EFFECT OF STEAM-HEAT TREATMENT ON MECHANICAL PROPERTIES OF CHINESE FIR

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Heat treatment often brings about some negative effects on mechanical properties of wood. Chinese fir is currently underutilized due to some inherent properties that limit its further applications. Using steam as a heating medium and a shielding gas, the heartwood and sapwood of Chinese fir were treated at a temperature ranging from 170º C to 230º C and time from 1 to 5 hours in an airtight chamber. Both the modulus of rupture (MOR) and modulus of elasticity (MOE) were increased for the sapwood specimens under the temperature less than 200º C for short treatment times. The hardness was increased for both two kinds of specimens under the temperature less than or about 200º C, compared to the untreated specimens. The temperature has a stronger effect on mechanical properties of wood than the time, and the temperature of 200 º C is a critical point in modifying mechanical properties of wood.

Keywords: Chinese fir; Heartwood; Sapwood; Steam-heat treatment; Hardness; Modulus of elasticity; Modulus of Rupture

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INTRODUCTION

Thermal treatment of wood has been known as an effective method to improve the dimensional stability and bio-durability of wood for a long time. In the meantime, it often causes a reduction of hygroscopicity, and it decreases the modulus of rupture (MOR), the modulus of elasticity (MOE), and even the hardness of wood. The thermal treatment of wood, however, compared to wood preservation by chemical treatments, is an environmentally friendly and pesticide-free process. There are currently five typical processes nowadays used in an industrial scale. They are the Thermowood process in Finland (Syjänen 2001; Jämsä and Viitaniemi 2001), the oil-heat treatment in Germany (Rapp and Sailer 2001), the Plato process in the Netherlands (Militz and Tjeerdsma 2001), and the Le Bois Perdure process and the Retification process in France (Vernois 2001). The foremost advantages of treating wood in these manners are the increased resistance to different types of biodegradation and the improved dimensional stability due to a reduction in wood hygroscopicity. Unfortunately, the negative effects are the loss of strength and increased brittleness of the thermally treated wood.

High temperature, generally speaking, often reduces the mechanical properties of wood during the thermal treatment. Stamm et al. (1946) reported that the impact modulus of rupture (MOR) and modulus of elasticity (MOE) could be reduced up to 50%. Kamdem et al. (2002) indicated that the MOR and MOE of beech (*Fagus sylvatica*) were decreased by 40% and 20%, respectively, due to the heat treatment. Bekhta and Niemz (2003) showed that the MOR of spruce (*Picea abies*) was decreased by 44 to 50% as the treating temperatures were raised from 100 °C to 200 °C, whereas the temperature had no effect on the MOE. A similar result was also obtained by Korkut et al. (2008), who reported that the MOR, MOE, and surface hardness in the radial direction of Scots pine (*Pinus sylvestris* L.) decreased by 33%, 32%, and 27%, respectively, at 180ºC for 10 hrs. Surprisingly, other researchers reported in an opposite way that the mechanical properties of wood were improved using proper treating temperatures. Many studies revealed that there was a slight increase in MOE when wood was thermally treated for short times (Millett and Gerhards 1972; Rusche 1973; Kubojima et al. 2000; Navi and Girardet 2000; Santos 2000; Poncsák et al. 2006; Shi et al. 2007). The MOR also showed an increase initially and then decreased with extended heating time, especially for treatment temperatures above 200ºC (Bekhta and Niemz 2003). Kubojima et al. (2000) stated that the MOR and MOE of Sitka spruce (*Picea sitchensis* Carr.) increased at the initial stage of the heat treatment and decreased later at 160ºC for times from 0.5 to 16 hrs. Shi et al. (2007) reported that the MOE and hardness of fir (*Abies* spp.) were increased by 17% and 6%, respectively, at 202ºC for 3 hrs, though the MOR and MOE were decreased by 37% and 6% at 212ºC for 3 hrs respectively, while the hardness was still increased by 7%.

Both wood species and process parameters (temperature/time) play important roles in determining the final mechanical properties of heat-treated wood. Therefore, it is necessary to deepen our understanding on how to control and minimize the loss of mechanical properties in order to extend industrial applicability of heat-treated wood. Chinese fir is one of the most commonly planted tree species in China, and it is widely using in many fields, such as furniture manufacturing, ornamental materials. However, as a fast-growing species, Chinese fir has low mechanical properties. This study was aimed at investigating how to control the treatment temperature and time to minimize and even improve the mechanical properties of wood by steam-heat treatment. The goal was to provide theoretical background that can support refinement of industrial processes.

MATERIALS AND METHODS

Materials and Steam-Heat Treatment

Fifteen Chinese fir (*Cunninghamia lanceolata* (Lamb.) Hook) trees were selected and freshly cut from a planted forest in Hunan Province, China. The Random Complete Block Design (RCBD) method was performed to design the experiment units in this study. The logs were broken down into boards that were dried to an initial moisture content (MC) of around 8% by a high-frequency drier. Then steam-heat treatment was conducted on wood specimens with a dimension of 50 × 25 × 500 mm (radial × tangential × longitudinal) at temperatures of 170, 185, 200, 215, and 230ºC and times of 1, 2, 3, 4, and 5 hours in an airtight chamber with an atmosphere comprising less than 2 per cent oxygen. Superheated steam was used as a heating medium and a shielding gas. The MC of the steam-heat-treated boards was about 4%. Those boards without any visual defects were selected for the properties testing. Correspondingly, the untreated boards of the same species were used as control specimens.

Specimen Preparation and Properties Testing

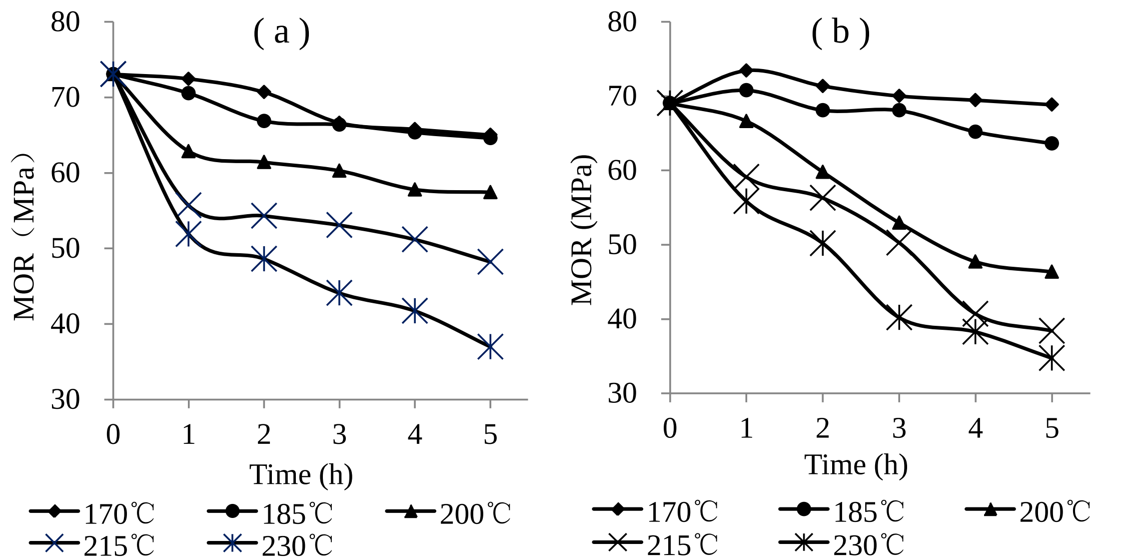

Fig. 1. MOR of untreated and steam-heat-treated Chinese fir heartwood (a) and sapwood (b)

The specimens for the MOR and MOE testing were cut from steam-heat-treated and untreated boards. There were fifteen replications for each series. All specimens were conditioned in a climate chamber at 20ºC and 65% relative humidity (RH) for three weeks prior to the properties testing. The specimens with a dimension of 20 × 20 × 300 mm (radial × tangential × longitudinal) were tested by pressing at tangential section using three-point bending test method (GB 1927～1943-91, China) with a span of 300 mm and a loading speed of 25 mm/min.

The specimens with 40 mm in width and length and 20 mm in thickness were used for the hardness testing. Based on the Japanese Standard (JIS Z 2101 – 1994), a ball with 10 mm in diameter was forced to penetrate into wood at a depth of 0.32 mm for all specimens during the hardness testing. The penetration positions were chosen far enough from the edges of the specimens to prevent from splitting or chipping. A load was applied to a specimen continuously at a rate of 0.3 mm/min. Total six tests on one tangential section of a specimen were carried out and fifteen replications were tested for each series.

RESULTS

The relative changes of the MOR, MOE, and hardness of steam-heat-treated and untreated Chinese fir are presented in Table 1. The tabulated values show that some treatment combinations decreased the MOR, MOE, and hardness of both heartwood and sapwood specimens, but the others slightly increased the MOR and MOE of sapwood. On the other hand, the hardness was increased for both heartwood and sapwood specimens when the treatment temperature was under or around 200ºC. Analysis of variance (ANOVA) (Table 2) indicated that there was a significant difference at the level of 0.01 on model testing of mechanical properties of steam-heat-treated heartwood and sapwood respectively, as also on block testing. This result validated the veracity of the use of RCBD method to arrange experiments of steam-heat treatment of Chinese fir in this study.

Mechanical Properties of Steam-Heat-Treated Heartwood

A reduction in both MOR and MOE occurred at initiation of steam-heat treatment until the end, as can be seen from Table 1, compared to the MOR of 73 MPa and the MOE of 11 GPa in the untreated heartwood. However, the hardness increased slightly when the treatment temperature was under or around 200 ^{o}C for a short time, even at 215ºC for 1h. Figure 1(a) shows that the MOR declined slowly from 170ºC to 185ºC at 1h. However, when the treatment temperature exceeded 185ºC, this downward trend was accelerated suddenly. Overall, the MOR decreased with an increase in treatment time. With respect to the MOE, its downward trend was also accelerated once the treatment temperature was over 200ºC, as can be seen from Fig. 2(a). Figure 2(a) also shows that the MOE slowly decreased with an increase in treatment time. The maximum loss values were 49% for MOR and 22% for MOE, respectively, at 230ºC for 5 hrs. This result implied that the treatment temperature had a stronger effect on mechanical properties of wood than the treatment time. Furthermore, an interesting phenomenon was observed that the hardness increased at the initial stage of steam-heat treatment and decreased later, as can be seen in Fig. 3(a). Compared to 10 N/mm^2 of untreated heartwood, the increased relative change of 6% for hardness not only could be obtained at 170ºC for 3 hours but also at 200ºC for 2 hours. This implies that the same result could be achieved by lower temperature and longer time or by higher temperature and shorter time. This finding is very helpful to industries to save energy in a production line. In this study, the maximum increase of hardness was 13% obtained at 200ºC for 1h (Table 1), while the maximum loss was 26%, obtained at 230ºC for 5 hours (Table 1).

ANOVA results on mechanical properties of heartwood are shown in Table 2. The results showed a significant difference at the level of 0.01 between the treatment temperature and the MOR, MOE, and hardness respectively. A similar result also existed between the treatment time and aforementioned three properties. However, the interaction of treatment temperature and time had no significant difference in the MOR and MOE, except for the hardness. Multi-comparison results as shown in Table 3 indicated that there was a significant difference for the MOE at the level of 0.05 between adjacent two treatment temperatures, but for the MOR not including between 170ºC and 185ºC, and for the hardness not including from 170ºC to 200ºC. Regarding treatment time, the significant difference only existed between 1 to 3 hours for the MOR and 4 and 5 h for the MOE. Therefore, it is obvious that the treatment temperature has a stronger effect on mechanical properties of wood than time. In particular, a temperature of 200ºC can be regarded as a critical point for modifying mechanical properties of wood.

Mechanical Properties of Steam-Heat-Treated Sapwood

It was observed that the sapwood specimen had a holocellulose content of 70% and an α-cellulose of 45%, while the heartwood specimen had a holocellulose of 68% and α-cellulose of 42% in this study determined according to China Standard GB/T 2677.10-1995 and GB/T 744-1989, respectively. Therefore, even the same treatment combination yields different effects on mechanical properties of wood. The MOR, MOE, and hardness of sapwood specimens increased at the initial stage of steam-heat treatment and decreased later, as seen in Table 1. At 170ºC and 185ºC, the increase of the MOR and MOE of treated sapwood, compared with 69 MPa and 12 GPa of untreated sample respectively, were decreased with an increase in treatment time. Figure 1(b) shows that the reduction of MOR was accelerated when the treatment temperature exceeded 200ºC. Figure 2(b) also indicates the MOE was increased only at 170ºC and 185ºC for different treatment times, and then decreased with the elevated treatment temperature and time. The maximum loss of MOE was 22%, which was obtained at 230ºC for 5 hrs. On the other hand, the hardness was increased remarkably under most treatment combinations. Compared to the hardness of 8 N/mm^2 in untreated sapwood, the maximum increase of 27% was achieved at 200ºC for 2 hrs. Figure 3(b) shows that when the treatment temperature exceeded 200ºC, a reduction of hardness occurred. This phenomenon implies the temperature of 200ºC is a critical point for modifying mechanical properties of wood.

ANOVA results on mechanical properties of sapwood are shown in Table 2. There was a significant difference at the level of 0.01 between the treatment temperature and the MOR, MOE, and hardness, respectively. A similar difference also was found between the treatment time and aforementioned three properties. However, the interaction of treatment temperature and time had no significant effect on the MOE. Multi-comparison results, as shown in Table 3, indicated that there was a significant difference for the MOR at the level of 0.05 between adjacent two temperatures but not including between 170ºC and 185ºC, for MOE not including between 185ºC and 200ºC, and for hardness only between 200ºC and 215ºC. With respect to the treatment time, there was a significant difference only for the MOE between adjacent two treatment times, but neither for the MOR nor for the hardness. This result implied that the properties of wood have substantially difference between sapwood and heartwood.

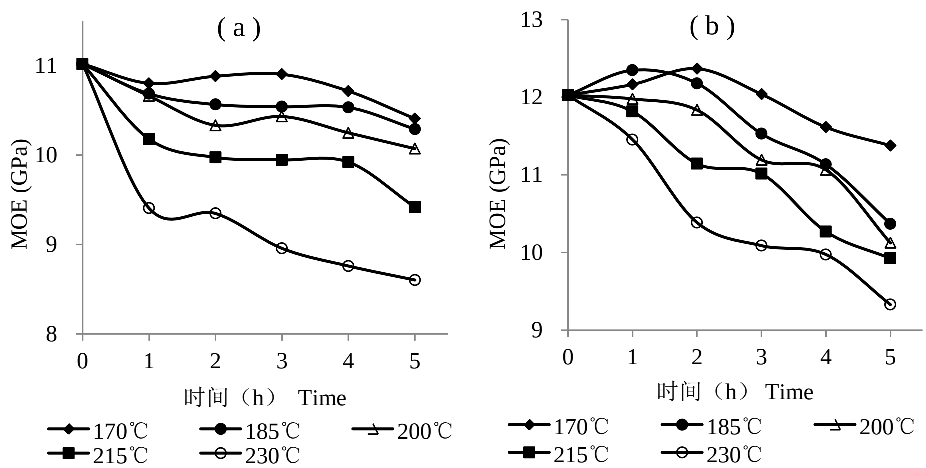

Fig. 2. MOE of untreated and steam-heat-treated Chinese fir heartwood (a) and sapwood (b)

Table 1. Relative Changes of Mechanical Properties of Steam-Heat-Treated Chinese Fir Heartwood and Sapwood

Properties	Time	Heartwood					Sapwood				
		170℃	185℃	200℃	215℃	230℃	170℃	185℃	200℃	215℃	230℃
Modulus of Rupture (%)	1h	1	3	14	24	29	-6	-3	4	14	19
	2h	3	8	16	26	33	-3	1	13	18	27
	3h	9	9	18	27	40	-1	1	23	27	42
	4h	10	11	21	30	43	-0.6	6	31	41	45
	5h	11	12	21	34	49	0.3	8	33	44	50
Modulus of elasticity (%)	1h	2	3	3	8	15	-1	-3	0.4	2	5
	2h	1	4	6	9	15	-3	-1	2	7	14
	3h	1	4	5	10	19	-0.1	4	7	8	16
	4h	3	4	7	10	21	3	7	8	15	17
	5h	6	7	9	15	22	5	14	16	17	22
Hardness (%)	1h	-0.3	-3	-13	-2	9	-0.3	-5	-23	-3	11
	2h	-3	-5	-5	5	16	-8	-24	-27	2	14
	3h	-6	-5	8	14	20	-10	-19	-1	8	16
	4h	3	-7	9	17	21	-12	-13	6	10	18
	5h	4	3	15	20	26	-13	-8	9	14	24

Note: relative change % = (value of after treatment – value of control) / value of control ×100%

DISCUSSION

Thermal modification is invariably performed between the temperatures from 180ºC and 260ºC, with temperatures lower than 140ºC resulting in only slight changes in mechanical properties, such as the MOR and MOE, and higher temperatures resulting in unacceptable degradation to the substrate (Hill 2006). The result of mechanical properties of wood obtained in this study is in good agreement with this conclusion. Sundqvist et al. (2006) found that thermal treatment enhanced mechanical properties of wood at temperatures around 180 to 200ºC for short treatment times. It was observed that the treatment temperature and time are two crucial factors affecting the final quality of steam-heat-treated wood, with the treatment temperature having a stronger effect than time.

The mechanical property of thermally treated wood usually depends on the degree of pyrolysis of three main organic components of wood, i.e. cellulose, hemicelluloses, and lignin. Generally speaking, wood begins to degrade obviously at a temperature of about 165ºC (Stamm and Hansen 1937). Among the three main components, hemicellulose is the most sensitive to temperature and decomposes much faster than cellulose, and cellulose decomposes faster than lignin (Stamm 1964; Yildiz et al. 2006). The three components behave differently during steam-heat treatment, since they have different structures and different distributions in wood cell walls, which also determine their functions in wood properties. As wood is heated, there is a decrease in its weight initially, due to the loss of bound water and volatile extractives, with less volatile extractives tending to migrate to the surface of the wood.

On the other hand, the holocellulose and α-cellulose content in thermal-treated Chinese fir heartwood, compared with untreated wood specimens, decreased from 3% to 21% with an increase of treatment temperature from 170 to 230°C and treating time from 1 to 5 hours, while the lignin content increased from 0.5% to 23% with the same treatment parameters, according to China standard GB/T 2677.10-1995, GB/T 744-1989 and GB/T 2677.8-94, respectively. Under the same treatment conditions, the holocellulose and α-cellulose content in thermal-treated sapwood decreased from 3% to 23% and 0.3% to 50%, respectively, while the lignin content increased from 0.5% to 37%. This result is regarded as one of the main proofs to explain the difference between steam-heat-treated heartwood and sapwood.

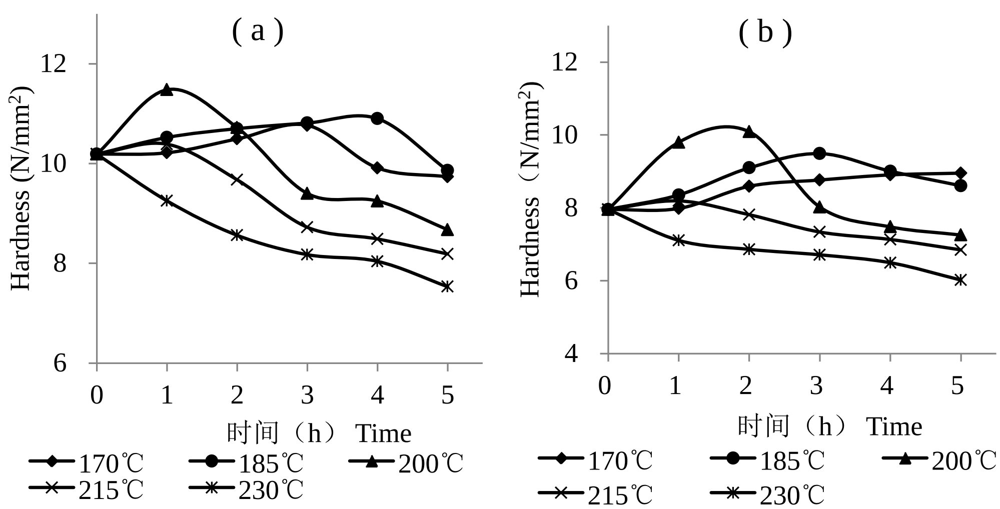

Fig. 3. Hardness of untreated and steam-heat-treated Chinese fir heartwood (a) and sapwood (b)

As the treatment temperature and time are increased, the most thermally labile polymeric components of the wood begin to degrade, resulting in the production of methanol, acetic acid, and various volatile heterocyclic compounds, such as furans, γ-valerolactone, and so on (Hill 2006; Bourgois and Guyonnet 1988; Sivonen et al. 2002). Loss of hemicelluloses leads to an increase in the degree of crystallinity of wood samples, in addition to those changes related to degradation/rearrangement of the amorphous cellulose content. The thermal degradation of cellulose and hemicelluloses is the main reaction in which the fiber chains become shorter and shorter along with the increase of temperature and time during the thermal treatment so as finally to decrease the mechanical properties of the wood.

Table 2. Repeated Two-way Analysis of Variance (ANOVA) on Mechanical Properties of Steam-heat-treated Chinese Fir Heartwood and Sapwood

Species	Source	DF	$P_r > F$		
			MOR	MOE	Hardness
Heartwood	Block	14	< .0001	< .0001	< .0001
	Temp.	4	< .0001	< .0001	< .0001
	Time	4	< .0001	0.0005	< .0001
	Temp.×Time	16	0.8962	0.9936	< .0001
	Model	38	< .0001	< .0001	< .0001
Sapwood	Block	14	< .0001	< .0001	< .0001
	Temp.	4	< .0001	< .0001	< .0001
	Time	4	< .0001	< .0001	0.0001
	Temp.×Time	16	0.0003	0.9745	0.0001
	Model	38	< .0001	< .0001	< .0001

Note: α=0.01

Table 3. Multi-Comparison of Treating Parameters on Steam-Heat-Treated Chinese Fir Heartwood and Sapwood

Species	Factor	MOR		MOE	Hardness	
		Level	Duncan grouping	Duncan grouping	Level	Duncan grouping
Heartwood	Temp.	170℃	A	A	185℃	A
		185℃	A	B	170℃	A
		200℃	B	C	200℃	A
		215℃	C	D	215℃	B
		230℃	D	E	230℃	C
	Time	1h	A	A	1h	A
		2h	B	B A	2h	B A
		3h	C	B C	3h	B C
		4h	D C	C	4h	D C
		5h	D	D	5h	D
Sapwood	Temp.	170℃	A	A	185℃	A
		185℃	A	B	170℃	A
		200℃	B	B	200℃	A
		215℃	C	C	215℃	B
		230℃	D	D	230℃	B
	Time	1h	A	A	2h	A
		2h	B A	B	1h	B A
		3h	B C	C	3h	B A
		4h	D C	D	4h	B A
		5h	D	E	5h	B

Note: α=0.05

CONCLUSIONS

In this paper in has been shown that steam-heat treatment is an efficient, environmentally friendly method to modify the mechanical properties of wood. The MOR, MOE, hardness of Chinese fir sapwood, and hardness of heartwood improved when temperatures were under or around 200ºC for short treatment times. Once the treatment temperature exceeded 200ºC, the pyrolysis and degradation reaction became more serious, incrementally destroying the construction and content of cellulose, hemicelluloses, and lignin in cell wall of wood, eventually leading to a serious reduction in mechanical properties of wood. Therefore, the temperature of 200ºC can be considered as a critical point in terms of modifying wood's mechanical properties. It was concluded based on plenty of experimental data in this study that the treatment temperature and time are two important factors in steam-heat treatment of wood, while the treatment temperature has a stronger effect than time.

In order to control and minimize the loss of mechanical properties of wood by steam-heat treatment, a good suggestion would be that the treatment temperature should be controlled below or about 200ºC. It is recommended to choose a reasonable treatment temperature and time according to the desired final properties of wood products and energy saving plans.

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