Preparation and Properties of Heat-treated Masson Pine (*Pinus massoniana*) Veneer

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The feasibility of heat treatment of Masson pine veneers (MPVs) was evaluated based on mass loss, tensile strength, bending strength, and water absorption of the heat-treated MPVs, and its application in plywood was explored. Fourier-transform infrared and X-ray diffraction results showed that heat-treated MPVs contained a lower amount of hydrophilic groups and had an increased crystallinity. The maximum tensile strength was 59.2 MPa when MPVs were heat-treated at 210 °C for 5.0 min. The corresponding mass loss, water absorption (384 h), and bending strength values were 1.72%, 105.44%, and 83.1 MPa, respectively. Plywood produced from heat-treated MPV (210 °C, 30 min) with the best fungal durability and the lowest shear strength (1.07 MPa) still met the requirements of the Chinese National Standard (GB/T 9846.3-2004, ≥0.80 MPa) for exterior plywood. These results indicate that products based on heat-treated MPV will have increased fungal durability.

Keywords: Mass loss; Fungal durability; Masson pine veneer; Heat treatment; Plywood

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INTRODUCTION

Heat treatment as a wood modification technique has been used extensively for wood protection against fungal attack and for reduction of wood swelling (Repellin and Guyonnet 2005; Korkut *et al.* 2008). In general, wood heat treatment has been conducted under non-oxidative conditions with a heat-transfer medium (such as steam, plant oils, air, and nitrogen) up to 180 and 240 °C (Esteves and Pereira 2009). Based on differences in heat-transfer mediums (liquid or gas), two kinds of equipment (ovens and vessels) are used in the heat treatment process (Esteves and Pereira 2009; Jiang and Lv 2012; Kačíková *et al*. 2013). The durability, color, mechanical properties, composition, and dimensional stability of heat-treated wood using ovens and/or vessels have also been investigated widely (Borrega and Kärenlampi 2008; Windeisen *et al*. 2009; Bak and Németh 2012; Huang *et al*. 2012). Calonego *et al*. (2012) reported on the physical and mechanical properties of *Eucalyptus grandis* wood after heat treatment in an oven with air as the heat-transfer medium. The equilibrium moisture content, volumetric swelling, modulus of rupture, and Janka hardness were found to decrease by 49.3%, 53.3%, 52.3%, and 20.7%, respectively, without changes in the compressive strength parallel to the grain and the respective modulus of elasticity. Bak and Németh (2012) showed that poplar wood has a decreased equilibrium moisture content and increased anti-swelling efficiency during heat treatment in a hot plant oil bath. Cademartori *et al*. (2013) reported on a combination heat treatment (in an oven at 180 to 240 °C and in an autoclave at 127 °C for 4 to 5 h) to decrease the amount of monomeric sugars and increase the hydrophobicity of

Gympie messmate wood. However, these heat treatment processes using an oven and vessel were designed for wood shaped like sticks or blocks and easily lead to flaws, such as cracks and warping, when applied to treat veneers. Press machines have been used widely as universal equipment in the wood industry to prepare wood-based panels. They can also be used to dry veneers. It has been reported that press machines dry veneers at a higher efficiency and with no cracks and warping (Michel 1975). Therefore, press machines may be used as equipment for heat-treating veneers.

Masson pine is the main afforestation tree planted in southern China, with a stock volume that is estimated to occupy one third of the total forest stock volume. Veneers produced from Masson pine logs are the primary raw material for the preparation of exterior plywood such as concrete formworks and laminated veneer lumber, and its service life has been impacted severely by antiseptic and other properties. It has been demonstrated that Masson pine plywood is easily degraded by microorganism attack (*e.g.*, fungus), as a result of the poor antiseptic properties of Masson pine (Laks *et al*. 2002). Thus, improved fungal durability could be expected if heat-treated Masson pine veneers (MPVs) were fabricated to prepare plywood. However, little information is available on heat-treated MPVs.

This work is designed to investigate the feasibility of heat treatment for MPVs. In this study, veneers that were rotary-cut from the sapwood of Masson pine logs were heat-treated under different conditions. The change in functional groups and crystal patterns of the heat-treated MPVs were characterized using Fourier-transform infrared spectroscopy (FTIR) and X-ray diffraction (XRD). The influence of heat treatment temperature (HTT) and time (HTt) on mass loss, tensile strength, bending strength, and MPV water absorption was investigated. The mass loss after fungal attack and the shear strength of plywood manufactured from heat-treated MPVs were also evaluated.

EXPERIMENTAL

Materials

Commercial phenol-formaldehyde adhesive was donated by Zhangzhou Shengmu Forestry Science Co., Ltd. (China). *Gloeophyllum trabeum* (Pers.) Pat. was donated by the Chinese Academy of Forestry. Veneers that had been rotary-cut from the sapwood of Masson pine logs (1.0 m from the stump height) were used as raw materials. These veneers of thickness 1.20 ± 0.05 mm were cut into boards with dimensions of 300 mm × 300 mm.

Methods

Heat treatment of MPVs

Thermal treatment of the MPVs was achieved using a programmable electric laboratory press machine (Xinxieli Machine Manufacturing Co., Ltd., China) with automatic HTT and HTt control. The MPV was oven-dried to 10% to 11% moisture content (MC) and was placed between two heating plates with a controlled constant distance (1.30 mm), HTT, and HTt. After heat treatment, the MPV was cooled to room temperature in a sealed relative humidity RH = 0% desiccator (containing anhydrous $CaCl_2$ desiccant). After approximately 30 min, the MPV was sealed in a polypropylene bag. According to the experimental design, two kinds of heat treatment processes were used: (i) HTt of 30.0 min with HTTs of 160, 170, 180, 190, 200, and 210 °C; and (ii)

HTT of 210 °C with HTts of 0.0 (control), 1.0, 3.0, 5.0, 7.0, and 9.0 min. Each heat treatment technique was repeated eight times.

Preparation of plywood samples

Plywood was prepared according to published work (Chen *et al*. 2013b), with minor modifications. Heat-treated MPV was used to prepare triplicate samples of 3-plywood by coating 160 g/m^2 of phenol-formaldehyde adhesive on each veneer layer. The assembly time, pressing temperature, pressure, and time were set at 30.0 min, 140 °C, 1.0 MPa, and 3.6 min, respectively. The samples were conditioned for one week at room temperature.

Characterization

The MPV samples used for FTIR and XRD characterization were ground into powder. The powders (2 mg) were mixed with potassium bromide (200 mg) at a ratio of 1:100. The mixtures were pressed at 20 MPa for 3 min and then scanned using a Nicolet 380 FTIR (USA) from 500 to 4000 cm^{-1}. The XRD spectra of the MPV samples were recorded using an X-ray diffractometer (X/Pert Pro MPD, Holland) with a CuKα radiation source at 40 kV and 30 mA from 5° to 60° 2θ (step of 0.02° and acquisition time of 40 s). The crystallinity index (CrI) was calculated according to the literature (Li *et al*. 2011), with minor modifications as follows,

$$\text{CrI}\ (\%) = [(I_{002} - I_{am})/I_{002}] \times 100\% \quad (1)$$

where I_{002} is the diffraction intensity from the (002) plane at $2\theta = 26.1°$ and represents both crystalline and amorphous material. I_{am} is the intensity of the background scattering measured at $2\theta = 21.9°$ and represents only amorphous material.

The mass loss of heat-treated MPV was calculated using the following equation:

$$\text{Mass loss}\ (\%) = [m_1 \times (1 - \text{MC}) - m_2]/[m_1 \times (1 - \text{MC})] \times 100\% \quad (2)$$

where m_1 represents the MPV sample mass, m_2 represents the MPV sample mass after heat treatment, and MC represents the moisture content of the MPV sample. Eight test specimens were measured per series.

The tensile strength of the MPV sample was determined using the conditions and methods described by Lang *et al*. (2013). A piece of MPV sample was cut into three specimens with 100 mm × 15 mm dimensions, and their tensile strengths were determined using a tensile testing machine (MTS, USA) with a cross-head speed of 5 mm/min. The number of test specimens for each heat treatment was 24 (3 × 8), and the average tensile strength was calculated.

The bending strength of the MPV sample was evaluated following the procedure described by Murata *et al*. (2013). A piece of MPV sample was cut into three specimens with dimensions 65 mm × 20 mm. The specimens were loaded on a tensile testing machine (MTS, USA) with the tight side on the lower surface (tension part) and the loose side on the upper surface (compression part). A three-point bending test was carried out with a span of 50 mm and a loading rate of 5 mm/min. Twenty-four (3 × 8) test specimens for each heat treatment were prepared, and the average bending strength was calculated.

Water absorption by the MPV samples was determined based on methods found in the literature (Miao *et al*. 2014). Twenty-four (3 × 8) test specimens for each heat treatment were prepared, and the results are presented as the mean of 24 specimens.

The plywood shear strength was determined according to the conditions and methods described by Chen *et al*. (2013b). A piece of plywood was cut into ten 100 mm × 25 mm specimens, which were then soaked in boiling water for 4.0 h and oven-dried at 63 ± 3 °C for 20 h, boiled again for 4.0 h, and cooled at room temperature for 10 min. The shear strength was measured in a tensile testing machine (MTS, USA) at a crosshead speed of 10 mm/min. The number of test specimens for each combination was 30 (10 × 3), and the average wet strength was calculated.

The plywood fungal durability was evaluated as described in the Chinese national standard GB/T 13942.1-2009 (Quarantine 2009), with some modifications. Conical flasks (500 mL) containing sterilized Masson pine sawdust culture were inoculated with *Gloeophyllum trabeum* (Pers.) Pat. and incubated until the Masson pine feeder block was covered before plywood samples (25 mm × 25 mm, three replicates) were introduced. After 12 weeks, the plywood samples were removed from the culture conical flasks, scraped clean to remove superficial mycelium, and oven-dried at 103 °C until their mass stabilized. Each experiment was duplicated. The mass loss caused by fungal attack was calculated according to the formula:

$$\text{Mass loss after fungal attack (\%)} = (m_3 - m_4)/m_3 \times 100\% \qquad (3)$$

where m_3 represents the initial oven-dried mass of the plywood samples before fungal attack and m_4 represents the oven-dried mass after fungal attack.

The collected data were assessed by Microsoft excel 2007 using analysis of variance ($p < 0.05$). When the null hypothesis was rejected, the average values were compared with Tukey Test at the level of significance of 5%.

RESULTS AND DISCUSSION

Ovens and vessels are the primary equipment used for the heat treatment of stick- and block-shaped wood. However, these techniques are unsuitable for heat treating MPV because it deforms. Press machines that contain two heating plates could prevent this MPV deformation. As for other wood-based panels that are prepared using press machines (Chen *et al*. 2012), pressure, HTT, and HTt are the main impact factors that affect the properties of heat-treated MPV during heat treatment. Pressure decreases the MPV thickness and results in an increased cost for the final products. Therefore, MPV compression was avoided by using two press machine heating plates at a constant distance (1.30 mm). FTIR and XRD were used to determine changes in the components of heat-treated MPV at the functional group and crystal structure level, and measurements of the mass loss, tensile strength, bending strength, and water absorption were used to clarify changes in the properties of heat-treated MPVs at the macro-level. Veneer is a primary raw material used to prepare plywood. Hence, potential applications of heat-treated MPV can be evaluated from the shear strength and fungal durability of its plywood.

FTIR Analysis

The FTIR spectra of MPVs are presented in Fig. 1. A comparison of the spectra of five samples shows the following: (i) samples B, C, D, and E had decreased absorption peak areas at 3300 to 3600 cm^{-1} and 1055 cm^{-1} compared to the control sample. This result indicates that the heat-treated MPV contained a lower amount of hydrophilic groups (*e.g.*, hydroxyl groups), which may decrease its water absorption (Huang *et al.* 2012; Chen *et al*. 2013a); (ii) the absorption peak areas at 1736 cm^{-1} decreased in the order C > E > A > D > B, which suggests that the functional group content (C=O) in heat-treated MPV increases as HTt increases, and decreases initially and then increases with increasing HTT. This might result from the degradation of colophony and hemicellulose during MPVs heat treatment; (iii) changes in the feature peaks of cellulose and lignin in Fig. 1 were not obvious, which implies that an extreme chemical reaction may not occur in these components during MPV heat treatment.

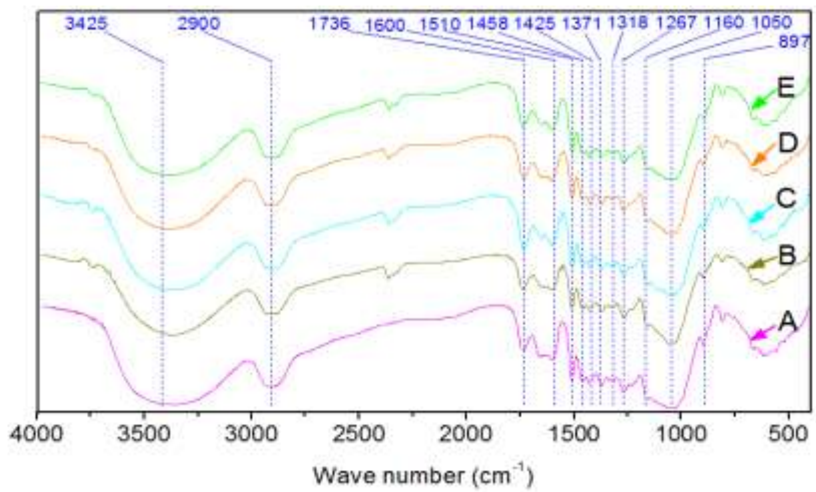

Fig. 1. FTIR spectra of heat-treated MPV samples (A: Control; B: 160 °C, 30.0 min; C: 210 °C, 30.0 min; D: 210 °C, 1.0 min; E: 210 °C, 9.0 min)

XRD Analysis

The X-ray diffractograms in Fig. 2 show that the CrI of cellulose in samples B (160 °C, 30.0 min), C (210 °C, 30.0 min), D (210 °C, 1.0 min), and E (210 °C, 9.0 min) were 54.5%, 62.6%, 54.8%, and 60.8%, respectively. These values were higher than sample A (control, CrI = 53.9%) by 1.11%, 16.14%, 1.67%, and 12.80%, respectively. The crystallinity of cellulose in the heat-treated MPVs therefore increased as the HTT and HTt increased. A comparison of the CrI values of samples B and D shows that the crystallinity of sample D is slightly higher than that of B. This suggests that MPVs that are heat-treated using the techniques of sample D (210 °C, 1.0 min) and B (160 °C, 30.0 min) may have similar mechanical properties, but the heat-treatment techniques of sample D (210 °C, 1.0 min) have better production efficiency.

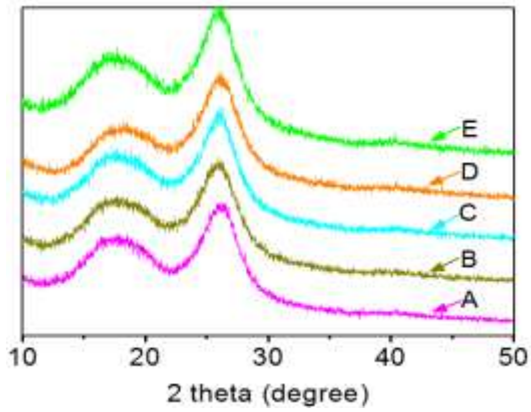

Fig. 2. XRD spectra of heat-treated MPV samples (A: Control; B: 160 °C, 30.0 min; C: 210 °C, 30.0 min; D: 210 °C, 1.0 min; E: 210 °C, 9.0 min)

Mass Loss

As shown in Table 1, mass loss increased with HTT and HTt, which indicates that the pyrolysis component of the heat-treated MPVs increased as the HTT and HTt increased. When the MPV was heat-treated at 160 °C for 30.0 min, its mass loss was 1.37%, which was higher than that of poplar veneer (0.5%) treated under similar conditions (Murata *et al*. 2013). Along with the FTIR analysis above, these results suggest that the additional MPV mass loss may occur because of the volatilization or oxidization of colophony during heat treatment.

Table 1. Some Properties of Heat-Treated MPV Samples

HTT (°C)	HTt (min)	Mass loss (%)	Tensile strength (MPa)	Bending strength (MPa)
control	control	-	44.4±10.6^{a}	53.7±10.5^{a}
160	30.0	1.37±0.42^{a}	42.9±10.2^{b}	56.3±4.8^{b}
170	30.0	1.52±0.45^{b}	39.6±10.9^{c}	75.4±10.2^{c}
180	30.0	1.97±0.49^{c}	34.0±12.2^{d}	98.1±8.2^{d}
190	30.0	2.12±0.50^{d}	35.0±8.3^{e}	102±5.8^{e}
200	30.0	2.19±0.43^{e}	31.8±7.4^{f}	89.2±7.8^{f}
210	30.0	3.21±0.84^{f}	21.6±7.8^{g}	61.4±3.8^{g}
210	1.0	1.55±0.08$^{b\ g}$	54.5±9.7^{h}	71.5±9.7^{h}
210	3.0	1.58±0.24^{g}	56.4±8.7^{i}	78.2±8.2^{i}
210	5.0	1.72±0.53^{h}	59.2±10.9^{j}	83.1±9.2^{j}
210	7.0	2.44±0.25^{i}	53.7±8.9^{k}	73.2±8.9^{k}
210	9.0	2.45±0.52^{j}	49.2±7.1^{l}	65.6±12.2^{l}

Note: same uppercase letters in each columns indicate that there is no statistical difference at level of 5% by the Tukey test.

The mass loss of heat-treated MPV increased with HTT from 200 to 210 °C, which indicates that serious degradation occurred in the MPV components for a HTT higher than 200 °C. The results agree with previously published studies in that the mass loss of *Phyllostachys pubescen* bamboo, which was ascribed to pyrolysis, occurred in the hemicelluloses (Zhang *et al*. 2013). During a HTt of 1.0 to 5.0 min, the variation in mass loss was not obvious. However, with an increase of HTt to 7.0 min, the mass loss increased significantly. This implies that the pyrolysis velocity of components in the veneer was accelerated when the MPVs were heat-treated at 210 °C, but only for a HTt of more than 5.0 min. The MPVs that were heat-treated at 170 to 180 °C for 30.0 min and at

210 °C for 1.0 to 5.0 min had similar mass loss, but those heat-treated at 210 °C for 7.0 min had a mass loss higher than MPVs treated at 200 °C for 30.0 min. These results indicate that HTT affects MPV mass loss more than HTt.

Tensile Strength

Results in Table 1 also show that tensile strength decreased with HTT, but increased first and then decreased with HTt. For samples heat-treated at 160 °C for 30.0 min, the tensile strength decreased by 3.4% compared to control samples. However, when samples were heat-treated from 200 to 210 °C (Table 1), they reached a maximum reduction of 51.4%. This variation in tensile strength is similar to the effect of HTT on mass loss, which suggests that changes in the MPV tensile strength are correlated with hemicellulose pyrolysis (Borrega and Kärenlampi 2008). A maximum tensile strength (59.2 MPa) was achieved when samples were heat-treated for 5.0 min, which implies that the MPV tensile strength was improved by heat treatment with a high HTT and short HTt. This result may occur because of the enhanced elastic extension of heat-treated veneer that is caused by reduced moisture content within the fiber saturation point (Borrega and Kärenlampi 2008; Zhang *et al*. 2013).

Bending Strength

Bending strength increased as HTT and HTt increased, and then decreased at a HTT of approximately 190 °C and HTt of 5.0 min and beyond (Table 1). The bending strength of the heat-treated MPVs was higher than that of the control samples, which indicates that bending strength could be improved during heat treatment. These results are similar to those in literature (Kubojima *et al*. 2000; Poncsák *et al*. 2006). Kubojima *et al*. (2000) reported that the bending strength of Sitka spruce increased first and then decreased with HTT, as a result of the variation or movement of plasticity components (*e.g.*, lignin) in wood during heat treatment. Compared with the control samples, the bending strength brought about by MPV heating at 180, 190, and 200 °C for 30.0 min was 82.7%, 90.0%, and 66.1%, respectively. However, the maximum bending strength that occurred at 210 °C for 1.0 to 9.0 min only increased by 54.8%. This implies that MPVs that were heat-treated at a low temperature for a long time (*e.g.*, 180 °C, 30.0 min) have a better bending strength than those heat-treated at high temperature for a short time (*e.g.*, 210 °C, 5.0 min). This result may be ascribed to the component plasticity (*e.g.*, lignin) in MPV, which results in better malleability when using the heat treatment technique of low temperature over a long time, flowed into the micropores of the heat-treated MPV, and made the wood tissue more homogeneous during heat treatment (Jamalirad *et al*. 2012).

Water Absorption

The water absorption of heat-treated MPVs and control samples soaked in distilled water at 20 °C is shown in Fig. 3. When the soaking time ranged from 192 to 384 h, the amplitude of MPV water absorption was within 1.0% of the MPVs under their initial conditions, whereas the control MPVs reached 10%. This indicates that the water absorption saturated the heat-treated MPVs that were soaked in distilled water at 20 °C for 384 h. Additional water could be absorbed for the control samples under the same conditions. The water absorption of all samples increased rapidly, and some heat-treated MPVs (those heat-treated at 190 to 210 °C for 30.0 min and at 210 °C for 7.0 to 9.0 min) had higher water absorption than the control samples for a soaking time of 24 h. When the soaking time was increased to 384 h, the water absorption of heat-treated MPVs increased slowly and was also lower than the control samples. This may have resulted from the different colophony content in the heat-treated MPVs and the control samples, because some colophony is hydrophobic (Yu *et al*. 2014). Figure 3 also shows that those MPVs that were heat-treated at 210 °C for 9.0 min, and at 200 and 210 °C for 30.0 min, and then soaked in distilled water for 384 h exhibited a small difference (within 2.0%) in water absorption. The effect of HTt on the hydrophilicity of heat-treated MPVs is therefore less than HTT, which is also in agreement with the crystallinity analysis above.

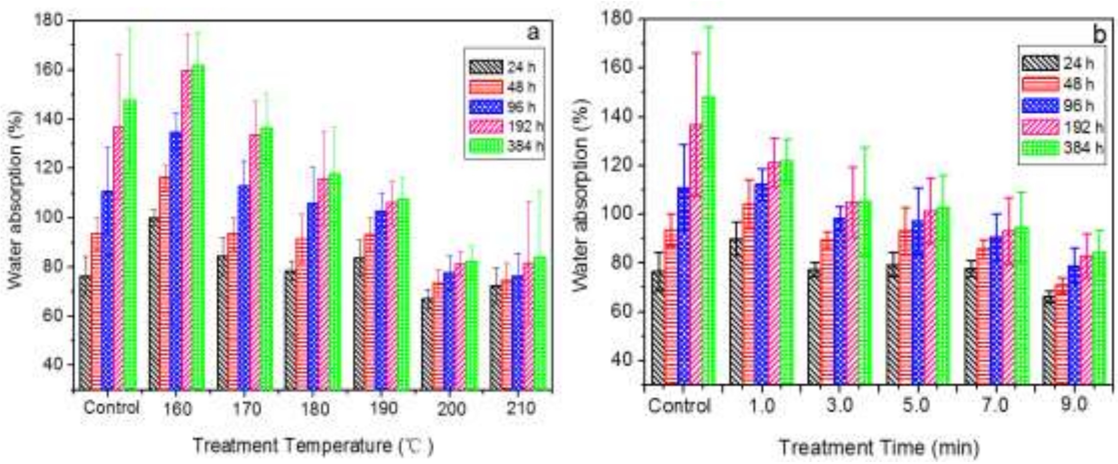

Fig. 3. Effects of HTT (a: with HTt for 30.0 min) and HTt (b: with HTT of 210 °C) on water absorption of MPV

Shear Strength and Fungal Durability

Table 2 displays the test results for plywood manufactured from the control and heat-treated MPV samples. The decrease in shear strength becomes more prominent when HTT and HTt increase. The adhesion interaction of the phenol-formaldehyde adhesive with MPV was therefore influenced adversely by the heat treatment and can be explained by considering the interplay of two competing process. (i) Heat treatment influences the distribution, penetration, and wettability of the phenol-formaldehyde adhesive on the heat-treated MPV adversely because MPV, which is usually hydrophilic, becomes hydrophobic after heat treatment (Ayrilmis *et al*. 2009; Kol *et al*. 2009). (ii) The formic and acetic acids produced by hemicellulose degradation decrease the pH, which hinders the curing of alkaline-catalyzed phenol-formaldehyde adhesive (Kol *et al*. 2009; Jamalirad *et al*. 2012). These results are in agreement with the FTIR and water absorption analyses of the heat-treated MPVs. The shear strength of the control plywood was 1.80 MPa, with decreasing values of 4.4% and 40.6% during heat exposures of 160 °C and 210 °C for 30.0 min, and of 8.3% and 13.9% during heat exposures of 210 °C for 1.0 min and 9.0 min, respectively. This can be explained by the heat-treatment method, as it first degrades the pectins, which make up the pit membranes. Thus, heat-treated MPV has greatly increased permeability, and this may cause heat-treated veneer to have excess adhesive soak into the wood, or "dry out". However, all plywood shear strengths met the Chinese national standard requirements (GB/T 9846.3-2004, ≥ 0.80 MPa) for exterior plywood (Quarantine 2004), which implies that the heat-treated MPV can be used to prepare exterior plywood (*e.g.*, concrete formworks). The variation in mass loss after fungal attack, as shown in Table 2, is similar to the shear strength, which suggests that there was an increased fungal durability of the plywood because of the degradation of hemicelluloses in the heat-treated MPV. The external conditions therefore affected the micro-environment (such as pH and chemicals), and fungal attacks were blocked (Boonstra and Blomberg 2007; Esteves and Pereira 2009).

Table 2. Shear Strength and Mass Loss of Plywood Produced from Heat-treated MPVs and Phenol-Formaldehyde Adhesive

HTT (°C)	HTt (min)	Shear strength (MPa)	Mass loss after fungal attack (%)
control	control	1.80±0.31[a]	16.9±0.8[a]
160	30.0	1.72±0.30[b]	16.6±1.0[a]
210	30.0	1.07±0.21[c]	6.8±0.3[b]
210	1.0	1.65±0.40[d]	11.4±0.6[c]
210	9.0	1.55±0.36[e]	8.1±0.3[d]

Note: same uppercase letters in each columns indicate that there is no statistical difference at level of 5% by the Tukey test.

CONCLUSIONS

1. MPV heat treatment using a press machine resulted in a decrease in hydrophilic functional groups, an increase in cellulose crystallinity, and a degradation of hemicellulose and colophony. However, the chemical variations of cellulose and lignin were not obvious.
2. Heat-treated MPVs with an increase in mass loss and a decrease in water absorption, showed an initial increase in bending strength, followed by a decrease with increasing HTT and HTt. The tensile strength decreased with HTT, but increased initially and then decreased with HTt.
3. Plywood produced from heat-treated MPV and phenol-formaldehyde adhesive had an increased fungal durability and decreased shear strength, but could still be used for exterior plywood.

ACKNOWLEDGMENTS

The authors thank the National Natural Science Foundation of China (31470589) and the Scientific Research Foundation of the Graduate School of Fujian Agriculture and Forestry University for providing financial support for this research.

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Article submitted: January 20, 2015; Peer review completed: April 12, 2015; Revisions received and accepted: April 15, 2015; Published: April 22, 2015.
DOI: 10.15376/biores.10.2.3451-3461