### EFFECTS OF THERMAL TREATMENT OF RUBBERWOOD FIBRES ON PHYSICAL AND MECHANICAL PROPERTIES OF MEDIUM DENSITY FIBREBOARD

N Ayrilmis<sup>1</sup>, S Jarusombuti<sup>2</sup>, V Fueangvivat<sup>3</sup> & P Bauchongkol<sup>3</sup>

<sup>1</sup>Department of Wood Mechanics and Technology, Forestry Faculty, Istanbul University, Bahcekoy, Sariyer, 34473, Istanbul, Turkey. E-mail: nadiray@istanbul.edu.tr

<sup>2</sup>Department of Forest Products, Forestry Faculty, Kasetsart University, Chatuchak, 10903 Bangkok, Thailand <sup>3</sup>Wood Research and Development Office, Royal Forest Department, 61 Phaholyothin Road. Lad-Yao Chatuchak, 10903 Bangkok, Thailand

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**AYRILMIS N, JARUSOMBUTI S, FUEANGVIVAT V & BAUCHONGKOL P. 2011. Effects of thermal treatment of rubberwood fibres on physical and mechanical properties of medium density fibreboard.** This study evaluated effects of thermal treatment of rubberwood fibres at elevated temperatures on physical and mechanical properties of medium density fibreboard (MDF). MDF panels were manufactured from untreated rubberwood fibres and fibres treated at three different temperatures (120, 150 or 180 °C) for 15 or 30 min in a laboratory autoclave. The thickness swelling of the panels decreased with increasing treatment temperature and time while mechanical properties, flexural properties, internal bond strength and screw withdrawal resistance, decreased. Results of the internal bond strength showed that thermal treatment of rubberwood fibres increased the hydrophobicity of fibre surfaces, which reduced the adhesion and penetration of the ureaformaldehyde resin. In general, all panel types met the general purpose use requirements of EN standards. MDF panels made from thermally-treated rubberwood fibres at 180°C for 30 min appeared to be a practical choice for applications requiring low thickness swelling.

Keywords: Thickness swelling, flexural properties, internal bond strength, screw withdrawal resistance, hydrophobicity

AYRILMIS N, JARUSOMBUTI S, FUEANGVIVAT V & BAUCHONGKOL P. 2011. Kesan rawatan terma gentian kayu getah terhadap ciri-ciri fizikal dan mekanik papan gentian berketumpatan sederhana. Kajian ini menilai kesan rawatan terma gentian kayu getah pada suhu yang tinggi terhadap ciri-ciri fizikal dan mekanik papan gentian berketumpatan sederhana (MDF). Panel MDF dihasilkan daripada gentian kayu getah yang tidak dirawat serta yang dirawat pada tiga suhu berbeza (120 °C, 150 °C atau 180 °C) selama 15 min atau 30 min di dalam autoklaf makmal. Pembengkakan ketebalan panel meningkat apabila suhu serta masa meningkat tetapi sebaliknya, ciri mekanik, ciri lenturan, kekuatan ikatan dalaman dan rintangan mengeluarkan skru semuanya menurun. Keputusan kekuatan ikatan dalaman menunjukkan bahawa rawatan terma gentian kayu getah meningkatkan sifat hidrofobik permukaan gentian yang menyebabkan pengurangan rekatan serta penembusan resin urea formaldehid. Secara umumnya, semua jenis panel memenuhi syarat penggunaan umum standard EN. Panel MDF daripada gentian kayu getah yang dirawat pada suhu 180 °C selama 30 min nampaknya merupakan pilihan yang praktikal untuk kegunaan yang memerlukan pembengkakan ketebalan yang rendah.

#### **INTRODUCTION**

Rubberwood (*Hevea brasiliensis*) is the main raw material for wood-based panel production such as particleboard and medium density fibreboard (MDF) in Asia. Projected rubberwood resources for the panel industry in Thailand is approximately 1.13 and 1.93 million m<sup>3</sup> for the years 2007 and 2017 respectively (Hiziroglu et al. 2004). MDF and particleboard produced in Asian countries are mostly used as substrate for thin overlay in cabinet and moulded door skin production.

Thermal treatment, frequently referred to as retification or torrefying, is a process that improves wood performance and leads to improved water repellency, reduced shrinkage and swelling, higher decay resistance, reduced extractive contents, lower equilibrium moisture content and increased thermal insulating capacity (Kamdem et al. 2002, Del Menezzi & Tomaselli 2006). The property change of thermally-treated wood mainly depends on the modification of hemicelluloses, which contribute greatly to sorption of water (Paul et al. 2007). Previous studies reported that thermal treatment improved water resistance of wood-based panels (Tomek 1966, Garcia et al. 2006, Paul et al. 2007). It was reported that the average thickness swelling value of panels made from untreated fibres was 38.7% while that of panels made from treated (180 °C for 30 min) fibres, 24.2% (Garcia et al. 2006). Treated (260 °C for 4 min) oak particleboards showed a reduction of 33% in water absorption and 45-50% in thickness swelling after 24 hours of being soaked in water (Tomek 1966). In another related study, flexural properties and internal bond strength of oriented strand board (OSB) panels were significantly decreased by pre-treatment temperature of wood strands, which was between 220 and 240 °C (Paul et al. 2006). Bending properties of the OSB panels decreased more due to the pretreatment compared with the internal bond (IB). The modulus of elasticity (MOE) was reduced between 6 and 30% compared with non-treated controls while the modulus of rupture (MOR) was reduced even further, i.e. 35 to 50%.

The use of wood-based materials for exterior applications is mainly limited by their irreversible thickness swelling. This causes a loss in their dimensional stability and leads to a weakened structure. Moisture can infiltrate into the core material and, therefore, enables biodegradation by fungal attack. When in contact with water, MDF generally swells more than plywood and a higher proportion of that swelling may not be recoverable after drying. When MDF has contact with water, wood fibres swell and some residual stress created within the fibre mat during hot pressing is released, causing an increase in the thickness of the MDF. Excessive thickness swelling not only causes a poor appearance but also markedly weakens panel products. As with other wood-based panels, MDF panels benefit from improved dimensional stability in applications where it may be subjected to changing moisture conditions.

Rubberwood is highly susceptible to biodeteriorating organisms such as insects and fungi because of its high starch and sugar contents (Edwin & Ashraf 2006). There is a need to increase the dimensional stability and decay resistance of rubberwood in order to make wood-based panels made from its fibres feasible for exterior application. Thermal treatment decreases thickness swelling and enhances durability against microorganisms of wood (Kamdem et al. 2002, Kaygin et al. 2009). Rubberwood treated at high temperatures (above 100 °C) has less hygroscopicity (Sik et al. 2009) but whether similar changes also occur in MDF panels made from thermally-treated rubberwood fibres have not been determined. The objective of this research was to investigate the physical and mechanical properties of MDF panels made from thermally-treated rubberwood fibres at different temperatures and duration.

#### MATERIALS AND METHODS

#### Materials

Rubberwood fibres were obtained from a commercial MDF plant in Thailand. The wood fibres were produced using a thermo-mechanical refining process without any chemical and resin. Moisture content of the fibres, determined by oven-dry weight, was found to be 2-3% prior to treatment. A commercial liquid urea-formaldehyde (UF) resin with 50% solid content was used as adhesive in the manufacture of experimental MDF panels. Ammonium chloride (NH<sub>4</sub>Cl) solution with 20% solid content was used as hardener for the UF resin.

#### Thermal treatment

Wood fibres were treated with saturated steam under pressure at 120, 150 or 180 °C for 15 or 30 min in the laboratory autoclave. The treated fibres were then dried to a moisture content of 2–3% based on oven-dry weight of fibres prior to the panel manufacture.

#### Manufacturing of MDF panels

MDF panels were manufactured in the laboratory of the Royal Forest Department, Bangkok, Thailand, using standardised procedures that simulated industrial production. The fibres were placed in a rotary drum type laboratory blender after which UF resin was added using an airatomised metered spray system at 11% based on the weight of the oven-dried fibres. As a hardener 1% of  $NH_4Cl$  solution based on the resin solid content was added and the fibres were then weighed and formed into mats on an aluminum caul plate using a  $350 \times 350$  mm forming box. The mats were then cold pressed to reduce their heights and make them dense. This procedure allowed for easy insertion of mats (10% moisture content) into the manually controlled, electricalheated press. The temperature of the hot press, its maximum panel pressure and total press cycle were 160 °C, 5 N mm<sup>-2</sup> and 6 min respectively.

A total of 21 experimental MDF panels were tested, i.e. three panels for each level of thermal treatment and control group (Table 1). Except for thermal treatment parameters, other process options such as wood species, resin type (UF), percentage of UF resin and press parameters were the same in all treated panels. The panels (10-mm thick) were then trimmed to a final size of  $300 \times 300$  mm. MDF panels produced were allowed to cool for 72 hours in a climate room set at 65% relative humidity and  $20 \pm 2$  °C before they were cut into test samples. The average density values of the panels varied from 739 to 752 kg m<sup>3</sup>.

#### **Determination of physical properties**

Fifteen samples, each  $50 \times 50 \times 10$  mm, from each type of panel were used for the determination of thickness swelling and water absorption properties. Prior to tests, samples were conditioned in a climatised room at 20 °C and 65% relative humidity. Duration of the conditioning process was determined by regular weighing of samples until no changes in the weights were detected. The thickness swelling and water absorption of samples were evaluated according to EN 317 (EN 1993). For this, samples were immersed vertically in water for 24 hours to determine their thickness and weight. At the end of the 24 hours, the samples were removed from the water and all surface water was wiped off with a dry cloth, and weighed to the nearest 0.01 g and measured to the nearest 0.001 mm. Densities of the samples were determined according to the test method and requirement of EN 323 (EN 1993).

#### Determination of mechanical properties

Flexural properties (MOR and MOE) of samples conditioned to equilibrium at 20 °C and 65% relative humidity were determined according to EN 310 (EN 1993). A total of 12 samples with dimensions of  $250 \times 50 \times 10$  mm, six

parallel and six perpendicular to the panel surface, were tested for each type of panel to determine their MOR and MOE using an Instron testing system equipped with a load cell of 10 kN capacity. The MOR test was conducted in accordance with the third point loading method at a span-to-depth ratio of 20:1. Load-deflection data for the calculation of MOE were recorded at 10 and 40% values of failure load ( $P_{max}$ ). The crosshead speed was adjusted so that failure occurred within an average of  $60 \pm 10$  s.

For the IB tests, 15 samples  $(50 \times 50 \times 10 \text{ mm})$ from each type of panel were tested according to EN 319 (EN 1993). The load was continuously applied to samples throughout the tests at a uniform rate until failure occurred. The speed of the movable crosshead was 1.2 mm min<sup>-1</sup>. To determine screw withdrawal resistance (surface SWR) perpendicular to the plane of the board, 15 samples with dimensions of  $75 \times 75 \times 10$  mm from each type of panel were tested according to EN 320 (EN 1993). The force required to withdraw each screw was recorded in Newton.

#### Statistical analysis

An analysis of variance (ANOVA) was conducted (p < 0.01) to evaluate the effects of temperature and time of treatment on physical and mechanical properties of the panels. Significant differences between the average values of the MDF groups were determined using Duncan's multiple range test.

#### **RESULTS AND DISCUSSION**

# Effects of thermal treatment on physical properties of MDF panels

The thermal treatment of rubberwood fibres showed a highly significant impact on the thickness swelling of the MDF panels after 24 hours of water soaking at a probability level of 0.01 (Table 1). No significant difference in density was observed from thermal treatment levels when compared with control samples. Statistical analysis found significant differences (p < 0.01) between some group averages for the thickness swelling and water absorption values. Significant differences between groups were determined individually for these tests by Duncan's multiple range tests (Table 1). For the thickness swelling values, the control group

Treatment	Physical property				
	Density (kg m <sup>-3</sup> )	Thickness swelling (%)	Water absorption (%)		
Control	751 (39)	23.8 (1.5) a	72.1 (8.2) a		
120 °C, 15 min	740 (38)	21.7 (1.2) b	74.3 (4.3) ab		
120 °C, 30 min	751 (37)	20.6 (1.4) c	75.6 (2.6) abc		
150 °C, 15 min	752 (28)	19.9 (1.8) cd	77.9 (4.9) bcd		
150 °C, 30 min	739 (34)	19.2 (1.3) d	78.7 (8.9) bcd		
180 °C, 15 min	749 (31)	17.8 (1.5) e	79.6 (4.9) cd		
180 °C, 30 min	744 (33)	16.2 (1.6) f	80.3 (4.3) d		
Quality requirements for MDF	> 650, < 800 <sup>1</sup>	Max. 15 <sup>2</sup>	-		

Groups with same letter in a column indicate that there is no statistical difference (p < 0.01) between samples according Duncan's multiply range test; values in parentheses are standard deviations; <sup>1</sup>quality requirement for dry-process fibreboards according to EN 316 (2009); <sup>2</sup>quality requirements for general-purpose MDF panels for use in dry conditions and nominal thickness > 9 to 12 mm according to EN 622–5 (2009).

showed significant differences with all treatment groups. The thickness swelling values of the panels decreased with increasing treatment temperature and time. On the other hand, our results for thickness swelling showed reductions similar to those found by Tomek (1966) for particleboards produced from heat-treated particles. A similar result was also reported by Paul et al. (2007) for OSB made from heat-treated chips. Thickness swelling values in this study are higher than the maximum required by EN 622–5 (EN 2009) for general purpose MDF panels for use in dry conditions, which is 15%. This can be explained by the fact that no wax or other hydrophobic substance was used in the MDF manufacture.

The reduction of thickness swelling as the temperature increased was mainly attributed to hemicellulose which is one of the chemical components of lignocellulosics. As hemicelluloses are very hydrophilic compounds, their alteration could affect the dimensional stability of wood (Yildiz & Gumuskaya 2007). Hemicelluloses which are the most heat sensitive polymers of wood components are hydrolysed during heat treatment, and this decreases the hygroscopicity of the lignocellulosic materials (Del Menezzi & Tomaselli 2006, Winandy & Krzysik 2007). Exposure time and temperature are two important factors affecting hemicellulose degradation. Lower thickness swelling values of MDF panels made from thermally-treated rubberwood fibres were mainly attributed to hemicelluloses being hydrolysed during the treatment.

Water absorption of panels was negatively affected by increasing temperature and time of the thermal treatment. The panels treated at 180 °C for 30 min had the highest water absorption value (80.3%) while the lowest was for the control (72.1%). Wood treated at high temperatures had less hygroscopicity than natural wood. However, it shows a certain porosity and when dipped in water, can absorb more than 20% of water (Vernois 2007). The absorbed water may have occupied void space in the wood and was therefore not directly associated with the fibre and thus did not promote swelling (Winandy & Krzysik 2007). Such behaviour is important for building materials. Similar results were also reported in OSB made from thermally-treated wood chips (Paul et al. 2007). The increment of water absorption in MDF panels made from thermally-treated wood fibres was attributed to the increased void space in the panels after thermal treatment. Results of the water absorption revealed that the porosity of MDF panels increased with the severity of the thermal treatment applied to the fibres.

## Effects of thermal treatment on mechanical properties of MDF panels

MOR and MOE of samples were significantly (p < 0.01) affected by the thermal treatment with

values decreasing with increasing temperature and time (Table 2). Although MOR and MOE values of all panel types decreased, they met the minimum requirements (22 and 2500 N mm<sup>-2</sup> respectively) of EN 622–5 (EN 2009) for general purpose MDF panels for use in dry conditions, except for the MOE of the treatments at 180 °C for 15 and 30 min.

The loss in mechanical properties during heat treatment could be related to the formation of soluble acidic chemicals (e.g. formic acid and acetic acid) from the degradation of hemicelluloses (Garrote et al. 2001). These acids accelerate depolymerisation of the cellulose by breaking down the long chain celluloses (crystalline structure) to shorter chains. Depolymerisation and shortening of the cellulose polymer could affect MOE and MOR of the wood (Rowell 2005). Acidic conditions at elevated temperature degrade wood by hydrolysis and affect the wood strength (Yildiz & Gumuskaya 2007). In addition to treatment temperatures, treatment times also decreased mechanical properties of the MDF panels. In another study, thermal treatment time mainly resulted in softening and degradation of wood and reduction of wood stiffness (Jiang et al. 2009).

The IB strength of panels showed a similar trend to flexural properties (Table 2). The

average IB strength values of panels made from thermally-treated fibres were 15.5 to 44.8% lower than the average of the control panel. The IB strength decreased because rubberwood fibres became inactive as temperature increased. Polarity of the surface of wood exposed to high temperatures is less compared with untreated wood and thus repels water, resulting in a lower wettability (Christiansen 1994, Sernek et al. 2004). Inactivation of the wood fibres results in a loss of bonding ability (Ayrilmis & Winandy 2009). A loss of hygroscopicity is assigned to a gradual loss of wood hydroxyl groups during thermal treatment (Zavarin 1984). With decreasing hydroxyl groups on the fibre surface, hydrogen-bonding sites for water molecules decrease on the wood fibre surface and this results in a higher contact angle value; in the other words, lower wettability. This is one of the mechanisms responsible for poor adhesion of the thermally-inactivated wood. As the UF resin is a polar adhesive, it needs to wet the fibres to acheive adequate bonding and to then develop bonds. It was concluded that rubberwood fibres treated at high temperatures was susceptible to inactivation, particularly if bonded with UF resin. The IB results can be explained in terms of the elevated hydrophobicity of fibre surfaces which reduces the adhesion and penetration of the

Treatment	Mechanical property				
	Modulus of rupture (N mm <sup>-2</sup> )	Modulus of elasticity (N mm <sup>-2</sup> )	Internal bond strength (N mm <sup>-2</sup> )	Surface screw withdrawal resistance (N)	
Control	33.7 (2.6) a	3285 (68.7) a	1.16 (0.11) a	1750 (100.4) a	
120 °C, 15 min	31.7 (1.2) b	2920 (108.7) b	0.98 (0.08) b	1671 (60.8) b	
120 °C, 30 min	30.4 (1.5) bc	2739 (98.9) с	0.93 (0.09) bc	1651 (80.2) b	
150 °C, 15 min	29.5 (1.4) c	2621 (76.0) d	0.88 (0.13) cd	1600 (50.9) c	
150 °C, 30 min	28.8 (1.3) c	2515 (102.2) e	0.84 (0.10) d	1541 (70.3) d	
180 °C, 15 min	26.9 (2.2) d	2455 (90.7) e	0.73 (0.09) e	1500 (80.6) e	
180 °C, 30 min	25.2 (1.5) e	2265 (113.2) f	0.64 (0.07) f	1450 (70.3) f	
Quality requirements for MDF	Min. 22 <sup>1</sup>	Min. 2500 <sup>1</sup>	Min. 0.60 <sup>1</sup>	Min. 1100 <sup>2</sup>	

 Table 2
 Mechanical properties of MDF panels

Groups with same letter in a column indicate that there is no statistical difference (p < 0.01) between the samples according Duncan's multiply range test; values in parentheses are standard deviations; <sup>1</sup>quality requirements for general-purpose MDF panels for use in dry conditions and nominal thickness > 9 to 12 mm according to EN 622–5 (2009); <sup>2</sup>medium density fibreboard (MDF), Grade 130, ANSI A208.2–2002, medium density fibreboard (MDF) for interior applications.

UF resin. The IB values of all panel types were higher than the minimum required by EN 622–5 (EN 2009) for general purpose MDF panels for use in dry conditions, which is 0.60 N mm<sup>-2</sup>.

The average surface SWR values of panels made from thermally-treated fibres ranged from 4.5 to 17.1% lower than the average of the control panels. Although surface SWR of the panels was negatively affected by the thermal treatment, all panel types met the minimum surface SWR (1100 N) requirement for grade 130 MDF (panel thickness  $\leq$  21 mm) specified in ANSI A208.2 (ANSI 2002). These results are in agreement with those reported by Okino et al. (2007) who conducted a study of post heattreated OSB panels. The surface SWR values (1750-1450 N) obtained from the present study were also higher than those (960-1090 N) of commercially manufactured general purpose MDF panels (Ayrilmis 2000).

#### CONCLUSIONS

The thermal treatment of rubberwood fibres significantly lowered the flexural properties, internal bond strength and screw withdrawal resistance of the MDF panels. Results of the internal bond strength clearly indicated that the bonding ability was affected by thermal treatment at elevated temperatures. Thickness swelling of panels significantly improved with increasing temperature and time of treatment. Lower thickness swelling values of the MDF panels made from thermally-treated fibres was mainly attributed to hemicelluloses being hydrolysed during the thermal treatment. MDF panels made from thermally-treated rubberwood fibres at 180 °C for 30 min appeared to a practical choice for applications requiring low thickness swelling.

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