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THE STUDY OF SURFACE DENSIFICATION AND TEMPERATE SUPERHEATED STEAM MODIFICATION FOR RUBBERWOOD

QINYI GU, HONGHAI LIU, TAO LI
NANJING FORESTRY UNIVERSITY
CHINA

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ABSTRACT

This study aimed to develop a high-value wood composite by surface-densification combined with temperate superheated steam modification technology. Rubberwood boards were hot-pressed, the impact of the peak density and thickness of densified layer on the physical and mechanical properties of wood were studied. In this study the platen temperature was kept constant, after hot-pressing the platen cooling phase was omitted replaced by special compression pressure decreasing and steam discharging technic, which could save much energy and time. Superheated steam treatment (190°C for 1.5 h) and conditioning (110°C for 2 h) were recommended for improving dimensional stability and durability. Study results indicated that the surface hardness and modulus of rupture of surface-densified followed by superheated steam modified rubberwood increased by 55.9% and 41.6% compared with non-densified one respectively. Dimensional stability improved substantially. The durability was improved to 'durable class' according to GB/T 13942.1-2009.

KEYWORD: Surface-densification, post heat treatment, mechanical properties, dimensional stability, durability.

INTRODUCTION

Rubberwood is an important raw material in Southeast Asia and China. The rubber tree cultivated in Thailand is renewed by approximately 300 thousand ha every year, and rubberwood exported to China accounts for 90% of the above (Cheng et al. 2017). Therefore, this is a sustainable development plantation tree material. Rubberwood features a beautiful texture and nice machining performance, and is suitable for making furniture and interior decoration. However, rubberwood is extremely prone to decay because of its content of approximately 8% starch and free sugar (Jie et al. 2018, Li et al. 2012). Thermal modification is an effective method to improve dimensional stability and biological durability without the use of chemicals to promote the application of low-value natural wood (Sandberg et al. 2017, Navi et al. 2012). Thermal modification can also change the color of wood from light yellow to dark

brown (Ding et al. 2017). The brown color is appealing to customers because it is similar to valuable wood, such as red oak. However, the mechanical strength of heat-treated wood is reduced because of the thermal degradation of hemicelluloses and part of the amorphous cellulose (Zhao et al. 2019, Cai et al. 2019, Korkut et al. 2015). Surface densification is a technology that can improve the mechanical and physical properties of wood, especially surface hardness and bending strength (Gong et al. 2010, Laskowska et al. 2017, Candan et al. 2013). Previous studies on wood densification mostly focused on fast-growing low-density plantation wood species such as poplar, pine, and spruce (Welzbacher et al. 2008, Zaman et al. 2000, Chen et al. 2020). Rubberwood density (592 kg/m^3, oven-dry density) is higher than that 417-435 kg/m^3) of above species. Therefore, surface-densified and heat-treated rubberwood can be used for structural parts, solid wood flooring, and paneling. Wood density has a linear relationship with most mechanical and physical properties. Surface-densified wood is similar to laminated wood composite structure, with densified layers on each surface and a non-densified layer in the core, higher-density surface layers contribute the most mechanical properties to the overall composites (Hao et al. 2016, Anshari et al. 2012). Therefore, the surface-densification could effectively enhance the wood hardness and strength while minimizing volume loss (Sandberg et al. 2013).

In this study, four groups of densification process parameters were selected to create a surface-densified board with different vertical density profiles. The impacts of the value and positions of peak densities on the surface hardness and bending properties of board were studied. Previous researches on the effects of the surface-densification and thermal modification on the wood properties, mainly concentrated on one or two aspects and specifically, in thermal-densification process, a platen cooling phase was applied after hot-pressing, then the platen was heating up again in next cycle (Welzbacher et al. 2008, Chen et al. 2020), which cost much energy and time. In this study, during hot-pressing process after pressure holding phase, the platen temperature cooling phase and the platen heating up again in next cycle were omitted, which could save much energy and time.

MATERIAL AND METHODS

Materials

Kiln dried flat-sawn clear boards of rubberwood (*Hevea brasiliensis*) with a moisture content of 8% was used for the preparation of specimens for thermo-mechanical densification and post-thermal modification. The boards were selected from Danzhou, Hainan Province, China, and had an average oven-dry density of 592 kg/m^3. The boards were prepared to the dimensions 330×100×26 mm (L×T×R). Seven groups of boards (four groups for surface densification, two for surface densification first followed by heat-treated, and one group for control) were prepared, and each group included 8 replicated boards.

Surface densification

Densification process

Rubberwood boards with a moisture content of 8±0.25% were surface-densified in the radial direction at the Laboratory of Nanjing Forestry University using a conventional

laboratory hot-press with an electric heating system and press platen of 500×500 mm. Metal stickers of 20 mm thickness were used to control the target thickness with a nominal compression ratio of 23.1%. The thermo-mechanical densification process consisted of three phases: preheating, compression, and pressure holding. Preheating was used to increase the plasticity of wood, making it softer and easier to densify. To determine the impact of the compression temperature and pressing duration on the dimensional stability and mechanical properties of surface-densified wood, two different temperatures (160°C, 180°C) and two durations (10 s, 60 s) were selected to form four types of densification processes (Tab. 1).

Tab. 1: Parameters for densification process of rubberwood.

Process	Preheating phase	Compression phase		Pressure holding phase		Platen temperature
	time (s)	time (s)	pressure (MPa)	time (min)	pressure (MPa)	(°C)
I	30	10	10	10	1.5	160
II	30	60	10	10	1.5	160
III	30	10	10	10	1.5	180
IV	30	60	10	10	1.5	180

Based on the optimization experiments of previous research in lab-scale, the densification temperature of 160°C was the most suitable for commercial production in industrial scale (Welzbacher et al. 2008). Ten min pressure holding phase is for release the residual compression stress, decrease compression set-recovery and spring back. Then the platen pressure was decreased step by step until 0.5 MPa, afterwards the platen discharged water vapor slightly to decrease the pressure between the densified wood surface and hot platen, for preventing the densified wood surface blistering and splitting. The scheme of the surface densification process I is shown in Fig. 1. In this research the platen temperature cooling phase and the platen heating up again in next cycle were omitted, which could save much energy and time.

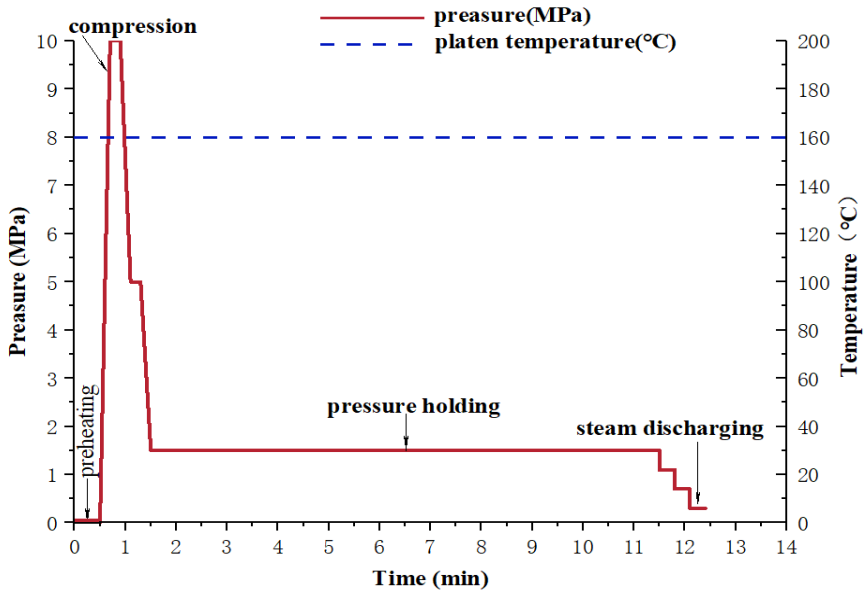

Fig. 1: Scheme of surface densification process I.

Vertical density profiles measurements

The vertical density profiles (VDP) of the wood specimens were measured using an X-ray densitometer (DENSE-LAB Mark3, Germany). Wood density was measured at intervals of 0.05 mm through the thickness of the specimens. Specimens with dimensions of 50×50×20 mm and 50×50×26 mm (L×T×R) were cut from each group of the surface-densified and non-densified board.

Post heat treatment

Thermal modifications were conducted in a superheated steam chamber with an electric heating and steaming system at laboratory scale. Two groups of board 330×100×26 mm (L×T×R) were surface-densified using the parameters of densification process I, then they were piled up in the chamber for superheated steam treatment and conditioning. Two heat-treatment schedules were used. A treatment temperature of 170°C and duration of 1.5 h were used for group 1 (T_1), and a temperature of 190°C and duration of 1.5 h for group 2 (T_2). Conditioning temperature was 110°C for 2 h. Afterwards, the temperature in the chamber was cooled down to 45°C, the boards were stayed overnight in the chamber. The boards were sawn into specimens. The information of specimens for dimensional stability, surface hardness and bending property tests was listed in Tab. 2. All wood specimens except for the dimensional stability specimens were conditioned at 20°C and 65% RH until equilibrium was reached.

Tab. 2: The dimensions and replicates of specimens for different physical and mechanical tests.

Test method	Dimension (mm)			Replicate of specimens
	Long.	Tang.	Rad.	
Dimensional stability	20	20	20	12
Surface hardness	50	50	20	12
Bending properties	300	20	20	12

Dimensional stability determination

Seven groups of specimens 20×20×20 mm were oven-dried at 103±2°C until constant mass. The specimens were then conditioned in a humidity chamber at 40°C and 70% RH until equilibrium was obtained. Three dry-humid cycles progressed. After each cycle, the compression set-recovery (SR) was determined for each specimen based on Eq. 1 and the thickness swelling (TS), width swelling (WS) were calculated according to Eqs. 2,3 resp.:

$$SR = (Tr-Tc)/(T_0-Tc) \times 100(\%) \quad (1)$$

where: T_r is the oven-dried thickness after each cycle (mm), T_c is the oven-dried thickness after surface densification (mm), T_0 is the oven-dried thickness before densification (mm).

$$TS = (Ti-Tc)/Tc \times 100(\%) \quad (2)$$

$$WS = (Wi-Wc)/Wc \times 100(\%) \quad (3)$$

where: T_r , W_i are the wet thickness and width during conditioning after each cycle (mm), T_c, W_c are the oven-dry thickness and width after surface densification, respectively.

Mechanical properties determination

Surface hardness

The surface hardness test was conducted based on ISO 13061-12:2017 using an electronic universal testing machine (SUNS-UTM-50KN). The machine was equipped with an 11.28 mm diameter indenter. The densified surface of the specimen was compressed by an indenter at a rate of 5 mm/min until a displacement of 2.82 mm was obtained. The maximum load was recorded. The surface hardness (N), Hw was calculated using Eq. 5:

$$Hw = K \times F \quad (5)$$

where: Hw is the surface hardness at moisture content w, N; F is the maximum load, N; and K= $\frac{4}{3}$ for the 2.82 mm displacement.

Determination of static bending properties

Static bending tests of the wood specimens were carried out on an electronic universal testing machine (SUNS-UTM-50KN). The modulus of rupture (MOR) and modulus of elasticity (MOE) were determined based on ISO 13061-3:2014 and ISO 13061-4:2014, respectively. Three-point bending was used in both the tests.

Microstructure observation

Scanning electron microscopy (SEM) (Fei Quanta 200, US) was used to characterize the microstructure of the densified and non-densified layers of the wood specimen. The specimen was softened with hot water, and transverse sections of the densified and non-densified layers were cut using a blade. The wood flake samples were dried at 65°C and observed using SEM.

Fourier transform infrared (FTIR) spectroscopy measurement

The four groups of wood specimens were characterized by FTIR spectroscopy (Bruker VERTEX 80V, Germany). Prior to measurement, the four types of specimens were cut into small slices with dimensions of 10×10×1(R) mm. The wavenumber range of the FTIR spectra was 500-4000 cm^{-1} with 32 scans for each slice and 4 cm^{-1} resolution.

Biological durability determination

The biological durability of the four groups of samples was determined according to GB/T 13942.1-2009. Wood samples were cut into specimens of dimensions 20×20×10 (L) mm. 24 specimens for each group were oven-dried at 103±2°C until a constant mass was reached. The oven-dried mass of each specimen is recorded as w_1. Then 48 specimens were inoculated with the white rot fungus *Coriolus versicolor*, and another 48 were inoculated with the brown rot fungus *Gloeophyllum trabeum*. The incubation period was 12 weeks at 28°C and 80% RH chamber. The specimens were then oven-dried and weighed again (w_2). The mass loss rate $(w_1-w_2)/w_1 \times 100\%$ was used to assess durability.

RESULTS AND DISCUSSION

Dimensional stability analyses

The set recovery (SR), thickness swelling (TS), and width swelling (WS) of different groups of specimens were listed in Tabs. 3 and 4 respectively. Set recovery is a measure of the irreversibly recovered thickness in the compression direction due to dry-humid climate change. As shown in Tab. 3, the SR values increased with the number of cycles, which indicated that the deformation of the wood cell wall in the densified layer was slowly recovering, and the mechano-sorption behaviour occurs in deformed wood cells during dry-humid cycles (Laine et al. 2013), due to the release of residual stress. As mentioned previously (Tab. 1), four types of desification process (I, II, III, IV) were did. Comparing the four types of compression processes, the SR of process IV was 7.84%-13.58%, which was smaller than that of process I, because the boards of process IV underwent higher temperatures and longer press times than those of process I (Tabs. 1 and 3). Post-heat treatment is an effective method to decrease the SR of densified board because the mechano-sorption behavior and residual stress of heat-treated wood decreased significantly. This made SR only 3.99% to 7.66%, which decreased by 75.8% to 64.9%, respectively, compared to that of surface densification process I.

Tab. 3: Compression set-recovery of surface-densified (I, II, III, IV), surface-densified and post heat-treated (I,T_1 and I,T_2) wood after cycles 1, 2 and 3 with the RH method.

Process	Cycle1	Cycle2	Cycle3
	Set-recovery (%)	Set-recovery (%)	Set-recovery (%)
Control	/	/	/
I	16.46(1.06)*	16.68（1.45）	21.82（1.43）
II	13.01(1.55)	12,74（1.52）	17.90（1.04）
III	8.96(0.74)	9.41（0.92）	15.44（1.20）
IV	7.84(1.60)	8.24（1.48）	13.58（1.63）
I,T_1	5.78(0.65)	5.45（0.44）	9.61（0.48）
I,T_2	3.99(0.49)	3.12（0.57）	7.66（0.56）

**Standard deviations were in brackets.*

Tab. 4: Thickness swelling (%) and width swelling (%) of seven groups wood after cycles 1, 2 and 3 with the RH method.

Process	Cycle1		Cycle2		Cycle3	
	Thickness (%)	Width (%)	Thickness (%)	Width (%)	Thickness (%)	Width (%)
Control	5.75(0.12)*	6.24（0.65）	4.59（0.25）	5.04（0.70）	4.65（0.20）	5.14（0.74）
I	7.94(0.45)	6.24（0.26）	7.50（0.40）	5.01（0.25）	8.35（0.48）	5.38（0.33）
II	7.67(1.94)	4.51（1.48）	7.09（2.00）	3.80（1.06）	7.86（2.01）	3.99（0.94）
III	5.59(0.17)	3.79（0.21）	4.65（1.42）	2.71（0.20）	5.35（1.62）	3.02（0.22）
IV	5.76(0.80)	3.01（0.74）	5.06（0.77）	1.83（0.76）	5.80（0.83）	2.25（0.78）
I,T_1	5.34(1.14)	2.73（1.05）	4.50（1.15）	1.99（0.65）	5.04（1.08）	2.16（0.67）
I,T_2	4.18(0.25)	2.52（0.30）	3.47（0.26）	1.72（0.30）	3.91（0.25）	2.05（0.33）

**Standard deviations are in brackets.*

The thickness swelling (TS) and width swelling (WS) of wood decreased as the temperature of densification and heat treatment increased (Tab. 4). The thickness swelling and width swelling of post-heat-treated wood were only 4.18% to 3.91% and 2.52% to 2.05%, respectively, which decreased by 27.3% to 15.9% and 59.6% to 60.1%, respectively, compared with non-densified wood. The reason is that after high-temperature treatment, hemicellulose and amorphous cellulose were degraded and the number of moisture-absorbable hydroxyl groups decreased, therefore, the moisture adsorption ability of wood decreased.

Vertical density profiles analyzing

The different vertical density profiles (VDPs) of the surface-densified and non-densified wood are presented in Fig. 2. Each VDP line represented a kind of densification process (Fig. 2, Tab. 1). The line1 indicated the left peak density (PDl) and right peak density (PDr) were close to the surfaces. This was because, after preheating, the wood surface reached a high temperature and contains a certain amount of moisture. Fast loading by platen the two surfaces led to thermal softening of the wood cell wall and densification (Laine et al. 2012, Lenth et al. 2001, Sadatnezhad et al. 2017). For a shorter compression time, VDP was sharp, high, and close to wood surface as shown in Fig. 2 line1. As the compression time and temperature increased, the amorphous hemicellulose and lignin of the subsurface will have lower structural rigidity, resulting in the subsurface densified more readily, as shown in Fig. 2 line 4.

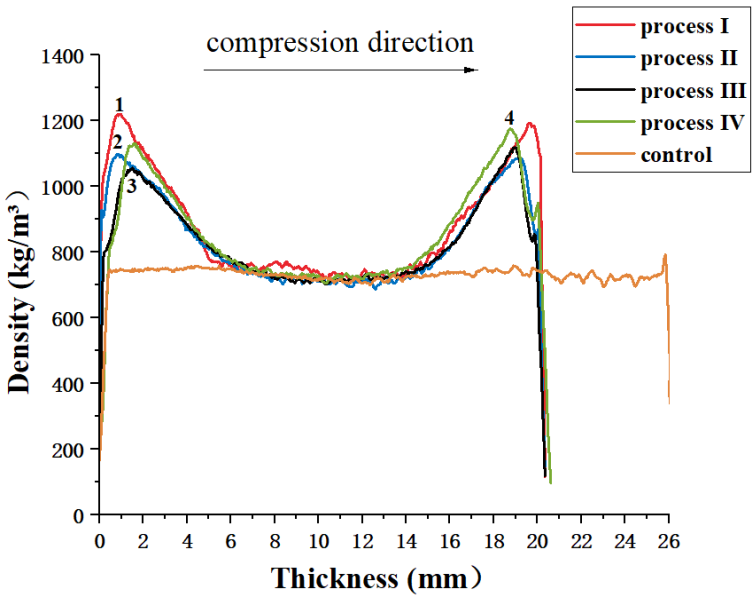

Fig. 2: VDPs of surface densified and non-densified wood specimen along thickness.

Therefore, the compression time and temperature have a significant impact on PDl and PDr as well as the thickness of the densified layer, as shown in Tab. 5. Based on the above analysis, suitable densification parameters can be selected to produce the desired surface-densified wood products and meet the requirements for physical and mechanical properties.

Tab. 5: The density (kg/m³) and thickness (mm) of VDPs of surface-densified and control wood.

Process	PD_l		PD_r		Thickness of den. layer		Thickness of non-den. layer	Average density
	Value	Pos.	Value	Pos.	Left	Right	Core	
I	1219	0.90	1192	0.66	7.09	6.08	7.13	873
II	1096	0.80	1088	1.14	6.24	5.20	8.59	831
III	1055	1.52	1117	1.32	6.61	5.39	8.09	822
IV	1129	1.56	1175	1.85	6.75	6.15	7.61	842
Control	/	/	/	/	/	/	26	723

PD_l - left peak density; PD_r - right peak density; Pos. - position, thickness between the surface and peak-tip.

Mechanical properties analyzing

Surface hardness

The surface hardness of surface-densified (I, II, III, IV), surface-densified and post-heat-treated (I,T_1 and I,T_2) wood and control as well as a comparison with a Chinese hardwood were presented in Fig. 3. The surface hardness value of the non-densified wood was 3426.1 N. The hardness values of the surface-densified wood had a range from 6982.4 N to 7558.3 N, which was 2.0-2.2 times higher than that of non-densified wood. In the four groups of densified wood, the higher the peak density and thickness of the densified layer, the higher the surface hardness. The surface hardness of post-heat-treated (190°C) wood decreased compared to that of surface-densified wood; but it was still 55.9% higher than that of non-densified wood and 7% higher than that of Manchurian ash (a well-known Chinese hardwood) (Chen et al. 2020). Surface hardness increased owing to surface densification, indicating that the structural rigidity and wearability increased, which is beneficial for making solid wood flooring and paneling.

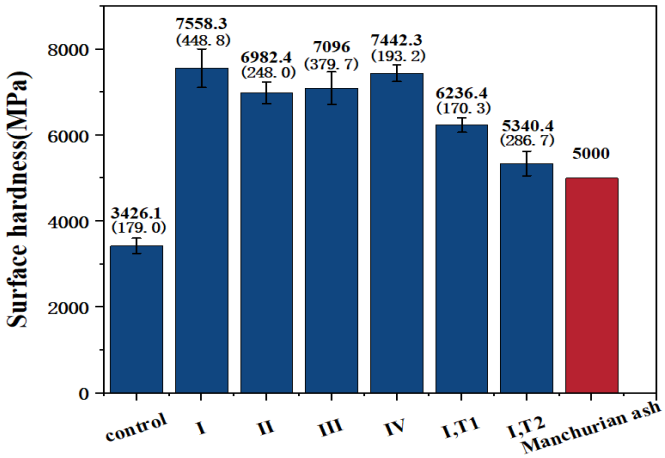

Fig. 3: Surface hardness of seven groups of wood and comparison with a well-known hardwood Manchurian ash (Chen et al. 2020).

Bending properties

The average MOR and MOE values of the seven groups of wood specimens were presented in Fig. 5. The MOR and MOE of non-densified wood were 90.2 and 9808.8 MPa respectively, but that of densified wood were in the range of 146.5-159.9 and 12387.3 - 13593.5 MPa respectively.

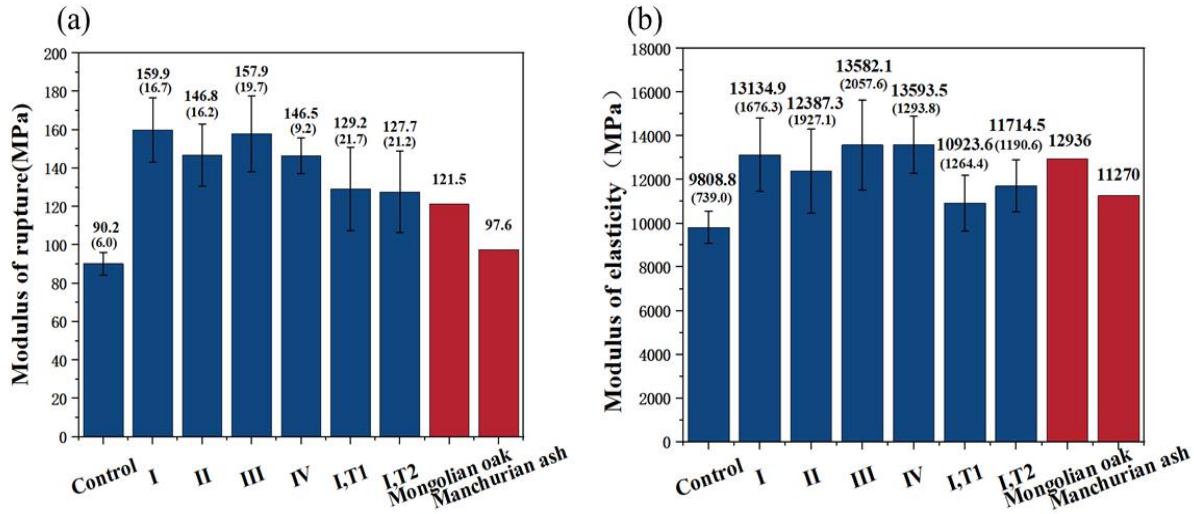

Fig 5. Seven groups of wood and comparison with well-known Chinese hardwood species: (a)modulus of rupture (MOR), (b) modulus of elasticity in static bending (MOE).

Among the four groups of densified wood, the peak density (PD) value and densified layer thickness of the densified wood specimens had an obvious influence on the MOR and MOE of the wood. The higher the PD and densified layer thicknesses, the higher the MOR and MOE of the densified wood. Although the MOR and MOE of post-heat-treated (190°C) wood were lower than those of surface-densified wood, they were still 41.6% and 19.4% higher, respectively, than those of non-densified wood, and even 5.1% and 30.8% higher in MOR than that of Mongolian oak and Manchurian ash, respectively (Chen et al. 1982), because of densification and temperate post-heat treatment. Based on the above analysis, the surface densified wood with process I had better mechanical properties and a densification temperature of 160°C is safer for industrial application; therefore, in the following discussion, densification process I was recommended.

Wood microstructure characterization

In the non-densified layer, the diffused pores of the vessel elements were large, and the wood fibers and rays were arranged in rows, as shown in Fig. 6c and d. However, in the densified layer, the pores of the vessel elements were much smaller and flatter than those of the non-densified layer, and the wood fibers and rays were densified and twisted by hot compression, as shown in Fig. 6a and b. This means that the densified layer contained more vessel elements and wood fibers than the non-densified layer with the same thickness. Wood fibers usually account for 50% of wood volume in hardwood species, and their main function is to support tree trunks and branches; therefore, their number and density have a significant influence on wood density and mechanical properties (Xu 2019). Therefore, the density and mechanical strength of the densified wood layer were much higher than those of the non-densified wood. In addition, it is clear that many wood fibers contained starch granules, which confirms that rubberwood is rich in starch; therefore, it is very easy to decay. Micrographs of the densified and non-densified layers of the surface-densified wood were presented in Fig. 6.

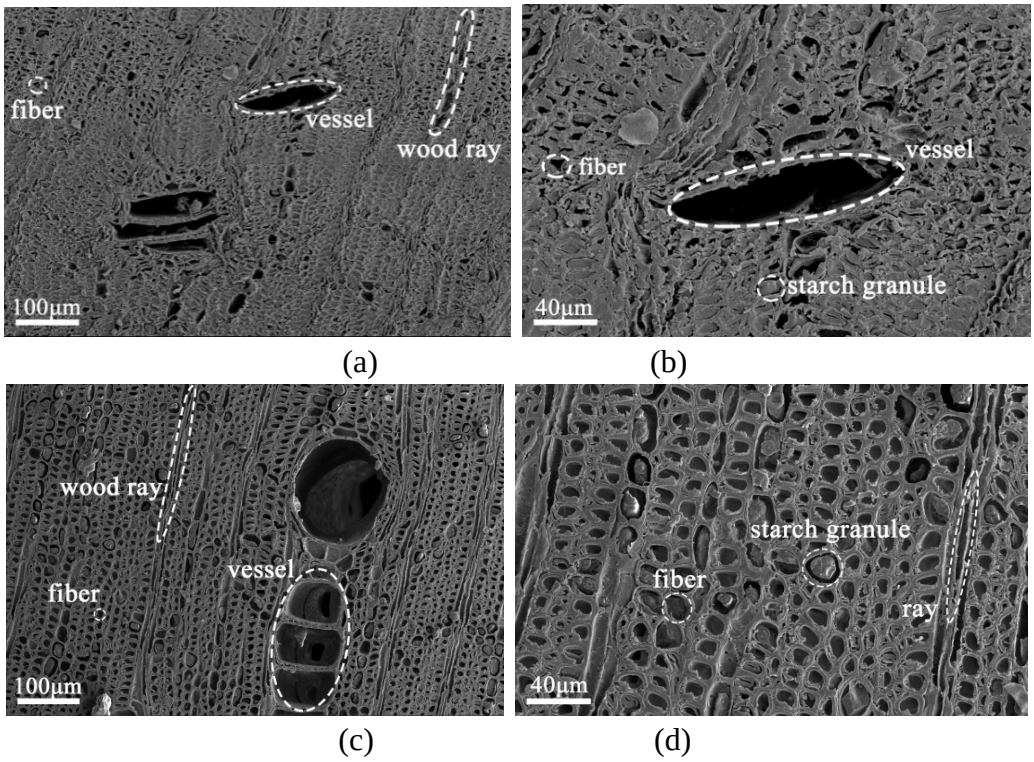

Fig. 6: SEM micrographs of surface densified rubberwood: (a) and (b) - SEM micrographs of densified layer, (c) and (d) non-densified layer.

FTIR spectra analysis

The FTIR spectra of the four groups were shown in Fig. 7. The chemical structure of the high-temperature treated wood exhibited changes compared to the control; therefore, the absorption peak intensities changed, but the wavenumbers of the functional groups generally remained unaltered in the infrared spectrum. Specifically, the band absorption intensity at 1730 cm^{-1} is attributed to the C=O stretching vibration of acetyl groups in hemicellulose, which showed weaker intensity after high-temperature (190°C) treatment, resulting from deacetylation caused by the cleavage of acetyl groups (Tjeerdsma et al. 2005).

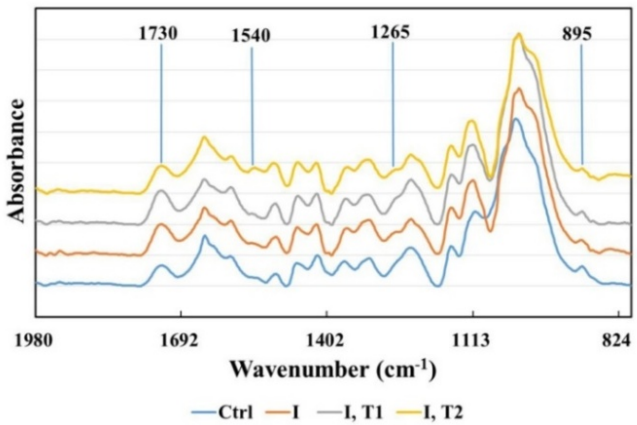

Fig. 7: FTIR spectra for Control (Ctrl), Surface-densified 160°C (I), Surface-densified and post heat-treated 170°C (I, T_1), Surface-densified and post heat-treated 190°C (I, T_2) of rubberwood.

In addition, the band at 1540 cm^{-1} corresponding to the C=C stretching vibration of the aromatic skeleton of lignin and the band at 1265 cm^{-1} were related to the C=O stretch vibration of Guaiacyl nuclei in lignin (Kuo et al. 1988), and both vibration intensities increased due to heat treatment. This was because during heat treatment, hemicelluloses were degraded, which increased the relative proportion of lignin. Furthermore, the band at 895 cm^{-1} was attributed to the C1 group vibration in cellulose and hemicelluloses (Kuo et al. 1988), which showed a weaker intensity after heat treatment because of the degradation of polyose. Based on the above analysis, it was indicated that during high-temperature (190°C) treatment, hemicelluloses degraded, the cellulose and lignin were increased in the relative proportion; moreover, the cross-linking reaction of lignin can increase wood hydrophobicity and consequently dimensional stability (Tjeerdsma et al. 2005).

Durability assessment

Based on laboratory tests of natural decay resistance, the average mass loss rates and corresponding classes of natural durability resulting from *Gloeophyllum trabeum* and *Coriolus versicolor* for the four groups of rubberwood specimens were presented in Tab. 6. It was clear that the mass loss rates of the control rubberwood were very high (45.54%) and (57.42%) respectively, both of which were classified as not durable (class IV).

Tab. 6: Average mass loss rate and corresponding class of natural durability for four groups of rubberwood specimens.

Type of specimen	*Gloeophyllum trabeum*		*Coriolus versicolor*	
	Mass loss rate (%)	Class of durability	Mass loss rate (%)	Class of durability
Ctrl	45.54(1.19) *	IV (Not durable)	57.42 (5.37)	IV (Not durable)
I	36.34(2.85)	III (Slightly durable)	50.00 (2.56)	IV (Not durable)
I,T_1	26.08(3.23)	III (Slightly durable)	47.55 (3.01)	IV (Not durable)
I,T_2	15.43(2.33)	II (Durable)	38.15 (4.89)	III (Slightly durable)

**Standard deviations are in brackets.*

The surface densification (I group) slightly improved the durability; it was classified as slightly durable in the test with *Gloeophyllum trabeum*, However the durability could be improved obviously by surface-densification and post heat-treatment 190°C (I,T_2 group), it was classified as durable (class II) in test with *Gloeophyllum trabeum*. It can meet the durability requirements of solid wood flooring and furnishing for interior usage. Therefore heat-treatment 190°C for 1.5 h was recommended. The reasons for the durability improvement by high-temperature treatment were during high temperature treatment the hemicelluloses were degraded, therefore the energy and metabolites for decay fungal growth decreased. In addition, decreased water absorption of the cell wall might hinder the access of enzymatic agents to cellulose. Furthermore, further cross-linking of the lignin network hindered the enzyme from degrading lignin (Boonstra et al. 2007).

CONCLUSIONS

A high-value three layers rubberwood composite was developed by surface-densification and temperate superheated-steam modification technic. The surface-densified structure can be expressed by the vertical density profile along thickness with densified peak density layers on each surface and non-densified layer in the core, which was controlled by the densification parameters. A short hot-pressing time can increase the peak density value and the thickness of densified layer consequently increase the mechanical properties of wood. In the hot- pressing process for release the residual compression stress and decrease set-recovery ten minutes pressure holding phase was utilized, finally the platen pressure decreasing step by step and steam discharging slight by slight for preventing the densified board warping, blistering and splitting. The platen cooling phase was omitted in this study, which could save much energy and time. After comparison tests, the superheated steam treatment (190°C for 1.5 h) and conditioning (110°C for 2 h) were recommended for releasing residual stress, changing chemical structure of wood, increasing cell-wall hydrophobicity, consequently improve dimensional stability and durability. Comprehensive tests indicated that surface hardness and MOR of combin-modified rubberwood increased by 55.9% and 41.6% compared with non-densified one respectively. Even 5.1% and 30.8% higher in MOR than that of Mongolian oak and Manchurian ash. Set-recovery, thickness and width swelling decreased by 64.9%, 15.9% and 60.1% respectively by heat-treatment. The durability was improved to ‘durable class’. The combin-modified rubberwood could replace valuable hardwood utilize for structure parts, solid wood flooring and panelling for interior environment. Therefore, its application value was promoted significantly.

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QINYI GU, HONGHAI LIU*, TAO LI
NANJING FORESTRY UNIVERSITY
COLLEGE OF FURNISHINGS AND INDUSTRAIL DESIGN
CHINA
*Corresponding author: liuhonghai2020@njfu.edu.cn