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THE EFFECT OF THERMAL MODIFICATION BY HOT PRESSING ON THE SOME PHYSICAL AND MECHANICAL PROPERTIES IN RUBBERWOOD (*HEVEA BRASILIENSIS*)

Zhou Qiaofang, Chen Chuanfu, Zhao Xiangyu, Tu Dengyun, Li Kaifu
South China Agricultural University, College of Materials
and Energy, Department of Wood Science and Engineering
Guangzhou, China

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ABSTRACT

Rubberwood (*Hevea brasiliensis*) was thermal modified by hot pressing in an open system at three different temperatures (170, 185, 200°C) and two different durations (1.5, 3 h), and the effect on the physical and mechanical properties was studied. Results show that the thermal modification increased the oven-dried density and decreased the EMC (equilibrium moisture content) by 7.93% and 37.15%, respectively, and the dimensions stability was improved. Hardness, bending strength, modulus of bending and compressive strength parallel to grain of modified samples basically decreased with increasing temperature and time, but they showed a meaningful increase compared to control samples. However, impacting bending and nail withdrawal resistance decreased after hot pressing and thermal treatment, and the failure of the compensation for the impairment was the rubberwood hot pressed and thermal treated in the presence of air, and the participation of oxygen provoked rapid degradation reactions during the treatment.

KEYWORDS: Thermal modification, compression ratio, rubberwood, physical properties, mechanical properties.

INTRODUCTION

Wood has been used in buildings and furniture because of its many outstanding features such as esthetic appearance, easy machinability, high ratio strength and low-carbon track in life circle (Percin et al. 2016). With population growth and economic development, wood consumption has increased exponentially for the past several decades. In order to balance the requirement of wood materials and environmental protection, fast-growing trees for wood supply are planted extensively around the world. Fast-growing wood usually has low density and is instable under changing moisture conditions, thus it's highly recommended to have it conducted some physical or chemical modification to reach the standards which high value added wood products call for.

Thermal modification is one of the many techniques applied to improve the natural quality properties of the low value wood, such as dimensional stability, surface quality and biological durability and equip the wood material with new properties (Kamden et al. 2002, Tjeerdsma et al. 1998, Tjeerdsma and Militz 2005, Hakkou et al. 2006, Jamsa and Viitaniemi 2001, Bekhta and Niemz 2003, Unsal and Ayrilmis 2005). Since this modification method doesn't add any toxic chemicals in the process, thermal wood is considered to be an ecological alternative to impregnated wood materials, and several processes have been industrialized: Plato process in the Netherlands, thermowood developed at VTT in Finland, Rectification or Bois Perdure in France, or the OHT in Germany (Rapp 2001, Esteves and Pereira 2009). The extent of the change in wood properties during the thermal treatment mainly depends on the processing method, the wood species and its characteristic properties, the initial moisture content of wood, the surrounding atmosphere, treatment duration and temperature (Yildiz et al. 2006). Temperature has greater effect on the thermal wood properties than time, and prolonging the treating time does not bring the corresponding properties compared to the treatment at higher temperature (Korkut et al. 2008a, Korkut and Guller 2008).

Through thermal treatment the wood is darkened and the deep color is appealing to customers for it is similar to the high quality tropical wood (Esteves et al. 2008, Aksoy et al. 2011, Santos et al. 2014). In a study on pine (*Pinus pinaster*) and eucalypt (*Eucalyptus globulus*) wood, thermal treatment by hot air or steam for a duration varying from 2 h to 24 h above 170°C generated a darker wood color with lightness decrease by 50% for both species (Esteves et al. 2008).

Thermal wood also has other advantages compared to unmodified wood such as lower EMC (equilibrium moisture content) (Obataya et al. 2000, Zhou et al. 2013, Santos et al. 2014), better heat insulation (Pasztory et al. 2017, Rapp 2001) and smoother contact surface (Unsal and Ayrilmis 2005, Korkut and Guller 2008). However, the thermal treatment is detrimental to wood mechanical properties, especially to static bending strength, dynamic bending strength, impact bending and compressive strength (Borrega and Karenlampi 2008, Gunduz et al. 2009, Kol 2010, Poncsak et al. 2006, Gunduz and Aydemir 2009, Zhang et al. 2015). In a study on pine (*Pinus pinaster*) and eucalypt (*Eucalyputs globulus*) wood, the bending strength reduced by 40% and 50% in a heat treatment for 12 h at 210°C, respectively (Esteves et al. 2007). Increase in temperature and time might severely reduce the mechanical properties of wood, MOR and MOE of treated beech (*Fagus orientalis* Lipsky) under 200°C decreasing by 16.82% and 16.11% were also observed (Percin et al. 2016).

How to remedy the mechanical impairment is the major topic for wood thermal modification today. There are two ways to compensate for the mechanical loss of thermal wood, one is by compressing the wood to increase its density prior to or after thermal modification (Gong et al. 2010, Tu et al. 2014, Rautkari et al. 2013, Welzbacher et al. 2008, Esteves et al. 2017), and another is thermal treating wood under some extent of pressure (Hidayat et al. 2016). This study compressed the Thailand rubberwood (*Hevea brasiliensis*) with relative low compression ratio prior to thermal treatment under pressure by hot pressing, and explored the influence on density, equilibrium moisture content, bending strength and other physical and mechanical properties.

MATERIAL AND METHODS

Wood material

Air-dried rubberwood (*Hevea brasiliensis*) came from Thailand was used in this study. Plain sawn clear wood specimens were prepared with dimensions of 300 mm long (grain direction) and 105 mm wide (tangential direction) and 20 mm thick (radial direction). Seven groups of specimens (six for thermal treatment by hot pressing and one for control) were prepared, and each group had 18 specimens.

Thermal modification

Thermal treatments were conducted on a laboratory type hot-press and in the presence of air. Three different temperatures (170, 185, 200°C) and two different durations (1.5, 3 h) were applied to specimens in this study. The total thermal treatment was performed in four continuous phases as shown in Fig. 1, taking 185°C/3 h thermal treatment as an example. In the first phase, rubberwood specimens with moisture content of ~12% were dried to zero moisture content by elevating the temperature of hot-press to 170, 185 and 200°C, meanwhile the specimens were loaded with the pressure of 2 MPa in the first 20 min and then 4 MPa until the target temperature was reached. In the second phase, the pressure increased to 12 MPa in 30 seconds and kept 3 minutes, and the wood was compressed to target thickness (18 mm) by using distance stop. In the third phase, the pressure decreased to 2 MPa and the specimens were heat-treated in the plate at the target temperature for 1.5 or 3 h. In the last phase, hot-press was cooled down to room temperature by introducing the flow water into the plate for 30 minutes under the pressure of 2 MPa, and then the experiment finished. There were 3 repeated tests for each thermal treatment condition.

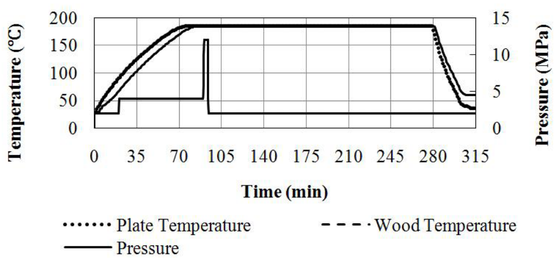

Fig. 1: Process schedule used in the thermal modification by hot pressing (185°C, 3 h).

After the thermal modification, the treated and control specimens were conditioned at 20°C and 65% relative moisture content for 2 months. In order to determine the physical and mechanical properties of rubberwood, all the specimens were cut into small clear samples and the density (20×20×18 mm) was measured according to GB/T 1933 (2009), EMC (20×20×18 mm) was measured according to GB/T 1931 (2009), hardness (50×50×18 mm) was measured according to GB/T 1941 (2009), bending strength (300×20×18 mm) was measured according to GB/T 1936.1 (2009), modulus of elasticity (300×20×18 mm) was measured according to GB/T 1936.2 (2009), compression strength parallel to grain (30×20×18 mm) was measured according to GB/T 1935 (2009), impact bending strength (300×20×18 mm) was measured according to GB/T 1940 (2009), and nail withdrawal resistance (150×50×18 mm) was measured according to GB/T 14018 (2009). For each test, 15 samples were prepared.

The density (D0 or D12), EMC, hardness (HD), bending strength (MOR), modulus of elasticity (MOE), compression strength parallel to grain (CS), impact bending strength (IB) and nail withdrawal resistance (WR) were calculated by the formulas of 1-8, respectively.

$$D_{0/12} = \frac{m_{0/12}}{V_{0/12}} \qquad (g\cdot cm^{-3}) \qquad (1)$$

where: D_0 - oven-dried density ($g\cdot cm^{-3}$), when the moisture content is 0%,
D_{12} - air-dried density ($g\cdot cm^{-3}$), when the moisture content is 12% in nominal,
m_0 - the weight of the oven-dried sample (g),
m_{12} - the weight of the sample in equilibrium in the conditioning chamber at 0°C/ 65% RH (g),
V_0 - the volume of the oven-dried sample (cm^3),
V_{12} - the volume of the sample in equilibrium in the conditioning chamber at 20°C and 65% RH (cm^3).

$$EMC = \frac{m_{12} - m_0}{m_0} \times 100 \qquad (\%) \qquad (2)$$

where: m_{12} - the weight of the sample in equilibrium in the conditioning chamber at 20°C and 65% RH (g),
m_0 - the weight of the oven-dried sample (g).

$$HD = K \times F \qquad (N) \qquad (3)$$

where: K - coefficient, being 1 or 4/3 when the indention depth made by the steel ball is 5.64 or 2.82 mm,
F - the load applied (N).

$$MOR = \frac{3 \times F_{max} \times l}{2 \times b \times h^2} \qquad (MPa) \qquad (4)$$

where: F_{max} - maximum load (N),
l - the distance between two supports (mm),
b - the sample width (mm),
h - the sample thickness (mm).

$$MOE = \frac{\Delta F}{\Delta f} \times \frac{23 \times l^3}{108 \times b \times h^3} \qquad (MPa) \qquad (5)$$

where: $\frac{\Delta F}{\Delta f}$ - the slope of the elastic zone ($N\cdot mm^{-1}$),
l - the distance between two supports (mm),
b - the sample width (mm),
h - the sample thickness (mm).

$$CS = \frac{F_{max}}{b \times h} \qquad (MPa) \qquad (6)$$

where: F_{max} - maximum load (N),
b - the sample width (mm),
h - the sample thickness (mm).

$$IB = \frac{1000 \times Q}{b \times h} \qquad (kJ\cdot m^{-2}) \qquad (7)$$

where: Q - the energy absorbed by the sample (J),
b - the sample width (mm),
h - the sample thickness (mm).

$$WR = \frac{F}{t} \qquad (N \cdot mm^{-1}) \qquad (8)$$

where: F - withdrawal force (N),
t - the depth of the nail drilling to the sample (mm).

For all parameters, all multiple comparisons were first subjected to an analysis of variance (ANOVA) and significant differences between mean values of control and treated samples were determined using Duncan's multiple range test.

RESULTS AND DISCUSSION

Tab. 1 shows the results of oven-dried and air-dried densities and EMCs of control and thermal treated samples. By hot compressing and thermal treatment, the oven-dried density of samples increased from 0.694 $g \cdot cm^{-3}$ to the maximum value of 0.749 $g \cdot cm^{-3}$ at the treatment condition of 185°C/3 h.

Tab. 1: The effect of thermal modification by hot pressing on physical properties in rubberwood (Hevea brasiliensis).

Thermal treatment (°C)	Hour	Statistical value	D0 ($g \cdot cm^{-3}$)	D12 ($g \cdot cm^{-3}$)	EMC (%)
control		Mean	0.694 A	0.698 A	9.26 A
		s	0.010	0.011	0.16
170	1.5	Mean	0.730 B	0.698 AB	7.96 B
		s	0.015	0.014	0.14
	3	Mean	0.746 BC	0.712 ABC	7.71 C
		s	0.018	0.017	0.08
185	1.5	Mean	0.743 BCD	0.708 ABCD	7.67 C
		s	0.007	0.007	0.30
	3	Mean	0.749 CDE	0.712 ABCD	6.94 D
		s	0.007	0.006	0.19
200	1.5	Mean	0.746 BCDEF	0.757 E	6.02 E
		s	0.010	0.024	0.08
	3	Mean	0.732 BCDEF	0.738 E	5.82 F
		s	0.009	0.009	0.06

Mean = average value, s = Standard deviation A: Homogeneity groups (same letters in each columns indicate that there is no statistical difference between the samples according to the Duncan’s multiply range test at $P < 0.05$).

In a study on heat treatment of Camiyani Black pine wood, the oven-dry density values decrease with increasing temperature and heat treatment time under the conditions used in work of Gunduz et al. (2008). Wood treated over 150°C would witnessed an apparent mass loss because the degradation of wood's components especially the hemi-cellulose took place. In this study, the rubberwood was mechanically densified prior to thermal treatment with a low compression ratio of 10%, but the mass loss induced by thermal treatment was compensated and the oven-dried density had an increase of 5.19% in minimum. As can be seen, the air-dried density of control and modified samples treating below 185°C has no significant difference, however, as the temperature is above 200°C the density of the treating samples has a 6% increase, and it is because samples treating at higher temperature are more stable in dimensions and the compression-set recovery is lesser.

As the temperature increased and the treating duration prolonged, the EMC of the samples were reduced significantly and this phenomenon is in agreement with the previous studies (Obataya et al. 2000, Zhou et al. 2013, Santos et al. 2014). The highest temperature (200°C) and longest duration (3 h) has caused 37.15% reduction in EMC and this attribute is due to the degradation of wood hydrophilic carbohydrates in severe conditions.

Tab. 2 shows the effect of the temperature and duration of the thermal treatment on the hardness, MOR, MOE, compression strength parallel to grain, impact bending and nail withdrawal resistance of the rubberwood samples. And Tab. 3 shows the percentage increasing and decreasing of values in relation to the control for each treatment and each measured parameter.

Tab. 2: The effect of thermal modification by hot pressing on mechanical properties in rubberwood (Hevea brasiliensis).

Thermal treatment (°C)	Hour	Statistical value	HD (N)	MOR (MPa)	MOE (MPa)	CS (MPa)	IB (kJ·m^{-2})	WR (N·mm^{-1})	
								R	T
Control		Mean	3258.6	85.6	7140	45.8	95.8	41.4	41.4
			A	A	A	A	A	A	A
		s	391.0	12.0	930	5.2	24	2.1	3.6
170	1.5	Mean	3674.0	93.3	8590	69.4	59.0	36.4	37.2
			B	B	B	B	B	B	B
		s	440.9	13.1	870	7.4	14.2	4.0	3.8
	3	Mean	3388.6	92.4	8340	62.8	54.0	34.8	35.1
			AC	BC	BC	C	C	C	BC
		s	447.3	12.5	860	6.2	14.7	3.6	3.9
185	1.5	Mean	3456.5	89.8	8110	59.1	50.8	34.3	33.4
			ACD	BCD	BCD	CD	D	CD	CC
		s	490.8	7.2	970	3.1	11.6	3.8	3.8
	3	Mean	3924.9	87.1	7970	54.8	45.3	32.1	31.8
			BE	AD	CDE	DE	E	DE	D
		s	549.5	14.2	1030	5.4	8.4	4.2	3.6
200	1.5	Mean	3312.9	71.8	7820	53.2	39.9	32.8	32.2
			ACDF	E	DE	DE	F	DE	CDE
		s	430.7	10.0	1010	5.8	11.3	2.6	2.7
	3	Mean	3175.8	61.4	7310	47.3	33.8	30.0	30.1
			ACDF	F	AF	AF	G	F	CDE
		s	397.0	16.9	1020	5.0	9.6	2.5	3.9

Mean = average value, s = Standard deviation, A: Homogeneity groups (same letters in each columns indicate that there is no statistical difference between the samples according to the Duncan's multiply range test at $P < 0.05$).

Tab. 3: Increases and decreases in the values of measured parameters, as a result of temperature and times in thermal modification, in relation to control.

Thermal reatment (°C)	Hour	HD (%)	MOR (%)	MOE (%)	CS (%)	IB (%)	WR (%)	
							R	T
170	1.5	12.75	9.00	20.36	51.51	-38.46	-11.99	-10.28
	3	3.99	7.92	16.93	36.98	-43.58	-15.80	-15.20
185	1.5	6.07	4.82	13.67	29.03	-46.97	-17.16	-19.28
	3	20.45	1.71	11.70	19.53	-52.70	-22.54	-23.21
200	1.5	1.66	-16.11	9.65	16.05	-58.39	-20.76	-22.39
	3	-2.54	-28.26	2.45	3.24	-64.69	-27.52	-27.36

Hardness is one of the most important properties for flooring and its increase is one of the biggest advantages of densified wood (Esteves et al. 2017). In this study only the hardness of the compressed faces (in radial direction) were tested and the results indicated that hot pressing and thermal treatment could improve the hardness of the rubberwood, and samples treated at 185°C/3 h have the highest value 3924.9 N which is 120% of the hardness of control samples. When temperature was above 185°C, the hardness of the treated samples diminished, so it is not advised to hot compressing and thermal treating rubberwood above this temperature for generating a desirable hardness. Bruno Esteves et al. (2017) combined densification and heat treatment on *Maritime pine* wood and found that the hardness increased from 50% to 220%, but in their study the compression ratio was close to 50% and these high values are realized in the expense of the much loss of wood volume.

The MOR of the thermal treated samples decreased with the increasing temperature and treating duration, but it was still higher than that of control samples when treating below 185°C. Although the rubberwood samples were mechanically densified with a relative ratio prior to thermal treatment, it is enough to compensate for the impairment of MOR which diminishes a lot in traditional heat treatment above 170°C. As the temperature increased to 200°C, the MOR showed a sever reduction comparing to the control samples because hemi-cellulose and the amorphous areas of the cellulose of the wood decomposed harshly in such high temperature. Another desirable finding is the increase of MOE, even treating at 185°C for 3 h. The MOE increased by 11.7% compared of the control samples.

The compression strength parallel to grain of the control samples was 45.8 MPa, by hot pressing and thermal treatment the CS improved in each treating condition and the maximum value was 69.4 MPa at 170°C/1.5 h, increasing by 51.5%. In a study on the Red-bud maple (*Acer trautvetteri* Medw.) wood, the CS was found to decrease with the increasing temperature and time and the total loss could reach 32% compared to the control (Korkut et al. 2008b).

The increase of the MOR, MOE and CS lies in the mechanical densification by hot pressing (12 MPa/3 mins) prior to thermal treatment on the one hand, but on the other hand the thermal treatment under pressure (2 MPa) also played an important role here. Hidayat et al. (2015) has studied the effect of clamping on the mechanical properties during heat treatment and found that the clamping method contributed positively to the mechanical properties of the wood, and it decreased the reduction of MOR, MOE, compressive strength, and shear strength.

Impact bending (radial direction) and nail withdrawal resistance (tangential and radial direction) are two important properties for furniture. In this study IB and WR decreased with the increasing temperature and exposure time, both were lower than those of the control samples. In particular the IB of the control was 95.8 $kJ \cdot m^{-2}$; by hot pressing and thermal treatment it tremendously reduced to 33.8 $J \cdot m^{-2}$ at 200°C/3 h, decreasing by 64.7%. Korkut et al. (2008a) heat treated Scots pine wood at 120~180°C and observed that the impact bending decreased with the temperature and time, treating in the most severe condition (180°C/10 h) resulted in a loss of 42.4%. Nail withdrawal resistance didn't decrease so harshly as that of the impact bending, and even treating at 200°C/3 h the loss was no more than 28%.

The failure of the compensation for the impairment of IB and WR was that the rubberwood samples were hot pressed and thermal treated in the presence of air, and the participant of oxygen provoked degradation reactions during the treatment. It has been known that thermal degradation of wood heated in the presence of oxygen is more rapid than that of wood heated in an oxygen-free atmosphere. The decrease in the IB and WR properties can be reduced using a closed system with an inert gas like nitrogen or water vapor as the shielding gas instead of air (Yildiz et al. 2006).

CONCLUSIONS

In this study, the rubberwood was thermal modified by hot pressing. Properties such as oven-dried density, hardness, bending strength, modulus of bending, compressive strength parallel to grain were investigated and increased compared to the control, and the impairment of those properties were compensated. After thermal treatment, the EMC reduced significantly and the dimensions stability was improved. However, impacting bending and nail withdrawal resistance decreased after hot pressing and thermal treatment. It was found out that the failure of the compensation for the impairment occurred in case where the rubberwood was hot pressed and thermal treated in the presence of air. The presence of oxygen provoked rapid degradation reactions during the treatment.

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Zhou Qiaofang, Chen Chuanfu, Zhao Xiangyu, *Tu Dengyun, Li Kaifu
South China Agricultural University
College of Materials and Energy
Department of Wood Science and Engineering
Guangzhou, 510642
China
*Corresponding author: tudengyun@163.com
Phone: +86 135 7091 4607