Effects of Vacuum Heat Treatment and Wax Impregnation on the Color of *Pterocarpus macrocarpus* Kurz.

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Pterocarpus macrocarpus Kurz. wood was vacuum heat treated (VHT) at 120, 150, and 180 °C, under a pressure of 13.3 kPa. Half of the VHT specimens at 120 and 150 °C were subjected to wax impregnation (WI) for 48 h at 90 °C under an atmospheric pressure. The effect of VHT and WI on wood color were investigated. The results showed that the VHT at 120 and 150 °C resulted in minor changes in lightness (L^*), green-red chromatic coordinate (a^*), blue-yellow chromatic coordinate (b^*), total color change (ΔE^*), and chroma (C^*). However, the effect of VHT on L^*, a^*, b^*, and C^* at 180 °C became more obvious over the duration. After WI, the L^*, a^*, b^*, and C^* of the VHT wood at moderate temperatures varied noticeably, showing similar behavior with the VHT wood at 180 °C as L^*, b^*, and C^* decreased and ΔE increased. However, a^* increased after WI compared to that of VHT at 180 °C. The wood color of *P. macrocarpus* Kurz. after WI became reddish and blue, and the color deviation decreased. The wood color was closer to the dark mahogany, which facilitates its further application in rosewood furniture and woodwork art.

Keywords: Pterocarpus macrocarpus Kurz.; Vacuum heat treatment; Wax impregnation; Color;

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INTRODUCTION

Pterocarpus macrocarpus Kurz. is a deciduous tree species found mainly in Southeast Asian countries. *P. macrocarpus* is used for its heartwood, which is reddish brown with a clear texture, high density, and large hardness. In addition, *P. macrocarpus* has a special fragrance (Wisittipanich *et al*. 2012). People can obtain visual enjoyment and psychological pleasure from its unique wood properties, such as wood color and textures. Therefore, *P. macrocarpus* is commonly used for high-value rosewood furniture, wood carving, woodwork art, *etc*. However, *P. macrocarpus* wood is difficult-to-dry due to its high density and high content of extractives. The poor drying of *P. macrocarpus* wood may lead to severe checks and deformations of furniture and woodwork art (Liu *et al*. 2019). The checks and deformations of furniture and wood products in service can be prevented using wood modification such as heat treatment (HT) (Gu *et al*. 2019; Zhou *et al*. 2020) and wax impregnation (WI).

Heat treatment is an environmentally friendly wood modification method. Heat treatment can remarkably reduce wood moisture absorption and improve the wood's dimensional stability; however, the wood's mechanical strength is reduced and wood color is darkened after heat treatment (Poncsák *et al*. 2006; Almeida *et al*. 2009; Srinivas and

Pandey 2012; Zhang *et al*. 2020). The main components in wood that affect the wood's color are lignin and extracts. During HT, the chemical changes of hemicellulose and lignin can result in a change in the wood color (Ding *et al*. 2017; Hu *et al*. 2020). The colors of *Pterocarpus macrocarpus* are easy to change due to its richness in extractives (13.8%) (Tomak *et al*. 2011; Chen 2015). During HT, the wood color changes due to an increase of chromogenic groups in lignin, and furthermore changes of extractives (Wang 2015). Additionally, polyphenols (*e.g.*, tannin or flavonoids) have a noticeable effect on wood color, and their thermal oxidation can cause wood discoloration (Dellus *et al*. 1997; Johansson *et al*. 2005; González-Peña and Hale 2009; Hu *et al*. 2012; Sandoval-Torres *et al*. 2012). The time and temperature during HT affect the wood color change, but temperature has a more noticeable effect than the other factors (Kocaefe *et al*. 2008; Veikko and Kärki 2008; Fan *et al*. 2010; Dubey *et al*. 2011). Generally, a higher temperature and a longer length of time can result in dark brown wood (Gonzalez-Pena *et al*. 2009; Zanucio *et al*. 2014).

After HT at a higher temperature, the wood color becomes dark, the overall lightness decreases, and the wood color becomes stable (Matsuo *et al*. 2014). Some heat-treated wood color deepens to a grayish brown, which decreases the aesthetics of the wood, while some colors change to a reddish brown, which increases the aesthetics. The color change during HT affects the overall visual effect of the wood and its application in wood products (Bekhta and Niemz 2003; Srinivas and Pandey 2012). Additionally, the color of the wood after WI can also change. The color change of wood after WI is different from that after HT because wax contains low-melting-point hydrocarbon, which may affect the gloss and color of wood. Although there have been many studies on how HT affects wood color, few are related to rosewood and the wood color of rosewood after a HT + WI treatment. In this study, the objective was to investigate the effects of HT and the combination of HT + WI on the color change of *P. macrocarpus* hoping to provide useful references for further studies and developments of rosewood furniture and woodwork art.

EXPERIMENTAL

Materials

The lumber pieces of *P. macrocarpus* Kurz. were collected from Xianyou Degoo Furniture Co., Ltd. (Xianyou, China). The initial moisture content (MC) of the lumber was approximately 10%. The lumber pieces were used to prepare specimens of 20 (T) × 20 (R) × 300 (L) mm for the VHT-and VHT + WI tests in the lab of Nanjing Forestry University. Specimens without knots or other drying defects were randomly divided into 15 treated groups and 1 control group, with each group having 2 specimens. Microcrystalline wax (Degoo, Xianyou, China) was used for the impregnation test. Microcrystalline wax has a melting point of 60 °C, a density of 0.8 to 0.92 g/mL, and a kinematic viscosity of 9.2 to 25.0 mm^2/s (99 °C).

Equipment

The main device was a vacuum heating chamber (HJ-ZK60; Dongguan Hengjun Instruments Co., Ltd., Dongguan, China). Other devices were an electric heating oven (DHG-905386-III; Shanghai Cimo Medical Instrument Co., Ltd., Shanghai, China), an electronic balance (JA21002; Shanghai Liangping Instrument and Meter Co., Ltd., 1200 g/1 mg), and a portable sphere spectrophotometer (SP60; X-Rite, Grand Rapids, MI, USA) for the color data measurement.

Methods

Prior to the VHT and WI tests, all specimens were dried in an electric heating oven to absolute dry conditions (*i.e.*, 0% MC). Then, three circles with a 10-mm diameter were marked in each specimen. The measuring locations were in the middle and close to the ends of a specimen. The color changes were measured using the CIELAB method, which quantifies color by a three-axes system: L*, a*, and b* stand for the chromaticity of an object and can also stand for the chroma coordinates (Mononen *et al*. 2002). The color data of the L*, a*, and b* before the tests were measured using a spherical spectrophotometer (SP60; X-Rite, Grand Rapids, MI, USA). Then, specimens were subjected to vacuum heat treatment according to the arrangement in Table 1. A previous study showed that VHT has certain advantages for wood modification (Chandelier *et al*. 2013). Therefore, the wood was heat treated in a vacuum heating chamber at 13.4 kPa in this study. For tests No. 11 to No. 16, specimens were subjected to wax impregnation after they had been heat treated under vacuum at different temperatures and times. After VHT, the color data at the marked points were measured again when the specimens were cooled to room temperature, and each point was measured at least three times to ensure the accuracy of the color data. After VHT + WI, the waxes on the surfaces were removed, and the color data was measured at the marked points following the steps above.

Table 1. Vacuum Heat Treatment and Wax Impregnation Conditions

Treatment	Test No.	Temperature (°C)	Time (h)	Temperature (°C)	Time (h)	Pressure (kPa)
Control	1	-	-	-	-	101.3
VHT	2	120	2	-	-	13.3
	3		4	-	-	13.3
	4		6	-	-	13.3
	5	150	2	-	-	13.3
	6		4	-	-	13.3
	7		6	-	-	13.3
	8	180	2	-	-	13.3
	9		4	-	-	13.3
	10		6	-	-	13.3
VHT + WI	11	120	2	90	48	101.3
	12		4	90	48	101.3
	13		6	90	48	101.3
	14	150	2	90	48	101.3
	15		4	90	48	101.3
	16		6	90	48	101.3

The cells containing '-' mean there was no treatment

The corresponding variations of Δa^*, Δb^*, and ΔL^* were calculated using Eqs. (1), (2), and (3), the total color difference ΔE^* after VHT and VHT + WI was calculated using Eq. (4). and the C* (chroma) was calculated using Eq. (5),

$$\Delta a^* = a^* - {a_0}^* \quad (1)$$

$$\Delta b^* = b^* - {b_0}^* \tag{2}$$

$$\Delta L^* = L^* - {L_0}^* \tag{3}$$

$$\Delta E^* = (\Delta a^{*2} + \Delta b^{*2} + \Delta L^{*2})^{\frac{1}{2}} \tag{4}$$

$$C^* = \left(a^{*2} + b^{*2}\right)^{\frac{1}{2}} \tag{5}$$

where Δa^*, Δb^*, and ΔL^*denote the changes in value before and after treatment of the lightness, green-red chromatic coordinate, and blue-yellow chromatic coordinate, respectively. The L^*, a^*, and b^* and L_0^*, b_0^*, and a_0^* are the color data before and after VHT and VHT + WI treatment, respectively.

RESULTS AND DISCUSSION

Visual Color Changes

The color changes after VHT and VHT + WI are shown in Fig. 1. Compared to the color of the control specimens, the wood color varied after both treatments. The color of wood after 120 °C and 150 °C VHT changed slightly, and there were no obvious differences in the visual observation. However, the wood color became dark after 180 ° C VHT and 120 °C VHT + WI and 150 °C VHT + WI treatments. For 180 °C VHT, 120 °C VHT + WI, and 150 °C VHT + WI treatments, the wood color darkened with the severity of treatments, but there was no obvious color difference. In contrast to the 120 °C and 150 °C VHT treatments, the wood color changed after wax impregnation, indicating that the color change was mainly attributable to wax impregnation. Furthermore, the wood color mostly turns brownish after VHT, while it presents a stable reddish color after WI.

Fig. 1. Color change of the wood after VHT and VHT + WI treatments (numbers denote the test number in Table 1)

Lightness Changes (*L**)

Changes in the wood lightness can remarkably affect the wood color. Therefore, the change of wood color was evaluated *via* the variation of ΔL^* after VHT and WI treatments. Table 2 shows the changes of L^* before and after wood treatment. There were no obvious changes in L^* for the 120 °C VHT as the range of $\Delta L^*$was from 0.19 to -1.74. However, for the VHT at 150 °C and 180 °C, L^* decreased due to increased temperature and times, especially for the 180 °C VHT. Temperature had a greater effect on L^* than time did for all VHT specimens. This result agrees with the study of Srinivas and Pandey (2012), who found that the color changes with the severity of the HT of silver oak wood in a vacuum. The largest change in the color showed that the lightness of the wood L^* darkened. Decrease of L^* shows that many wood components that absorb visible light were formed during HT. The darkening of wood results from the degradation reactions of hemicellulose, which forms low-molecular-weight sugars and oxidation products (Esteves *et al*. 2008; Kamperidou and Barmpoutis 2015). In addition, wood darkening can be influenced by the increase of extractives and the relative content of lignin during the HT. Compared with the largest change in L^* during the 180 °C VHT, L^* decreased and the wood darkened severely after wax impregnation. Although the L^* of the wood after 150 °C VHT decreased compared with the 120 °C VHT, the L^* of them did not change much after WI, and there was no obvious difference in L^* for all WI specimens. The apparent wood darkening after WI was probably due to an increase in the extractives of tropical wood species, which could condense and form by-products during WI, consequently contributing more to color degradation (Zhang *et al*. 2019).

Table 2. The ΔL^* of Wood Before and After Treatment

Treatment	Time (h)		
	2	4	6
120 °C VHT	0.19	-1.74	-0.81
150 °C VHT	-2.43	-2.77	-3.10
180 °C VHT	-7.65	-5.99	-10.51
120 °C VHT + WI	-13.25	-13.76	-14.50
150 °C VHT + WI	-14.54	-14.84	-14.94

The Changes of *a** and *b**

Changes in the a^* coordinate of the wood before and after treatment are presented in Table 3. The change in a^* coordinate was small for the VHT at 120 °C and 150 °C, but decreased noticeably at 180 °C, which indicated that the wood color changed directions to a greener color after 180 °C VHT, and varied with increased treating time. However, the a^* coordinate of the wood increased obviously after WI, changing direction toward a redder color. This indicates that the wood color became red after WI treatment. There were no apparent differences in the a^* coordinate between 120 °C VHT + WI and 150 °C VHT + WI, both in the same duration. However, a different treating duration presented a different a^* coordinate after wax impregnation, showing that the VHT affected the subsequent WI, which resulted in an increased a^* coordinate. The a^* coordinate decreased with longer exposure at a higher temperature (180 °C) HT (Table 3). These findings agree with a previous study (Brischke *et al*. 2007). However, the a^* coordinate increased after wax impregnation. This may be related to more condensation, degradation and oxidation of certain wood elements during wax impregnation (Chen *et al*. 2012). Additionally, the impregnated wax contributed to the increase in the a^* coordinate.

Table 3. The Δa^* of Wood Before and After Treatment

Treatment	Time (h)		
	2	4	6
120 °C VHT	-0.34	0.79	-0.54
150 °C VHT	0.91	1.61	-0.45
180 °C VHT	-5.26	-12.18	-14.77
120 °C VHT + WI	3.02	7.88	5.09
150 °C VHT + WI	3.50	6.59	5.41

Table 4 shows the changes in the b^* coordinate of wood before and after treatment. Similar rules were found in the yellow-blue component represented by the b^* coordinate, as that few changes occurred during VHT at 120 °C and 150 °C. The *b** coordinate of wood decreased obviously with treating duration at 180 °C VHT. The reduced *b** coordinate of wood indicated that the color changed toward a blue color. In contrast to the *a** coordinate, the *b** coordinate of VHT wood after WI decreased, indicating that the wood color also changed towards a blue color after WI. In addition, there were no obvious differences in the *b** coordinate between 120 °C VHT + WI and 150 °C VHT + WI, except the 2 h duration, suggesting that the effect of WI and of VHT on changes in the *b** coordinate wood was virtually the same.

Table 4. The Δ***b**** of Wood Before and After Treatment

Treatment	Time (h)		
	2	4	6
120 °C VHT	-0.39	0.97	-0.50
150 °C VHT	-0.50	-1.04	1.47
180 °C VHT	-12.17	-9.61	-12.74
120 °C VHT + WI	-10.99	-13.43	-13.13
150 °C VHT + WI	-13.42	-13.33	-12.95

Total Color Change (Δ*E**)

The total color changes (Δ*E**) are shown in Table 5. There were no obvious differences at 120 °C VHT and 150 °C VHT. Color changes cannot be distinguished with the naked eye when the Δ*E** is smaller than 3 National Bureau of Standard (NBS). The Δ*E** value in Table 5 agreed with the color change in the photos presented in Fig. 1. However, the Δ*E** increased at 180 °C VHT, and the effect of HT duration was remarkable on the Δ*E** of the wood. The maximum improvement of Δ*E** was the 6 h heat treatment at 180 °C under vacuum. The Δ*E** of VHT wood after WI also increased obviously. Compared with 180 °C VHT, the Δ*E** improvement of VHT wood after WI was greater than that of 2 h and 4 h of VHT. However, the 6 h at 180 °C VHT had the greatest Δ*E**. Both 180 °C VHT and WI influenced the wood's color obviously. Wood after WI and 180 °C VHT became dark, while the color deviation became small. The wood color of *P. macrocarpus* Kurz. after WI became reddish and blue, which is closer to dark mahogany and facilitates its further application in rosewood furniture, *etc*.

Table 5. The Δ*E** of Wood Before and After Treatment

Treatment	Time (h)		
	2	4	6
120 °C VHT	0.55	2.15	1.09
150 °C VHT	2.64	3.37	3.46
180 °C VHT	15.30	16.63	22.16
120 °C VHT + WI	17.48	20.78	20.21
150 °C VHT + WI	20.09	21.01	18.74

The Changes in Chroma (*C**)

The changes in chroma (*C**) in the wood before and after treatment are shown in Table 6. The value of *C** depended on the *a** and *b** of the wood. The lowered chroma indicated dimmer visual effects in the wood. The chroma of the wood varied slightly after 120 °C VHT and 150 °C VHT; however, the chroma decreased greatly with the treating duration for VHT at 180 °C. In addition, the chroma after WI decreased sharply for VHT wood, especially for 120 °C VHT wood, which decreased remarkably with the treatment duration. However, for 150 °C VHT wood, the decreased chroma was not obvious. These results indicated that effect caused by WI on wood chroma was influenced by VHT. The effects of 180 °C VHT and WI on changes of *C** of wood were both severe.

Table 6. Change of *C** Before and After Wood Treatment

Treatment	Time (h)	*C**		
		Before	After	Rate of Change (%)
120 °C VHT	2	32.91	32.24	-2.05
	4	31.34	32.56	3.89
	6	28.04	27.28	-2.70
150 °C VHT	2	31.56	30.27	-4.09
	4	30.44	30.94	1.65
	6	28.02	28.84	2.95
180 °C VHT	2	39.59	28.20	-28.79
	4	38.24	23.35	-38.95
	6	38.90	20.02	-48.54
120 °C VHT+WI	2	35.12	23.28	-33.71
	4	35.75	14.83	-58.52
	6	39.23	10.11	-74.23
150 °C VHT+WI	2	37.44	26.31	-29.72
	4	38.92	25.07	-35.57
	6	37.62	26.68	-29.06

CONCLUSIONS

1. The lightness of *L** of *P. macrocarpus* Kurz wood changed little after exposure to the moderate temperature of 120 °C in vacuum heat treatment (VHT), which decreased obviously with increased temperature and duration at 150 °C and 180 °C VHT. The effect of temperature on *L** was greater than time for all VHT specimens. Compared with the largest change of *L** at 180 °C VHT, *L** decreased greatly, and the wood darkened severely after WI. Although *L** of wood after 150 °C VHT decreased compared with 120 °C VHT, they changed few after WI, there was no obvious difference in *L** for all WI specimens.
2. The change in *a** coordinate was small for the VHT wood at 120 °C and 150 °C, whereas it decreased remarkable at 180 °C. However, the *a** coordinate of wood increased obviously after WI showed a reverse direction toward red color. There were no apparent differences in *a** coordinate between 120 °C VHT and 150 °C VHT wood for the same HT duration after WI. However, for different HT duration, VHT wood presented different *a** coordinate after WI, showing the VHT affected the subsequent WI and resulted in increased *a** coordinate.
3. The *b** coordinate of wood changed minimally after VHT at 120 °C and 150 °C. The *b** coordinate of wood decreased obviously with HT duration at 180 °C VHT. The

reduced b^* coordinate indicated color changed toward a blue color. In contrast to the a^* coordinate, the b^* coordinate of VHT wood after WI decreased, indicating wood color changed also towards a blue color after wax impregnation. In addition, there were no obvious differences of b^* coordinate between 120 °C VHT + WI and 150 °C VHT + WI, this suggested the effect of WI on changing the b^* coordinate of VHT wood was same.

4. No obvious total color changes ($\triangle E^*$) could be found for wood after 120 °C VHT and 150 °C VHT. The $\triangle E^*$ decreased dramatically at 180 °C VHT, but the effect of HT duration was not obvious on the $\triangle E^*$ of wood. The $\triangle E^*$ of VHT wood after WI also decreased obviously, and there were no differences of $\triangle E^*$ between 180 °C VHT and VHT + WI. Wood color of *P. macrocarpus* Kurz after WI became reddish and blue, which is more close to the dark mahogany and facilitates its further application in rosewood furniture and woodwork art.
5. The C^* varied little in the wood after 120 ℃ VHT and 150 ℃ VHT; however, the C^* of wood decreased greatly with HT duration at 180 ℃ VHT. The C^* also decreased sharply for the VHT wood after WI, and the variation depends on HT temperatures and duration, this indicates that VHT did affect the treatment of WI to wood. The effects of 180 ℃ VHT and WI on changes of C^* of wood was almost same.

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