COLOR CHANGE OF CHINESE FIR THROUGH STEAM-HEAT TREATMENT

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Dark brown wood color is a current trend and widely appreciated by consumers in the furniture and decoration markets. Heat treatment is one of the most effective methods to darken wood's appearance. The influence of steam-heat treatment on color change of Chinese fir (*Cunninghamia lanceolata* (Lamb.) Hook.) was investigated within the temperature range from 170 to 230 ^{o}C and time from 1 to 5 hours in an air-tight chamber within an atmosphere comprising less than 2 percent oxygen. Saturated steam was used as a heating medium and a shielding gas. The results showed that the chroma difference ($\triangle C^*$) decreased gradually, while the color difference ($\triangle E^*$) and hue difference ($\triangle H^*$) increased with an increase in temperature and length of time. An analysis of variance (ANOVA) and a multi-comparison analysis revealed that the treatment temperature plays a more important role in darkening wood color during the process of steam-heat treatment in comparison with the treatment time. The results suggest that a more desirable wood color can be achieved with the technology of steam-heat treatment.

Keywords: Cunninghamia lanceolata (Lamb.) Hook; Steam-heat treatment; Color difference

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INTRODUCTION

Steam-heat treatment has been known for a long time as one of the most effective methods to improve the dimensional stability, decay resistance, and durability of wood, while simultaneously darkening the wood color. In terms of the mechanism for the dark color development, the properties and quantities of major chemicals and extractive compositions in wood are modified during the steam-heat treatment. Color is usually of considerable importance in wood species when it is used for furniture and decorative purposes. Regarding the color responses during the heat treatment, Bekhta and Niemz (2003) reported that treatment temperature and time were more important than relative humidity, and in general the darkening accelerated when treatment temperature exceeded approximately 200^{o}C. Morita and Yamazumi (1987) showed that the color difference was increased with an increase in treatment time and pressure. Brauner and Conway (1964) reported that temperature and wood moisture content were found to affect color changes significantly during steaming and noted that the purplish brown heartwood of black walnut changed to a chocolate brown within 3 to 5 hours at 100 ^{o}C.

Wood is composed of cellulose, lignins, hemicelluloses, and a small amount of extraneous materials contained in a cellular structure, which includes chromophoric groups, such as the ethylene groups, carbonyl groups, benzene rings, orthoquinone, biaromatic rings, and paraquinone *etc*., as well as auxochromic groups, such as the hydroxyl groups, carboxyl groups, amino groups, and ether groups. Among them, coniferyl groups in lignins and oxidation of lignins are major reasons for generating color (Ayadi *et al.* 2003; Dennis and Morén 2006). In addition, however, Bourgois *et al.* (1991) indicated that the decrease in lightness and the increase in the color difference of wood under heat treatment at 240 to 310 ^{o}C are caused by a decrease in hemicellulose content, especially pentosans. Wiberg (1996) found that drying time and drying temperature level affect the value of lightness more than any other color properties. Furthermore, some chromophoric materials in extractives, such as tannic acid, resin, pigment, and others are also responsible for the color change in wood (Sundqvist and Morén 2002). Tolvaj *et al*. (2010) reported that the color change of wet wood is faster than that of dried wood material at the beginning of the steaming, but once temperature exceeds 95 ^{o}C the color change of dry and wet black locust samples are similar; their experiments refuted the idea that the robinetin was a main factor to generate brown color during steaming. Varga and van der Zee (2008) found that the result of steaming depends highly on the wood species. For example, steaming is a suitable method of color homogenization and colorization for black locust. They concluded that steaming time is more important than temperature, while with respect to color change both time and temperature have the same significance based on experiment data of four wood species, *Robinia pseudoacacia* L., *Quercus robur* L., *Intsia bijuga*, and *Hymenolobium petraeum*. Tolvaj and Molnár (2006) reported that the wood color change was faster and less time was needed for homogenization when temperature was rising. They found that the optimum homogenization time was 12 hours at 80 to 95 ^{o}C temperature range and 6 hours at 110 ^{o}C for Turkey oak, and 18 hours at any temperature for beech.

The use of plantation-grown Chinese fir and other fast growing species can be increased if some shortcomings in the wood, such as light color, lower durability, and dimensional instability, *etc*., can be modified by certain methods. A number of studies regarding the effect of heat treatment on color change of wood have been completed (Wiberg 1996; Bekhta and Niemz 2003; Mitsui and Tolvaj 2005). However, studies providing simultaneous analysis of the influence of temperature and time on Chinese fir wood color change were not found in the literature. In order to investigate the possibility of controlling color changes in wood during steam-heat treatment, this study was aimed at the influence of varying process parameters (temperature/time) on the color change in wood. A desired wood of darker color can be obtained by using different treatment temperatures and times through steam-heat treatment.

MATERIALS AND METHODS

Materials

Fifteen Chinese fir (*Cunninghamia lanceolata* (Lamb.) Hook) trees were selected from a planted forest in Hunan province, China. All specimens sized 500 × 50 × 25 mm

(Longitudinal × Radial× Tangential) were cut at the position of annual ring ranging from 18 to 22 for heartwood and from 27 to 34 for sapwood, respectively.

Methods and Treatment Parameters

The Random Complete Block Design (RCBD) method was used to arrange each experimental unit in this study to avoid the variations from different trees. The steam-heat treatments were performed at 170, 185, 200, 215, and 230 °C for 1, 2, 3, 4, and 5 hours in an airtight chamber with an atmosphere comprising less than 2% oxygen. Using a high frequency-vacuum dryer, the heartwood and sapwood boards were dried to a moisture content (MC) of 8% prior to the steam-heat treatments. Around 4% MC was obtained after the steam-heat treatments. Saturated steam with temperature of 152 °C was used as a heating medium and a shielding gas. An electric heater was used to reach set temperature in whole thermal treatment. There were 15 replications of each specimen, and three locations on each specimen were chosen as color measure points. The color difference values of ΔC^*, ΔE^*, and ΔH^* were determined respectively before and after the treatment. Then the values of color difference were calculated based on the relative formulae in the CIE (1976) $L^*a^*b^*$ color space system. The arithmetical average of each color measure point was calculated from the 15 replications.

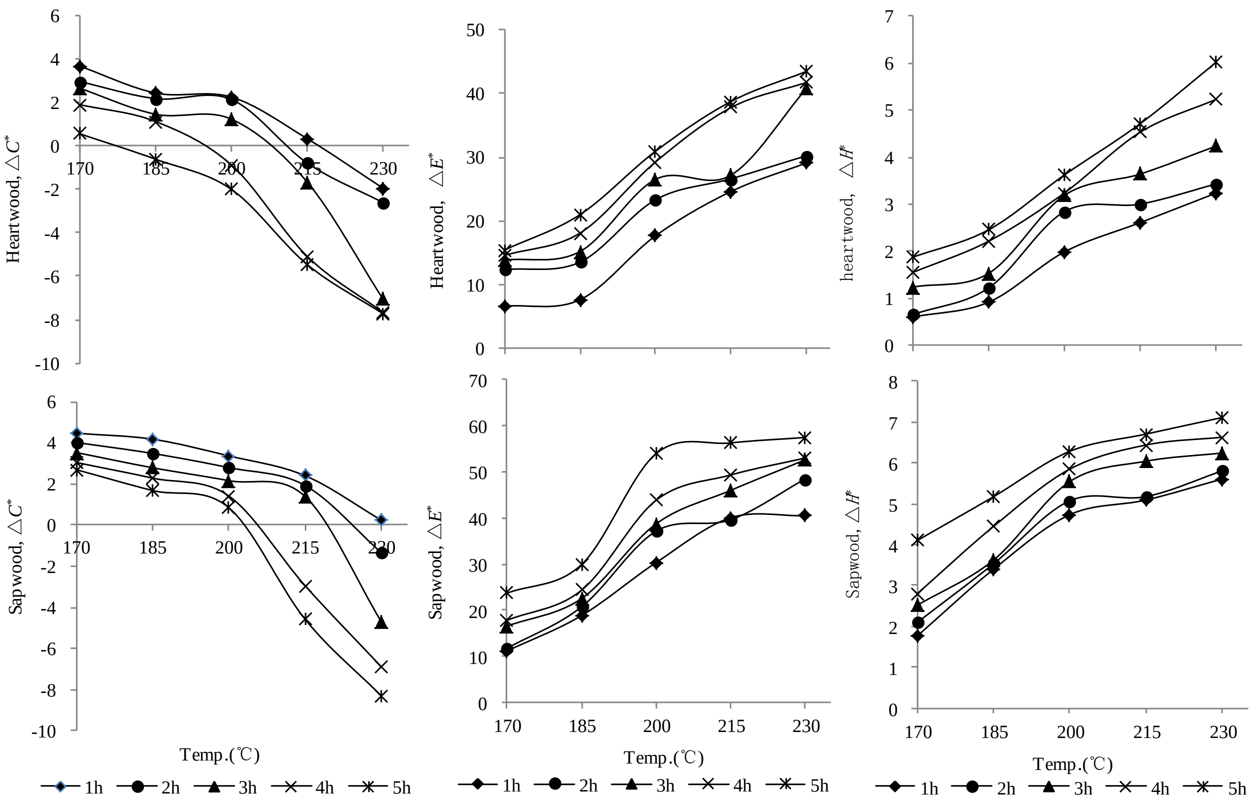

Fig. 1. Changes in color difference of steam-heat treated wood under different treatment temperatures

Color Measurement

Measurements of the color coordinates were performed with a Minolta CR-300 spectrocolorimeter equipped with an integrating sphere according to the CIE $L^*a^*b^*$ system (Robertson, 1977). The L^* axis represents the lightness, and it varies from 100 (white) to zero (black), whereas a^* and b^* are the chromaticity coordinates. In the CIE $L^*a^*b^*$ diagram a positive a^* is the red direction, negative a^* is the green, positive b^* is the yellow, and negative b^* is the blue. C^* means chroma and $\triangle C^*$ is chroma difference. A positive value of $\triangle C^*$ means more vivid, while a negative value means more dull compared with the original color. The parameter $\triangle E^*$ means total color difference value. $\triangle H^*$ is hue difference, which implies the degree of color change far away from original color. These values then were used to calculate the color difference values of $\triangle C^*$, $\triangle E^*$, and $\triangle H^*$ according to the following equations,

$$C^* = [(a^*)^2 + (b^*)^2]^{1/2} \tag{1}$$

$$\triangle L^* = L^* - L^*_s \tag{2}$$

$$\triangle a^* = a^* - a^*_s \tag{3}$$

$$\triangle b^* = b^* - b^*_s \tag{4}$$

$$\triangle C^* = C^* - C^*_s \tag{5}$$

$$\triangle E^* = [(\triangle L^*)^2 + (\triangle a^*)^2 + (\triangle b^*)^2]^{1/2} \tag{6}$$

$$\triangle H^* = [(\triangle E^*)^2 - (\triangle L^*)^2 - (\triangle C^*)^2]^{1/2} \tag{7}$$

where, a^*, b^*, and L^* refer to steam-heat treated wood specimen; a^*_s, b^*_s, and L^*_s refer to the untreated wood specimen.

Method for Determining Chemical Components Contents in Wood

The holocellulose, α-cellulose, and lignins contents in untreated and steam-heat treated wood were determined according to Chinese standards GB/T 2677.10-1995 Fibrous raw material - Determination of holocellulose, GB/T 744-1989 Pulps – Determination of α-cellulose, and GB/T 2677.8-94 Fibrous raw material – Determination of acid-insoluble lignin, respectively.

RESULTS AND ANALYSIS

In general, before treatment, the color of heartwood was found to be darker than that of sapwood. The color of both heartwood and sapwood gradually changed from original light color to brown and even dark brown with an increase in treatment temperature and time during the process of steam-heat treatment. $\triangle C^*$ decreased while

$\triangle E^*$ and $\triangle H^*$ increased during the steam-heat treatment. The treatment temperature had a stronger effect on wood color than treatment time.

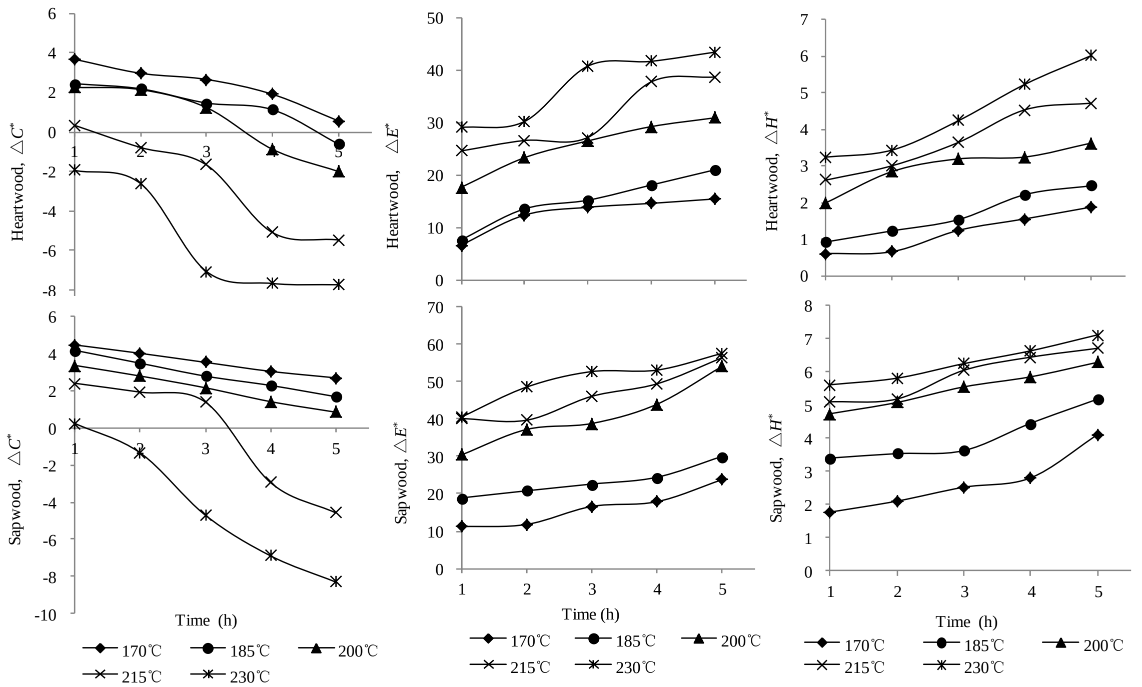

Fig. 2. Changes in color difference of steam-heat treated wood under different treatment times

The Effect of Treatment Temperature on Wood Color

The effect of temperature on $\triangle C^*$, $\triangle E^*$, and $\triangle H^*$ is shown in Fig. 1(a), 1(b), and 1(c), respectively. The $\triangle C^*$ values of heartwood and sapwood decreased, respectively, while the values of $\triangle E^*$ and $\triangle H^*$ increased with an increase in the treatment temperature and the length of time. When the temperatures increased from 170 ^{o}C to 200 ^{o}C, the value of $\triangle C^*$ decreased slightly. Once the temperatures were over 200 ^{o}C, however, the value of $\triangle C^*$ decreased quickly. This result is in a good agreement with the conclusion by Bekhta and Niemz (2003), who found that color changes were drastic after heat treatment at 200 ^{o}C. This implies that 200 ^{o}C is a significant critical point to darken the wood during the steam-heat treatment.

Increases in $\triangle E^*$ and $\triangle H^*$ were observed with the increase in temperature and time. In particular, a rapid increase in $\triangle E^*$ was shown at the temperatures from 185 ^{o}C to 230 ^{o}C for heartwood. However, a slow increase in $\triangle E^*$ was also shown at temperatures ranging from 200 ^{o}C to 230 ^{o}C for sapwood. The value of $\triangle H^*$ increased quickly for sapwood at temperatures ranging from 170 ^{o}C to 200 ^{o}C but more slowly from 200 ^{o}C to 230 ^{o}C. But for heartwood, the value of $\triangle H^*$ increased approximately evenly over the whole process.

Table 1. Repeated Two-Way Analysis of Variance (ANOVA) for Color Change in Steam-Heat Treated Wood in Random Complete Block Design

Items	Source	SS		DF		MS		*F*		$F_{0.01}$		Significance	
		HW	SW	HW	SW	HW	SW	HW	SW	HW	SW	HW	SW
$\triangle C^*$	Temp.	3030	2998	4	4	757	750	1385	1041	3	3	* *	* *
	Time	1044	1046	4	4	261	261	477	363	3	3	* *	* *
	Interact	246	494	16	16	15	31	28	43	2	2	* *	* *
	Total Error	191	252	350	350	1	1						
$\triangle E^*$	Temp.	32397	66675	4	4	8099	16669	1601	945	3	3	* *	* *
	Time	8148	11315	4	4	2037	2829	403	160	3	3	* *	* *
	Interact	1297	1321	16	16	81	83	16	5	2	2	* *	* *
	Total Error	1771	6171	350	350	5	18						
$\triangle H^*$	Temp.	554	676	4	4	139	169	533	254	3	3	* *	* *
	Time	178	150	4	4	45	37	171	56	3	3	* *	* *
	Interact	24	10	16	16	2	1	6	1	2	2	* *	
	Total Error	91	233	350	350	0	1						

Note: α=0.01 "*" means significant at 0.05, "**" means significant at 0.01.

The Effect of Treatment Times on Wood Color

The effect of treatment time on $\triangle C^*$, $\triangle E^*$, and $\triangle H^*$ is shown in Fig. 2(a), 2(b), and 2(c), respectively. The values of $\triangle C^*$ in heartwood decreased significantly at the temperature of 215 ^{o}C from the 3rd to 4th hour and at the temperature of 230 ^{o}C from 2 to 3 hours then became stable, while the value of $\triangle C^*$ in sapwood appeared similar to that in heartwood, but the decrease trend still went down quickly after 215℃ for 4h and 230℃ for 3h. This result indicated that an increase in treatment temperature during the process of steam-heat treatment can bring about a reduction in the treatment time in order to obtain a desired color. On the other hand, the value of $\triangle E^*$ in heartwood increased quickly at 215 ^{o}C from the 3rd to 4th hour and at 230 ^{o}C from the 2nd to 3rd hours, then became stable. However a similar phenomenon was not found in sapwood. This implied that the response system to high temperature is different between heartwood and sapwood. Regarding the value of $\triangle H^*$, it increased quickly only at 230℃ from 2 to 5 hours in heartwood and at 170 ^{o}C from 4 to 5 hours in sapwood, while it increased gradually in other treatment combinations. This result showed that heartwood has a distinct response system to high temperature which is different from that of sapwood.

Table 2. Multi-Comparison of Parameters on Steam-Heat Treatment Processes

Species	Factor	ΔC^* Level	ΔC^* Mean	ΔC^* Duncan grouping	ΔE^* Level	ΔE^* Mean	ΔE^* Duncan grouping	ΔH^* Level	ΔH^* Mean	ΔH^* Duncan grouping
HW	Temp.	170℃	2.3	A	230℃	37.1	A	230℃	4.4	A
		185℃	1.3	B A	215℃	31.0	B	215℃	3.7	B
		200℃	0.5	B	200℃	25.5	C	200℃	3.0	C
		215℃	-2.5	C	185℃	10.1	D	185℃	1.7	D
		230℃	-5.4	D	170℃	12.6	D	170℃	1.2	E
	Time	1h	1.3	A	5h	29.9	A	5h	3.7	A
		2h	0.8	A	4h	28.3	A	4h	3.4	A
		3h	-0.7	B	3h	24.7	B	3h	2.8	B
		4h	-2.1	C	2h	21.2	C	2h	2.2	C
		5h	-3.1	C	1h	17.1	D	1h	1.9	C
SW	Temp.	170℃	3.5	A	230℃	50.4	A	230℃	6.3	A
		185℃	2.9	A	215℃	46.3	B	215℃	5.9	B
		200℃	2.1	A	200℃	40.8	C	200℃	5.5	C
		215℃	-0.4	B	185℃	23.3	D	185℃	4.0	D
		230℃	-4.2	C	170℃	16.2	E	170℃	2.7	E
	Time	1h	2.9	A	5h	44.3	A	5h	5.9	A
		2h	2.2	A	4h	37.7	B	4h	5.2	B
		3h	1.0	B A	3h	35.3	B	3h	4.8	C
		4h	-0.6	B C	2h	31.6	C	2h	4.3	D
		5h	-1.5	C	1h	28.2	D	1h	4.1	D

Note: α=0.05

Table 3. Percent Change in Chemical Components Contents in Steam-Heat Treated Chinese Fir Wood

		Holocellulose					α-cellulose					Lignin				
	℃	1h	2h	3h	4h	5h	1h	2h	3h	4h	5h	1h	2h	3h	4h	5h
HW	170	2.9	3.0	4.3	5.2	7.8	0.3	1.4	2.1	2.3	2.4	-0.5	-0.8	-1.0	-1.1	-1.4
	185	5.2	6.3	7.6	8.8	9.1	0.9	2.1	3.5	4.2	5.6	-0.4	-0.9	-1.1	-1.9	-2.9
	200	10.3	11.4	14.4	15.2	16.1	1.6	6.6	7.5	9.1	11.2	-3.4	-3.9	-5.4	-7.9	-8.8
	215	16.6	17.1	18.8	18.9	21.0	9.2	13.6	14.6	28.6	29.8	-7.8	-13.2	-14.5	-18.1	-20.4
	230	19.4	20.1	20.6	21.0	21.4	17.0	18.4	26.9	32.6	35.3	-15.9	-16.1	-22.2	-22.5	-22.6
SW	170	2.9	4.2	5.6	7.2	7.5	0.3	2.3	2.6	5.3	7.2	-0.5	-0.8	-1.1	-2.3	-2.4
	185	3.2	5.2	6.5	9.3	10.2	2.9	3.2	3.8	12.3	13.1	-0.5	-2.2	-2.6	-3.2	-3.3
	200	11.2	12.7	14.2	15.5	16.5	15.2	17.3	26.6	28.5	32.0	-3.7	-3.8	-5.9	-9.0	-9.4
	215	13.4	13.8	16.4	18.7	21.6	18.0	20.0	32.5	40.5	47.9	-9.2	-9.9	-12.5	-20.0	-21.9
	230	15.3	16.9	20.7	21.1	22.7	32	39.3	44.9	46.8	50.3	-15.7	-19.3	-26.2	-31.0	-37.2

Note: Negative value means an increasing percentage in ratio of lignins compared to untreated sample due to the losses of cellulose and hemicellulose decrease evidently; positive value means a decreasing percentage in holocellulose and α-cellulose content compared to untreated sample, respectively.

Figure 1 shows that at temperatures from 170 to 230 ^{o}C with a treatment time of 1 hour, the $\triangle C^*$ decreased significantly by 6 in heartwood and by 4 in sapwood; the $\triangle E^*$ increased significantly by 22 in heartwood and by 30 in sapwood, and the $\triangle H^*$ increased by 2 in heartwood and by 4 in sapwood. However, Fig. 2 shows that at 170 ^{o}C for different treatment times from 1 to 5 hours, the $\triangle C^*$ decreased slightly by 3 in heartwood and by 4 in sapwood; the $\triangle E^*$ increased slightly by 8 in heartwood and by 13 in sapwood, and the $\triangle H^*$ increased only by 1 in heartwood and by 2 in sapwood. This implies that the velocity of color change in wood was accelerated when the treatment temperature was rising under the same treatment time; however, the velocity of color change in wood became slower when the treatment time was extended from 1 to 5 hours at the same treatment temperate. The similar phenomena were observed in other treatment combinations. This result reveals that treatment temperature has a stronger effect on wood color than treatment time.

Repeated two-way analysis of variance (Table 1) showed there was a significant difference at the level of 0.01 between the $\triangle C^*$ value and the treatment temperature, time, and interaction of above two factors, respectively, due to $F > F_{0.01}$. The same result was also obtained for $\triangle E^*$ and $\triangle H^*$ except for the interaction of temperature and time to $\triangle H^*$ in sapwood due to $F < F_{crit}$. This revealed that the temperature is very important with respect to color change in wood during the process of steam-heat treatment, because the color darkening was accelerated generally, and especially when the treatment temperatures exceeded 200°C. The results of multi-comparison of parameters on steam-heat treatment are shown in Table 2. At the level of 0.05, there was a significant difference between two temperatures within the range from 170 °C to 230 °C for $\triangle H^*$, $\triangle E^*$ except for the comparison of 170 °C to 185 °C with respect to heartwood, and except for the comparisons of 170 °C to 185 °C and185 °C to 200 °C in $\triangle C^*$.

Chemical components of wood are modified significantly, in addition to the changes in wood color due to thermal treatment (Viitanen *et al.* 1994; Katsuya 2006). The pyrolysis reaction of chemical components in wood usually happens at above 165 °C (Stamm and Hansen 1937). During the steam-heat treatment of wood, hydroxyl groups are oxidized to carbonyl groups and carboxyl groups so that the wood color is darkened remarkably due to the carbonyl groups belonging to chromophoric groups and the carboxyl groups belonging to auxochromic groups, respectively. On the other hand, lignins content and extractive composition of wood are major factors in wood color. During the thermal treatment of wood, lignins modification mainly involves diphenyl-methane type condensation and pyrolysis reactions (Funaoka *et al.* 1990). The increase of lignins content usually leads to darker wood color (Sundqvist 2002). In this study, the ratio of lignins was increased by 23% for heartwood and by 37% for sapwood, respectively due to the losses of cellulose and hemicellulose decreased evidently, as seen in Table 3. In a way, this result contributed to darkness of wood color. The treatment temperature and time play important roles in changing wood color during the steam-heat treatment. The treatment temperature has a stronger effect on wood color than time.

CONCLUSIONS

Based on the laboratory experiments, the following conclusions were drawn from this study. Color improvement of Chinese fir can be achieved through the steam-heat treatment. The treatment temperature has a stronger effect on wood color than the treatment time. 200 °C is a significant critical point to darken wood color during the steam-heat treatment. The chroma difference ($\triangle C^*$) decreased, while the color difference ($\triangle E^*$) and the hue difference ($\triangle H^*$) increased, with an increase in treatment temperature and time. It was observed that the wood color was gradually changed from the original light color to brown and even dark brown color. It was found that the lignins are some of the main contributions to darkening the wood color because their relative content in wood increased remarkably during the treatment.

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