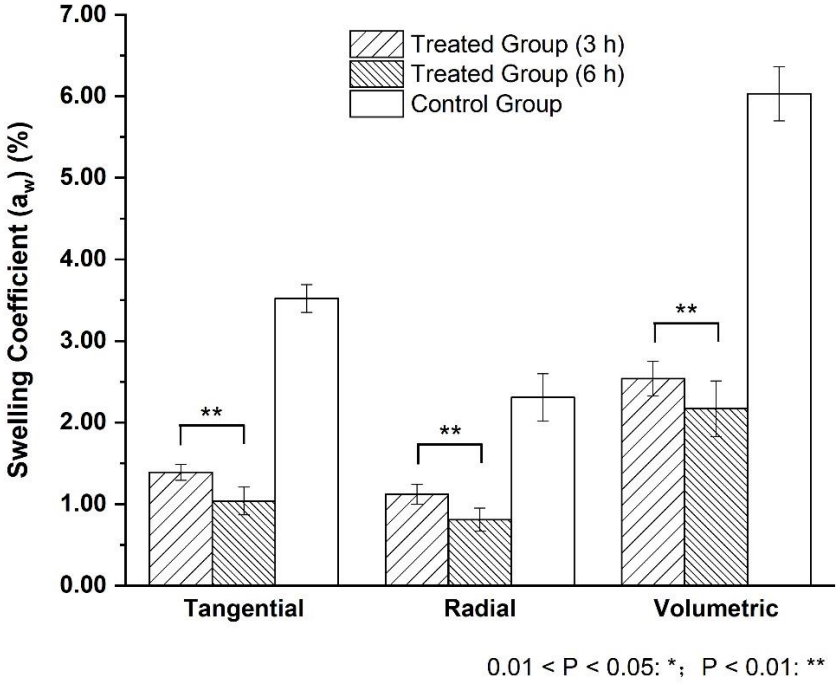

Fig. 2. Effects of beeswax treatment on wood swelling during the moisture absorption process

Water Absorption Test

As Fig. 3 indicates, the swelling coefficient (a_{max}) for African padauk when it has absorbed water to a stable size was similar to what is shown in Fig. 2.

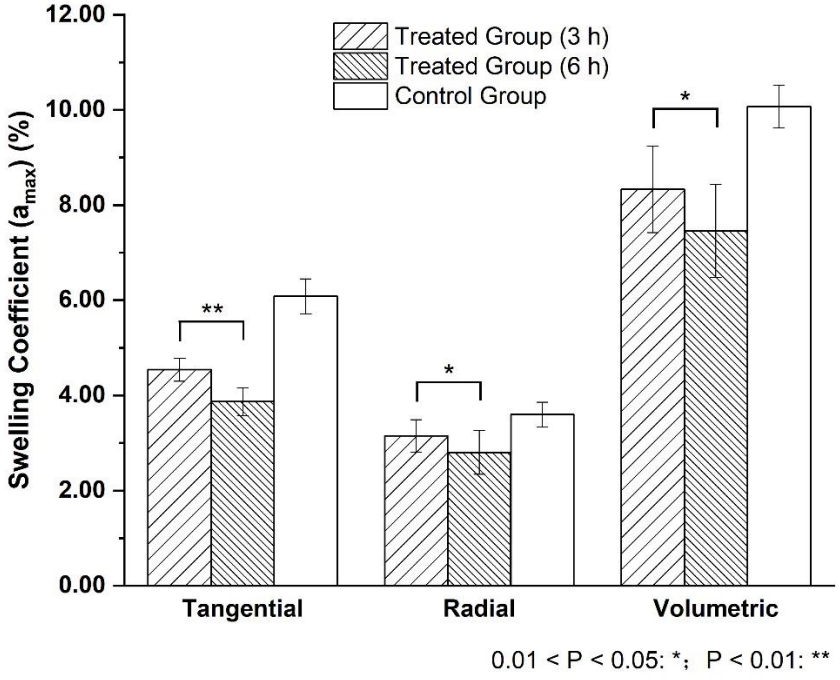

Fig. 3. Effects of beeswax treatment on wood swelling during water absorption

The value of the tangential water absorption control group was 6.08, and the tangential water absorption value of the sample impregnated with beeswax for 6 h was 3.87, a decrease of 36.3%. The value of the sample that experienced a 3 h impregnation period was 4.54. The value of the radial water absorption control group was 3.60, the radial water absorption value of the sample impregnated with beeswax for 6 h was 2.80 (a 22.2% decrease), and the value of the sample impregnated with beeswax for 3 h was 3.15. The water absorption volumetric swelling ratio of the control group was 10.07, the water absorption volume swelling ratio of the sample impregnated with beeswax for 6 h was 7.46 (a 25.9% decrease), and the value of the sample impregnated for 3 h was 8.33. The same conclusion as above also applied here, beeswax impregnation improved the dimensional stability of wood, and the modification achieved after a 6 h impregnation period was superior to that achieved after 3 h.

Contact Angle

Each curve in Fig. 4 illustrates the change in the contact angle between the droplet and the surface of the specimen, as well as the change in contact angle of the control group and the groups that underwent beeswax treatment. Evidently, the beeswax impregnation treatment noticeably improved the hydrophobic properties of the wood surface, with the initial test resulting in the contact angle of wood treated for 3 h increasing 28.7% and that of wood treated for 6 h increasing 26.1%. At 100 s, the contact angle of wood treated for 3 h increased 32.6%, and the contact angle of wood treated for 6 h increased 26.8%. There was no significant difference between the contact angle of the samples impregnated with beeswax for 3 h and 6 h.

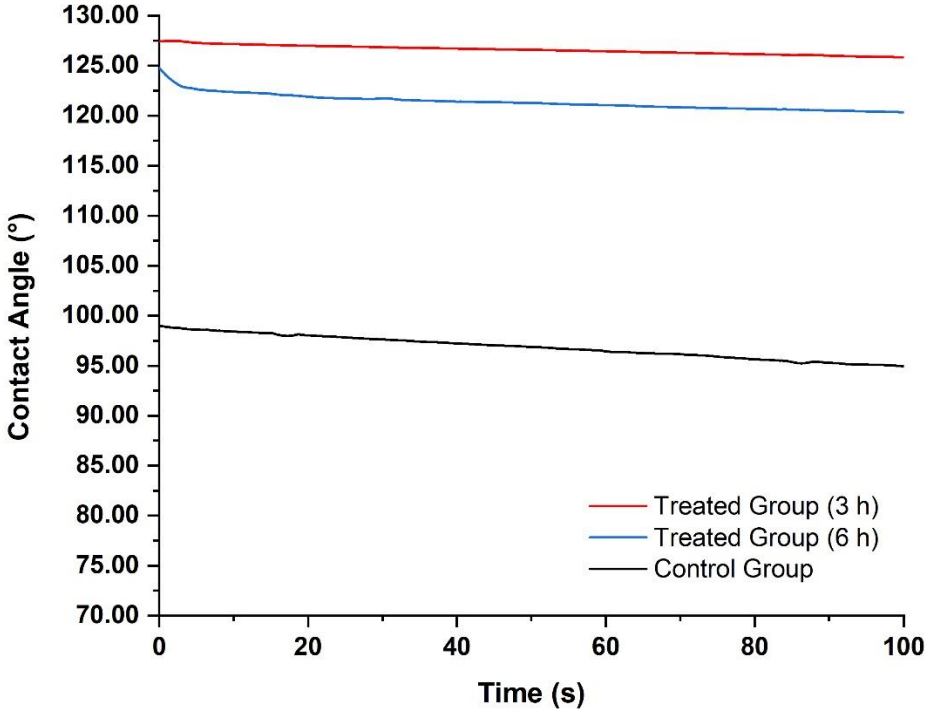

Fig. 4. Contact angle curves for the beeswax-treated material

Table 1 shows the average value, standard deviation, and curve drop of the initial contact angle, as well as the contact angle at 100 s for different impregnation samples. As

this table demonstrates, beeswax-impregnated wood had a higher initial contact angle, as well as a higher average contact angle at 100 s, than those achieved by the control group, which indicated that the hydrophobicity of wood surface was remarkably improved. At the same time, the standard deviation of the control group was noticeably higher than that of the impregnated wood, which indicated that the surface properties of treated specimens were more stable. The curve drop is the ratio of the decrease of the contact angle of 100s and the initial contact Angle. Based on the curve decline, the decline of the control group was also remarkably greater than the decline of the beeswax-impregnated wood.

Table 1. Effects of Beeswax Treatment on the Initial Contact Angle and the Average Contact Angle Value at 100 s

Experimental Conditions	Initial Contact Angle		Contact Angle at 100 s		Curve Drop (%)
	Average Value (°)	Standard Deviation	Average Value (°)	Standard Deviation	
Control Group	99.00	7.33	94.94	7.34	4.46
Treated Group (3 h)	127.43	6.02	125.85	6.14	0.99
Treated Group (6 h)	124.82	7.33	120.36	4.43	6.35

Scanning Electron Microscopy

Figure 5 respectively shows the electron microscope images of the surface layer and the center layer of beeswax-treated wood samples of African Paduak. As can be seen from the figure, beeswax can be observed on both the surface layer and the central layer of the samples after immersion. It shows that beeswax can be impregnated into the inner of African Paduak so as to improve the dimensional stability of African Paduak.

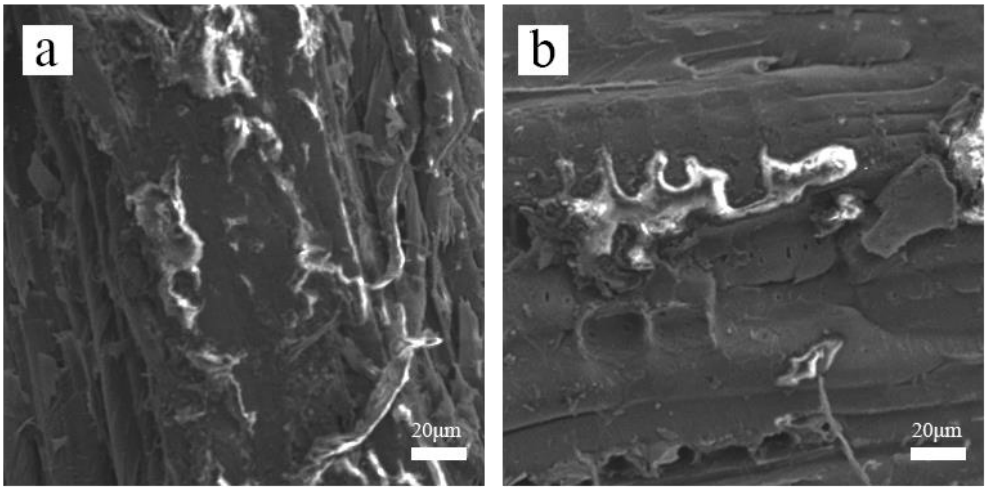

Fig. 5. SEM of African padauk treated by beeswax; a. superficial layer; b. central layer

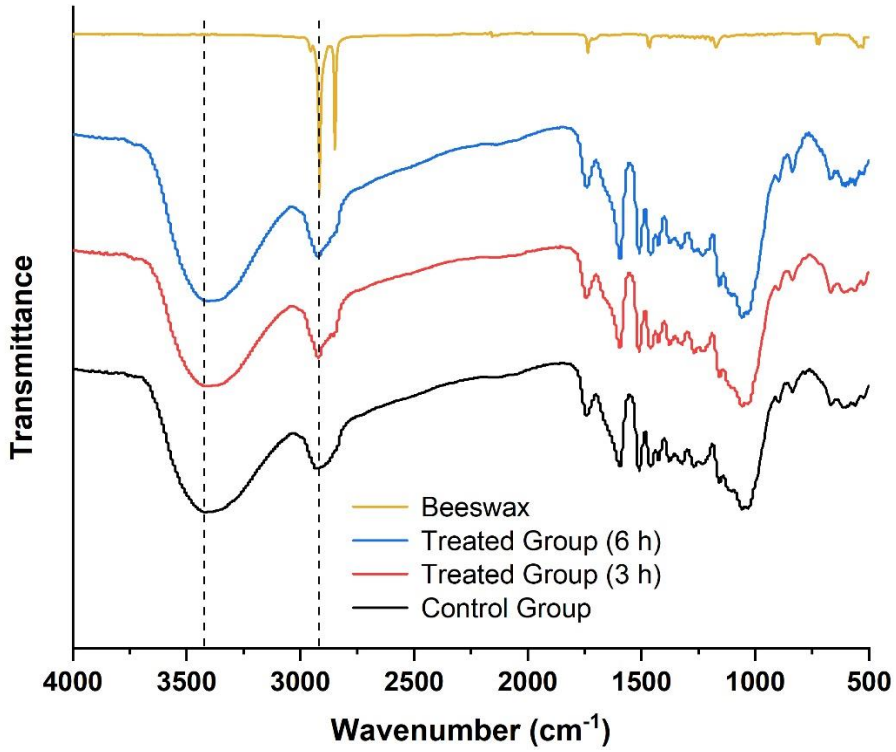

Fig. 6. FTIR spectra of the beeswax and samples treated with beeswax

FTIR Spectra

Figure 6 shows the infrared spectra obtained from the beeswax and the wood using Fourier infrared spectrometric determination of the wavelength between 400 cm^{-1} and 4000 cm^{-1} (Esteves *et al.* 2013; Nami Kartal *et al.* 2013; Qian *et al.* 2019). Beeswax has a significant peak at the wavelength of 2900 cm^{-1}, representing C-H. The chemical composition of the wood cell walls can be identified by wave peaks in the infrared spectrum (Li *et al.* 2015a). The first peak at 3400 cm^{-1} represents -OH, reflecting the moisture absorption of wood. Compared with the control group at a wavelength of 3400 cm^{-1}, the content of -OH in the impregnated group was slightly reduced, but there was little difference with regards to the control group. As mentioned above, esters, fatty acids, and sugars synthesized by higher fatty acids and mono-alcohol are the main components of beeswax. Although there was little difference between the treated and untreated samples at a wavelength of 3400 cm^{-1}, beeswax significantly improved the dimensional stability of African sandalwood.

Thermogravimetric Analysis

The pyrolysis process of wood passes through a series of stages, including free water precipitation, preheating the solution, major pyrolysis, and the decomposition of carbon residue. Free water precipitation in wood occurs at approximately 100 °C. The thermal decomposition of wood cellulose starts at 240 °C and ends at approximately 400 °C, with the thermal decomposition reaction the most intense between 300 °C to 375 °C. The thermal decomposition of wood hemicellulose starts at 145 °C, and the reaction is most intense between 225 °C to 325°C (He *et al.* 2019). Hemicellulose has the lowest cracking temperature and the worst thermal stability among the three main components of wood. The temperature range for thermal decomposition of lignin is wide, with pyrolysis

occurring between 250 °C to 500 °C and the most intense pyrolysis reaction occurring from 310 °C to 420 °C (Poletto *et al.* 2012; Hazarika *et al.* 2014). When heated to 250 °C, the wood began to release gases such as carbon dioxide and carbon monoxide. When the temperature increased to 310 °C, the wood generated a large number of gaseous products, as well as condensable gases such as acetic acid, formaldehyde, and wood tar. When the temperature reached 420 °C, the quantity of steam products decreased. At this point, the reaction was considered to be basically complete.

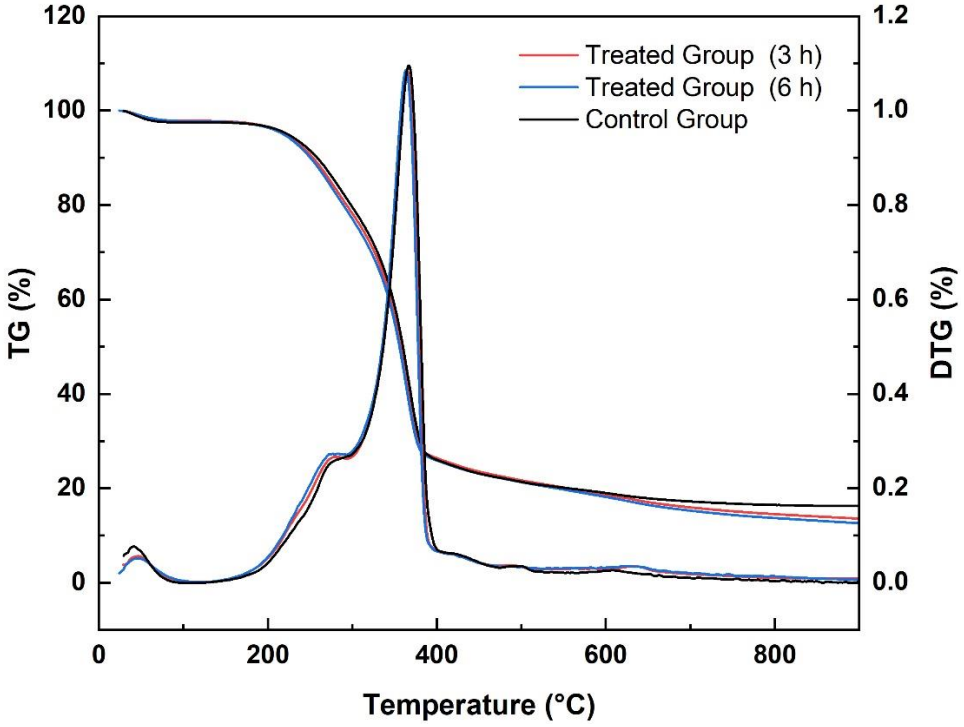

Fig. 7. TG-DTG curves of the samples treated with beeswax

The TG and DTG curves for sandalwood impregnated with beeswax for different lengths of time are shown in Fig. 7. Between the start of the heating process and a temperature of approximately 100 °C, the decrease in mass for timber treated for 6 h was less than that of both timber treated for 3 h and untreated timber. This indicated that the wood treated with beeswax for 6 h had the lowest water absorption rate under air drying conditions, the wood treated for 3 h had the second highest water absorption rate, and the untreated wood had the highest water absorption rate. These results further confirmed that the dimensional stability of sandalwood treated for 6 h, was superior to both wood treated for 3 h and untreated wood.

The maximum pyrolysis rate of the treated and untreated samples was within the range of 330 °C to 380 °C. The TG curve shows that the main pyrolysis processes of material treated for 3 h, material treated for 6 h, and untreated material took place at almost the same time and exhibited similar curves. As the DTG curve indicates, the shoulder peak appeared near 280 °C, which was the most intense stage of hemicellulose reaction. The peak value at around 380 °C aligned with the most intense cellulose pyrolysis phase, followed by the pyrolysis process of lignin. The widths of the zones in which the weight did not change significantly for the three test materials were similar, but the peak value for the material treated for 3 h was lower than that of the material treated for 6 h, as well as

that of the untreated material. The pyrolysis rate of the material treated for 3 h was higher than that of the material treated for 6 h, as well as that of the untreated material. In the latter stage of pyrolysis, the carbon residue rate of the wood treated for 6 h was relatively low, followed by that of wood treated for 3 h, with the untreated wood exhibiting the highest carbon residue rate. The carbon residue rate for the timber treated with beeswax for 6 h was 12.62%, and the timber treated for 3 h and the untreated timber had carbon residue rates of 13.59% and 16.25%, respectively.

When beeswax reaches temperatures of approximately 300 °C, the pyrolysis results in smoke and decomposition into carbon dioxide, acetic acid, and other volatile substances. Therefore, when heated to 900 °C, the beeswax would be completely decomposed and would not exist in the residual carbon composition. As mentioned above, the weight gain rate of timber treated for 6 h was higher than that of timber treated for 3 h. The content of beeswax in the timber treated for 6 h was higher, so the carbon residue rate of timber treated for 6 h was lower than that of timber treated for 3 h and that of untreated timber. Therefore, the beeswax is impregnated into the wood. To some extent, a longer immersion time of wood in beeswax resulted in greater beeswax content, which resulted in improvements for the dimensional stability of wood.

CONCLUSIONS

1. Beeswax impregnation could penetrate into the wood and noticeably improve the dimensional stability of African padauk (*Pterocarpus soyauxii*). The dimensional stability of wood treated for 6 h was superior to that of wood treated for 3 h, and the swelling coefficient (a_w) of wood treated for 6 h decreased 70.5% compared to the untreated wood. Dimensional stability improved as the wax soaking time increased. However, the weight gain rate achieved in the last 3 h of soaking was not as large as that achieved in the first 3 h. Therefore, to improve the dimensional stability of wood and preserve resources, it is important to determine a reasonable and optimal processing time rather than blindly increasing processing times.
2. The surface hydrophobicity of wood was remarkably improved by beeswax impregnation. In the initial test, the contact angle of wood treated for 3 h increased 28.7%, and the contact angle of wood treated for 6 h increased 26.1%. At 100 s, the contact angle of wood treated for 3 h increased 32.6%, and the contact angle of wood treated for 6 h increased 26.8%. Therefore, beeswax impregnation was able to improve the surface properties of wood.
3. The maximum pyrolysis rate of treated and untreated wood was achieved within the range of 330 °C to 380 °C. According to the TG curve, the main pyrolysis processes for the untreated wood, as well as the wood treated for 3 h and 6 h, occurred almost simultaneously and had similar curves. During the later stage of pyrolysis, the carbon residue rate of wood treated for 6 h was relatively low, at 12.62%, followed by that of wood treated for 3 h at 13.59% and that of untreated wood at 16.25%. Because the weight gain rate of wood treated for 6 h was higher than that of wood treated for 3 h, this indicated that the content of beeswax in wood treated for 6 h was higher, which further indicated that beeswax played an important role in improving the dimensional stability of wood.

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