

SURFACE CHARACTERISTICS AND OVERLAYING PROPERTIES OF MDF PANELS MADE FROM THERMALLY TREATED RUBBERWOOD FIBERS

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The objectives of this research were to investigate surface characteristics and overlaying properties of medium density fiberboard (MDF) panels, as affected by thermal treatment of the fibers. MDF panels were manufactured from untreated rubberwood fibers and fibers treated at three different temperatures (120, 150, or 180°C) for 15 or 30 min. Contact angle measurements were obtained by using a goniometer connected with a digital camera and computer system. Roughness measurements, average roughness (R_a), mean peak-to-valley height (R_z), and maximum roughness (R_y), were taken from the sanded samples along and across the sandmarks using a fine stylus tracing technique. With the increasing thermal treatment temperature and time of the fibers, surface roughness of the panels decreased, while their wettability and adhesive bonding strength decreased. Statistical analyses showed significant differences in the surface roughness, contact angle, and adhesive bonding strength of the panels following thermal treatment. Based on the findings obtained from this study, the contact angle and surface roughness parameters of the MDF panels made from thermally treated rubberwood fibers can provide a good information on their ability to bond.

Key words: Adhesive bonding strength; Contact angle; Medium density fiberboard; Rubberwood; Surface roughness; Thermal treatment; Wettability

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INTRODUCTION

Rubberwood (*Havea brasiliensis*) is a main raw material for wood composite panel production in Asia. It plays a vital role as a raw material in the manufacture of wood composites in Thailand. Projected rubberwood resources for the composite panel industry was approximately 1.13 and 1.93 million m³ for the years 2007 and 2017, respectively (Hiziroglu et al. 2004). A majority of medium density fiberboard (MDF) and particleboard produced in Thailand is used as a substrate for thin overlay in cabinet and molded door skin production.

When the panels are used as substrate for thin overlays, their surface characteristics in terms of roughness play an important role in determining the quality of the final product. Standard contact measuring devices employing a stylus tracer, such as used in the metal and plastic industry were successfully employed to evaluate roughness characteristics of various wood composites (Hiziroglu 1996). One of the main advantages of the stylus method is to have an actual profile of the surface and standard numerical roughness parameters, which can be calculated from the profile. Any kind of

irregularities and magnitude of show-trough on the overlaid substrate can be objectively quantified. Therefore, it is important to quantify surface roughness of the panel to have a better overlaying of the substrate. Previous studies reported that surface roughness values of thermally-treated wood and veneer sheets decreased with the increasing treatment temperature and treatment times (Unsal and Ayrimis 2005; Korkut and Akgul 2007; Korkut and Guller 2008).

Wettability is defined as a condition of a surface that determines how fast a liquid will wet and spread on the surface or whether it will be repelled and not spread on the surface. Wettability is crucial for good adhesion in wood bonding. Wettability of the wood can be characterized by various methods (Gray 1962; Casilla et al. 1981; Gardner et al. 1991). Recently, the contact angle method has been commonly used to determine surface characteristics of wood and wood based composites (Sernek 2002; Aydin 2004; Petrissans et al. 2003; Ayrimis et al. 2009a) (Fig. 1). This method is important to determine the adhesive and coating properties of wood and wood-based composite surfaces (Petrissans et al. 2003). When the contact angle is close to zero, perfect wetting of a surface occurs. A wood surface, which is exposed to high temperature condition, can experience surface inactivation (Hakkou et al. 2005). Several known changes, especially oxidation, occur to the wood surface over time during exposure to high temperature. Inactivation of wood surfaces, which results in poor bond quality, is a time-dependent process accelerated by increasing temperature (Aydin 2004). An inactivated wood surface can cause adhesion problems because of the interference with wetting, flow, and penetration of adhesive, and also interfere with the cure and resulting cohesive strength of the adhesive (Aydin 2004). Sernek et al. (2004) reported that drying of wood at temperatures between 160 and 180 °C caused modifications in the surface composition.

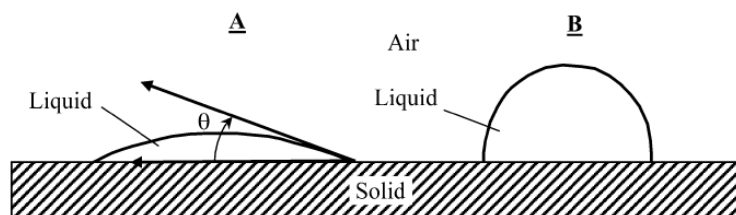


Fig. 1. Apparent contact angle (θ) of a sessile drop resting on a MDF surface, showing a high degree of wetting (A) and a low degree of wetting (B)

Rubberwood is susceptible to the attack of staining fungi and insect borers because of its high starch and carbohydrates content (Edwin and Ashraf 2006). Thermal-treatment improves dimensional stability and enhances durability against micro-organisms of wood and wood based composites (Kamdern 2002; Ayrimis et al. 2009b). It has also been observed that heat-treatment of solid wood at high temperatures causes changes in the surface characteristics and adhesive bonding performance of wood (Podgorski et al. 2000). But it has not been widely studied whether there are similar changes in MDF panels made from thermally treated wood fibers. An extensive literature search did not reveal any information about the thermal treatment on surface characteristics and overlaying properties of the MDF panels made from rubberwood. The objective of this research was to investigate the effects of thermal treatment on the surface characteristics and overlaying properties of the MDF panels. We also aimed to obtain first indications of secondary processing properties of the MDF panels made from thermally treated wood fibers.

EXPERIMENTAL

Materials

Rubberwood fibers (*Hevea brasiliensis*) were obtained from a commercial MDF plant in Thailand. The wood fibers were produced using a thermo-mechanical refining process without any chemical and resin. The moisture content of the fibers, as determined by oven-dry weight, was 2 to 3% prior to treatment. A commercial liquid urea-formaldehyde (UF) resin with 50% solid content was used as an adhesive in the manufacture of the experimental MDF panels. Ammonium chloride (NH_4Cl) solution with 20% solid content was used as a hardener for the UF resin. Rubberwood is composed of fibers (58%), vessel elements (8.5%), axial parenchyma (11.5%), and rays (22%) and are distributed in different patterns and proportions as in other typical hardwood species. The fibers are non-septate, and belong to the medium group with a length of 0.8-1.8 mm. The width of the fibers ranges from 19-27 μm (Mathew 2004).

Thermal Treatment

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



Fig. 2. Laboratory autoclave used for the thermal treatment of the rubberwood fibers

MDF Panel Preparation

MDF panels were manufactured in the laboratory of Royal Forest Department, Bangkok, Thailand, using standardized procedures that simulated industrial production. The fibers were placed in a rotary drum-type laboratory blender, and then UF resin was applied to the fibers, with an air-atomized metered spray system at 11% based on the weight of the oven dried fibers in rotating blender. As a hardener 1% of ammonium chloride solution based on the the resin solid content was added to the UF resin. Fibers were weighed and formed into a mat on an aluminum caul plate, using a 350 mm \times 350 mm forming box. To reduce the mat height and to densify the mats, they were subjected

to a cold-press. This procedure allowed for easy insertion of the mats into the hot-press. Fiber mats having 10% moisture content were subjected to hot-press, using a manually controlled, electrically heated press. The press temperature, maximum panel pressure, and total press cycle were 160°C, 5 N/mm², and 6 min, respectively.

A total of 21 MDF panels, three panels for each level of thermal-treatment and control groups were tested (Table 1). Except for thermal treatment parameters, other process options, such as wood specie, resin type (UF), percentage of UF resin, and press parameters, were unchanged in all treated panels. The resulting MDF panels had 6 to 7% moisture content based on the weight of oven dry fibers. The panels were allowed to cool for 72 hours in the climate room having 65% relative humidity (RH) and 20°C before they were cut into test samples. 10 mm thick panels were then trimmed to a final size of 300 × 300 mm following the cooling process. The average density values of the panels varied from 0.739 to 0.752 g/cm³. The samples used for wettability and surface roughness tests were sanded with a sequence of 100- and 150-grit sand papers.

Determination of Wettability

Contact angle analysis was used to evaluate the wettability characteristics of the panels in this study. The contact angle was defined as the angle through the liquid phase formed between the surface of a solid and the line tangent to the droplet radius from the point of contact with the solid. The contact angles were obtained using with a KSV Cam-101 Scientific Instrument (Helsinki, Finland). A sessile drop method was used to measure a contact angle (θ) of a 5 μ l distilled water drop, which was applied to the surface by means of a pipette. The sample size for the wettability test was 50 mm × 50 × 10 mm. Before evaluating of the wettability tests, the samples were conditioned at 65% RH and 20°C in a climate chamber. An imaging system was used to measure contact angle and shape and size of water droplets for the tested surfaces of the MDF samples. The drop measurements were done from one point of view (one-camera device), since the MDF surface was isotropic (random orientation of the fibers on the surface).

The contact angle measurements were obtained by using a goniometer system connected with a digital camera and computer system. The liquid employed for the measurements was distilled water at 20°C with a surface tension of 72.80 mN/m. The contact angle from the images was measured at 5 s after the 5 μ L droplet of distilled water was placed on the sample surface. Ten samples with a size of 50 mm x 50 mm x 10 mm were taken used from each type of the treatment for contact angle measurements. A total of twenty contact angle measurements, two from each of ten samples, were performed for each type of treatment.

Determination of Surface Roughness

Surface roughness test samples with dimensions of 50 mm × 50 mm × 10 mm were conditioned in a climate chamber having 65% RH and 20°C. Ten samples were used from each type of the treatment for surface roughness measurements. A total of forty roughness measurements (four from each of ten samples: two measurements parallel to the sand marks and two measurements perpendicular to the sand marks from each of the samples) were taken from each type of formulation. A Mitutoyo SJ-301 surface roughness tester, stylus type profilometer, was employed for the surface roughness tests.

Three roughness parameters characterized by ISO 4287 standard (1997), respectively, average roughness (R_a), mean peak-to-valley height (R_z), and maximum peak-to-valley height (R_y) were considered to evaluate the surface characteristics of the

panels. The surface roughness parameters can be calculated from the profiles. R_a is the arithmetic mean of the absolute values of the profile deviations from the mean line and is by far the most commonly used parameter in surface finish measurement. Specification of this parameter is described in previous studies (Hiziroglu 1996; Hiziroglu and Graham 1998; Mummery 1993). Roughness values were measured with a sensitivity of 0.5 μm . Measuring speed, pin diameter and pin top angle of the tool were 10 mm/min, 4 μm and 90°, respectively. The length of tracing line (L_t) was 12.5 mm and the cut-off was $\lambda = 2.5\text{mm}$. Measuring force of the scanning arm on the samples was 4 mN (0.4 gf). Measurements were done at room temperature and pin was calibrated before the tests.

Overlaying of MDF with Veneer Sheet

The top and bottom surfaces of the sanded MDF samples conditioned at 65% RH and 20°C were then overlaid with 0.60 mm thick sliced beech (*Fagus orientalis* L.) veneer having dimensions of 50 mm \times 50 mm \times 10 mm. UF resin was spread on the surface of the MDF samples at the rate of 180 g/m² using a roller prior to curing using a Carver bench-top press at a temperature of 130°C and a pressure of 6.5 N/mm² for 4 min. Ten replicate overlaid samples were made from each type of formulation before adhesive bonding strength was evaluated using the delamination test.

Delamination Strength Between MDF Surface and Wood Veneer Sheet

Adhesive bonding strength between MDF surface and veneer sheet (delamination test) was evaluated on the veneer faced MDF samples according to DIN 68765 B1 (1987). On the surface of the samples, a circle with a 35.7 mm diameter was drilled through the veneer thickness. This veneer circle on the sample surface was separated from the surrounding veneer. A metal tension seal (pull-up seal) was glued with polyurethane adhesive and placed in the movable crosshead of the universal test machine to remove the veneer circle from the panel surface. The force was applied at an even rate and the rate of application was adjusted so the time from the initial application of the force until failure of the test sample was not less than 30 s and not more than 120 s. One measurement from each of the ten replicate samples for each type of formulation was evaluated for adhesive bond strength.

Statistical Analysis

For the surface roughness, wettability, and delamination tests, all multiple comparisons were first subjected to an analysis of variance (ANOVA) at $p < 0.01$ and significant differences between mean values of the control and treated MDF test samples were determined using Duncan's multiple range test.

RESULTS AND DISCUSSION

Surface Roughness and Wettability

Table 1 shows results of the surface roughness parameters of the MDF samples. Statistical analysis showed some significant differences ($p < 0.01$) between the average roughness values of the samples. The control group showed a significant difference compared to all treatment groups. The differences between the treatment groups are given in Table 1 as letters. The panels made from fibers treated at 180°C for 30 min had the smoothest surface with an R_a value of 4.02 μm , while the roughest surface was found for the control panels having an R_a value of 6.93 μm . The R_y and R_z values of the panels also

decreased with the increasing thermal treatment temperature and time. When compared to the control MDF, all surface roughness parameters (R_a , R_y , and R_z values) for the panels were significantly improved by the thermal treatment. This can clearly be observed by inspection of raw data from the surface roughness profilometer that recorded noticeably shallower ridges and valleys when compared to control panels as it traversed the MDF surface at a constant speed (Fig. 3). MDF samples made from thermally treated fibers exhibited a glossy and smooth appearance as compared to the control MDF samples. It was concluded that pressing of the MDF panels made from thermally modified rubberwood fibers resulted in the densification of the panel surface due to increasing elastic properties of the cell walls as compared to the control panels. A lower surface roughness results generally in higher surface density.

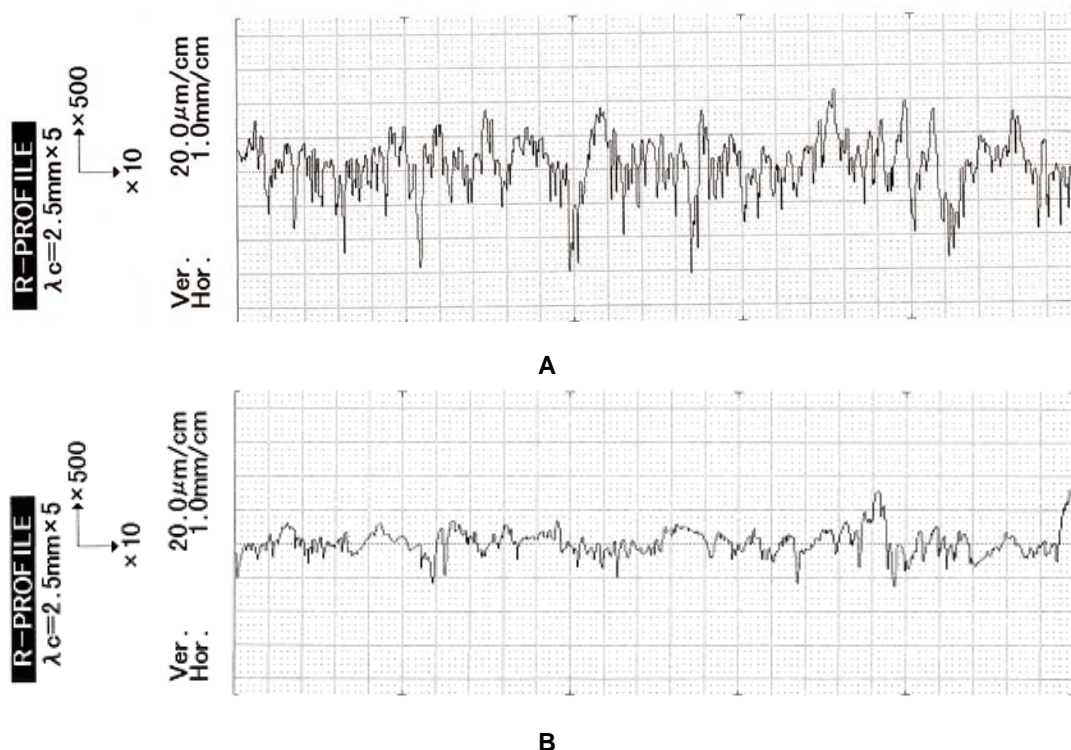


Fig. 3. Typical surface roughness profiles of the control panel (A) and panel made from rubberwood fibers treated at 180°C for 30 min (B)

Wettability

The wettability of the panels was negatively affected by the increasing treatment temperature and time. The contact angle values for all treated panels increased after the thermal treatment. Significant differences between the treatment groups are shown in Table 1 as letters. The contact angle values of the panels made from treated fibers were between 5.7 to 28.8% higher than the average of the control panels. The MDF panels made from untreated fibers had the lowest contact angle value of 91.20°, while the highest contact angle with 117.45° was found for the MDF panels made from fibers treated at 180°C for 30 min.

Table 1. Variations in Average Surface Roughness, Contact Angle, and Adhesive Bonding Strength Values of the MDF Panels as a Function of Thermal Treatment

Thermal treatment	Panel density (g/cm ³)	Surface roughness parameters			Contact angle ^b degree (°)	Adhesive bonding strength (N/mm ²)
		R _a	R _y	R _z		
Control	0.751 (0.039)	6.93 A ^a (0.64)	52.08 A (6.24)	41.15 A (4.83)	91.20 A (5.16)	2.45 A (0.31)
120°C 15 min	0.740 (0.038)	6.41 B (0.47)	49.12 B (5.56)	38.84 B (4.12)	96.44 B (4.77)	2.31 B (0.28)
120°C 30 min	0.751 (0.037)	6.33 B (0.57)	48.18 B (5.17)	38.12 B (3.46)	100.76 C (4.12)	2.18 C (0.22)
150°C 15 min	0.752 (0.028)	5.78 C (0.52)	45.77 C (4.38)	35.12 C (4.37)	101.52 C (5.22)	2.11 C (0.24)
150°C 30 min	0.739 (0.034)	5.08 D (0.49)	43.28 D (4.92)	34.24 C (3.83)	108.68 D (5.45)	1.95 D (0.18)
180°C 15 min	0.749 (0.031)	4.93 D (0.46)	42.82 D (4.15)	32.44 C (2.88)	115.17 E (6.74)	1.89 D (0.26)
180°C 30 min	0.744 (0.033)	4.02 E (0.38)	38.56 E (3.49)	28.06 D (2.56)	117.45 E (7.12)	1.76 E (0.18)

^a Groups with same letters in column indicate that there was no statistical difference ($p < 0.01$) between the samples according to Duncan's multiple range test. Values in parentheses are standard deviations.

Decreasing of the wettability in relation to thermally treated wood fibers can be explained at the cellular level. Hemicelluloses are hydrolyzed during thermal-treatment, and this decreases the hygroscopicity of thermally treated-wood fibers (Winandy and Smith 2006). Exposure duration and temperature are two important factors affecting hemicelluloses degradation (Levan and Winandy 1990). Due to a decrease of hydroxyl groups on carbohydrate chains, the cell wall of wood exposed to high temperatures absorbs less water. This is expected result because wood is a hydrophilic porous composite of cellulose, lignin, and hemicellulose polymers that are rich in functional groups such as hydroxyls that readily interact with water molecules by hydrogen bonding. With the decreasing hydroxyl groups on the fiber surface, hydrogen-bonding sites for water molecules decreased on the MDF surface, and this resulted in a higher contact angle value, in the other words, lower wettability.

It should be noted that the lower wettability of rough surfaces may be due to the higher amount of peaks and valley points on the surface where liquid can be captured by capillary force. Surface roughness was proposed to enhance intrinsic adhesion by providing greater interfacial area and some mechanical interlocking mechanism. A low contact angle is very important to capillary flow in to the complex porous structure of wood to achieve a strong bond between adhesive and material surface. Therefore, the lower contact angle on the surface should be analyzed as a function of surface roughness. The increase in contact angle for the MDF panels made from thermally treated fibers may be interpreted as a decrease in hydrophilicity (Sernek 2002). The surface of wood exposed to high temperatures is less polar and thus repels water, resulting in a lower wettability than in the case of untreated wood (Christiansen 1990; 1994). The wettability change could be due to a modification of the conformational arrangement of wood biopolymers resulting from the loss of residual water or, more probably, from the plasticization of lignin (Esteves and Pereira 2009). Kollmann and Schneider (1963) reported that sorption capacity of wood exposed to various temperatures ranging from

100 to 180°C was decreased. In the same study, it was stated that sorption capacity decreased with thermal treatment temperature and duration.

Thermally treated wood also exhibits lower affinity to water and a strongly modified wettability leading to important changes of its behavior with most coating or gluing processes (Petrisans et al. 2003). A wood surface that is exposed to a high-temperature condition can experience inactivation. Oxidation and/or pyrolysis of wood surface bonding sites are real and inevitable inactivation mechanisms at high enough temperatures and long times. A loss of hygroscopicity is assigned to a gradual loss of wood hydroxyl groups during drying. This is one of the mechanisms responsible for poor adhesion of the thermally inactivated wood. The rate of degradation is much faster at extremely high processing temperature. In a previous study, it was reported that overdrying inactivated the surfaces of Douglas-fir veneer, resulting in poor wettability (Christiansen 1990). Similar results showing negative effect of thermal treatment on wood wettability were also found by many authors (Podgorski et al. 2000; Petrisans et al. 2003; Hakkou et al. 2005; Esteves et al. 2007). Wettability is directly related to the oxygen:carbon (O/C) ratio and inversely related to the C1/C2 ratio (Sernek 2002). The C1 component is related to carbon-carbon or carbon-hydrogen bonds, and the C2 component represents single carbon-oxygen bonds. A low O/C ratio and a high C1/C2 ratio reflects a high concentration of nonpolar wood components (extractives/volatile compounds) on the wood surface, which modifies the wood surface from hydrophilic to more hydrophobic.

Adhesive Bonding Strength between MDF Surface and Veneer Sheet

The adhesive bonding strength values of the MDF panels were significantly affected by the increasing treatment temperature and time. The strength values of the panels made from thermally treated fibers were between 6.1 to 39.2% lower than the average of the control panels. The highest adhesive bonding strength was of 2.45 N/mm² for the control, and the lowest was of 1.76 N/mm² for the panels made from fibers treated at 225°C for 30 min. It is evident that the bonding strength of the panels was decreased with the increasing contact angle. This result was consistent with previous studies (Poncsak et al. 2007; Korkut et al. 2008). The control group showed a significant difference compared to all treatment groups (Table 1).

The adhesive bonding strength results indicated that the hydrophobic character of the MDF made from thermally treated rubberwood fibers could diminish the ability of waterborne thermoset adhesives (aminoplasts) such as UF resin and melamine/urea formaldehyde (MUF) resin to adequately wet the surface and establish physical adhesion. As the UF resin used to adhere the veneer to the MDF was a polar adhesive, it needed to wet the fibers to achieve adequate bonding and to then develop bonds. However, its wetting capability was influenced by a loss in the wettability of the fiber, resulting from thermal treatment. This is why binding the cell polymers after thermal treatment, which are chemically modified compounds, became problematic. Micropore closure also affects adhesive penetration and wetting of the wood cell walls. The closure of larger micropores limits penetration by larger resin molecules, and thus, the bond strength and wood failure decreases (Wellons 1980). This applies particularly in those cases where mechanical interlocking plays an important part of the adhesion. The adhesion between the inactivated wood and MDF panel surfaces may be improved by several means. Treatment with chemicals, such as sodium hydroxide, calcium hydroxide, nitric acid, and hydrogen peroxide can partially improve adhesion (Christiansen 1990). Surface cleaning and

surface removal, for example, by sanding also improve the adhesion between inactivated surfaces.

CONCLUSIONS AND FURTHER WORK

These preliminary findings indicated that the surface characteristics and overlaying properties of the MDF panels were significantly affected by the thermal treatment of the rubberwood fibers. Increasing the severity of the thermal treatment of the rubberwood fibers resulted in smoother surfaces. However, the wettability and bonding strength between the veneer and panel surface were negatively affected by the thermal treatment of the fibers. Based on the findings obtained from the contact angle values, it can be concluded that decreasing in the hydroxyl groups on the fibers resulted in a lower wettability on the panel surface. Wettability and surface roughness of the MDF panels can provide good information on their ability to bond. Further study will focus on new surface treatment applications to improve adhesive bonding and overlaying properties of the MDF panels made from thermally treated rubberwood fibers.

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