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Effects of Post Heat-Treatment on Surface Characteristics and Adhesive Bonding Performance of Medium Density Fiberboard

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A series of commercially manufactured medium density fiberboard (MDF) panels were exposed to a post-manufacture heat-treatment at various temperatures and durations using a hot press and just enough pressure to ensure firm contact between the panel and the press platens. Post-manufacture heat-treatment improved surface roughness of the exterior MDF panels. Panels treated at 225°C for 30 min had the smoothest surface while the roughest surface was found for the control panels. Wettability and the adhesive bonding strength between veneer sheet and panel surface were decreased as post-treatment press temperature increased. A significant relationship ($R^2 = 0.92$) existed between contact angles and adhesive bond strength.

Keywords Bonding strength; Contact angle; Delamination; Heat treatment; Mechanical properties; Medium density fiberboard; Physical properties; Post heat-treatment; Surface characteristics; Surface roughness; Surface wettability; Thermal modification; Thermal treatment; Wood; Wood composite.

1. Introduction

Medium density fiberboard (MDF) is one of the most rapidly growing composite panel product in the market. Smooth and solid edges of MDF can be easily machined and finished, and the uniform surface provides an excellent substrate for painting or applying decorative overlays. However, the disadvantage of MDF compared to plywood is that when MDF has contact with water, it generally swells more than plywood, and a higher proportion of that swelling may not be recoverable after drying. The in-plane movements arised from increased or decreased moisture content of the panel can cause high internal stresses due to the restraint offered by fastening such as nails in construction. These stresses may be large enough to cause buckled panels, pushed-out nails, and separation of the panel from the structure. Swelling and expansion properties, thus, is one of the most important properties of the fiberboards.

Heat treatment improves the hygroscopic characteristics of wood, but it can also change its surface characteristics and, therefore, their wetting properties [1]. A wood surface, which is exposed to high temperature condition, can experience surface inactivation [2, 3]. 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 [4]. 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. Sernek et al. [5] reported that wood drying at temperatures between 160 and 180°C caused modifications in surface composition.

Wettability is crucial for good adhesion in wood bonding. The adhesive has to wet, flow, and penetrate the cellular structure of wood in order to establish intimate contact between molecules of wood and adhesive [6]. There is evidence about the positive relationship between wood wettability and adhesion [7]. Many experiments have shown that high drying temperature reduces the wood adhesive bonding strength, or that high temperature decreases wood hygroscopicity and hinders wettability [3, 8–10]. Wood fibers become hydrophobic after heat treatment, and their wetting capability becomes less after heat treatment [3]. The wettability of wood can be characterized by contact angle analysis. This analysis is important to determine the adhesive and coating properties of wood and wood-based composite surfaces [2]. When contact angle is zero, perfect wetting of a surface occurs. Contact angle is a useful index of adhesive effectiveness.

MDF panels are used to manufacture molding, laminated flooring, overlaid panels for furniture and cabinet industry. When the panels are used as substrate for thin overlays their surface characteristics in terms of roughness play an important role in determining quality of final product. There are various methods to evaluate surface roughness of composite panels, which include acoustic emission, pneumatic, laser, and stylus [11]. The stylus technique is extensively used and well established to quantify surface roughness of industrial metal and plastic parts. The main advantage of the stylus method is having standard numerical

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parameters and profile of the surface. Previous studies reported that surface roughness values of heat-treated wood and veneer sheets decreased with increasing treatment temperature and treatment times [12–14].

From the literature, we knew that post-manufacture heat-treatment decreased swelling, enhanced resistance of the wood-based panels to moisture absorption, and enhanced durability and fungal resistance of materials [15–18]. 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 [8, 9]. But it has not been widely studied whether similar changes in dry process fiberboard (MDF). An extensive literature search did not reveal any information about the effects of post heat-treatment on surface characteristics and adhesive bonding performance of exterior MDF. The objective of this research was to investigate the effects of post-manufacture heat-treatment on the surface characteristics and adhesive bonding strength of the heat-treated MDF panels.

2. Materials and methods

2.1. MDF Panels

Commercially manufactured MDF panels (16-mm thick) for the exterior siding and trim market were used in the experiments. These MDF panels had been bonded with a phenol-formaldehyde (PF) resin and shipped without the coatings or primers typically applied for typical exterior applications. We choose panels bonded with PF resin because PF resin is a more heat-resistant, exterior-type resin. Thirty 1.2 m × 2.4 m commercial MDF panels were then cut into smaller test panels (100 cm × 100 cm). The sixty 100 cm × 100 cm test panels were then randomly assigned to four experimental groups (an control and three levels of heat-treatment).

2.2. Post-Manufacture Heat-Treatment

The MDF panels were loaded into a heated press using a computer controlled single-opening hot press and were thermally treated at either 175°C for 15 min, 200°C for 30 min, or 225°C for 30 min. This press system includes specially designed temperature-pressure probes for measuring internal panel temperature and gas pressure during heat-treatment. A platen contact pressure of 150 (kPa) was applied to provide light but uniform contact between press plates and the panels' surfaces. After heat treatment, all panels were cooled prior to stacking to further minimize fire hazards. 50 mm × 50 mm samples for the surface characteristic properties were cut from each 1 m × 1 m MDF panel. In this study, we used these 50 mm × 50 mm samples to evaluate surface roughness, wettability, and wood veneer-to-MDF adhesive bond strength. The average density values of heat-treated and control panels varied from 0.79 to 0.81 g/cm^3. The treated samples at all temperature levels showed no differences in density when compared to control samples.

2.3. Determination of Surface Roughness of MDF Panels

The eight 50 mm by 50-mm MDF samples (without veneer) from each of the four levels of treatment were used for surface roughness evaluations. A total of six measurements with a 15-mm tracing length, 3 parallel to the sand marks and 3 perpendicular to the sand marks, were taken from each face of the samples. Prior to the test, each sample was conditioned in a climate chamber with a temperature of 20°C and a relative humidity of 65% until they reached to the equilibrium moisture content (12%). The points of roughness measurements were randomly marked on the surface of test samples. A Mitutoyo SJ-301 surface roughness tester, stylus type profilometer, was employed for the surface roughness tests.

Three roughness parameters characterized by ISO 4287 [19] standard, 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 average roughness is by far the most commonly used parameter in surface finish measurement. R_a is the arithmetic mean of the absolute values of the profile deviations from the mean line. Typical roughness profiles of the samples are shown in Fig. 1. The profilometer used for the measurements consisted of main unit and a pick-up which has a skid-type diamond stylus with 5 μm tip radius and 90° tip angle. The stylus traversed the surface at a constant speed of 10 mm/min, and measuring force of the scanning arm on the samples was 4 mN (0.4 gf).

2.4. Determination of Wettability of MDF Panels

The wetting behavior of the heat-treated MDF surfaces without veneer sheets was characterized by the contact angle method. The measurements were conducted with the contact-angle instrument of Kruss GmbH (Easy Drop DSA-2) using distilled water drop at room temperature. Determination of contact angle was performed using the conic section method. 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 image of the liquid drop was captured by a video camera and the contact angle was measured by digital image analysis software. Contact angle from the images was measured at 5 s after the 5 μL droplet of distilled water was placed on the sample surface. Eight 50 mm × 50 mm samples from each of the four treatment groups were used for contact angle measurements of the MDF surfaces without veneers sheets. Sixteen measurements (two from each of eight samples: one measurement for top surface and one measurement for bottom surface) were taken. Following to the contact angle measurements, the droplets (5 μL) on the MDF surfaces were wiped off using an absorbent tissue paper. The samples were then reconditioned in a chamber with a temperature of 20°C and a relative humidity of 65% until they reached to the equilibrium moisture content before the next evaluation of adhesive bonding strength.

2.5. Overlaying of MDF with Veneer Sheet

The top and bottom surfaces of the equilibrated MDF samples were then overlaid with 0.65 mm thick sliced beech (*Fagus orientalis* Lipsky) veneer having dimensions of 50 mm × 50 mm. Urea-formaldehyde (UF) resin was spread on the surface of the MDF samples at the rate of 180 g/m^2 using a roller prior to curing using a Carver bench-top press

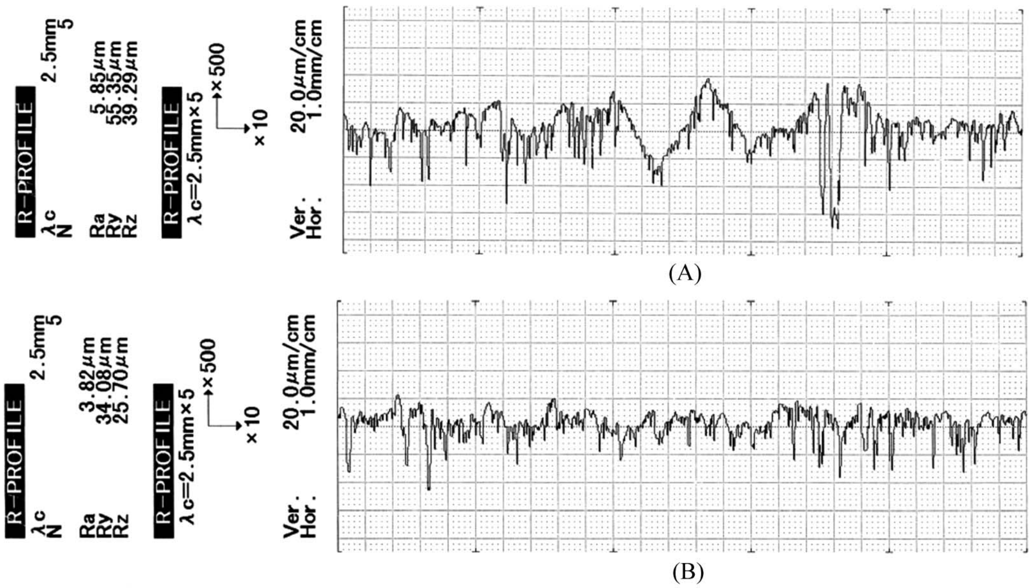

FIGURE 1.—Typical surface roughness profiles of control panel (A) and panel treated at 225°C for 30 min (B).

at a temperature of 130°C and a pressure of 65 bar for 4 min. Eight replicate overlaid samples were made from each of the four treatment groups before adhesive bonding strength was evaluated using the delamination test.

2.6. Delamination Strength between MDF Surface and Wood Veneer Sheet

Adhesive bonding strength between MDF surface and veneer sheet (delamination test) was evaluated on veneer faced MDF samples according to DIN 68765 B1 [20]. 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 MDF 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 MDF 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 eight replicate samples from each treatment group were evaluated for adhesive bond strength.

2.7. Statistical Analysis

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

3. RESULT AND DISCUSSION

3.1. Surface Roughness Parameters

The R_a, R_y, and R_z values of variously heat-treated MDF samples at each exposure condition are shown in Table 1.

TABLE 1.—Variations in average surface roughness, contact angle, and adhesive bonding strength values of the MDF samples as a result of thermal treatment.

Panel type	Heat-treatment level (temperature/time)	Panel density (g/cm^3)	Surface roughness parameters R_a	(μm) R_y	R_z	Wettability (Contact angle method) degree (°)	Adhesive bonding strength (N/mm^2)
A	Control	0.81	5.38 A[1]	53.26 A	40.71 A	95.22 A	2.30 A
		(0.02)	(0.55)	(9.16)	(5.21)	(2.67)	(0.05)
B	175°C/15 min	0.78	4.84 B	46.62 B	35.26 B	114.19 B	2.17 B
		(0.01)	(0.67)	(5.76)	(3.76)	(3.63)	(0.18)
C	200°C/30 min	0.80	4.11 C	43.34 C	32.19 C	122.33 C	1.98 C
		(0.02)	(0.29)	(5.09)	(2.95)	(3.82)	(0.08)
D	225°C/30 min	0.79	3.82 C	41.57 C	30.32 C	130.32 D	1.85 D
		(0.02)	(0.35)	(4.22)	(4.10)	(4.09)	(0.11)

[1]Groups with same letters in column indicate that there is no statistical difference ($pr < 0.01$) between the samples according to the Duncan's multiply range test. Values in parentheses are standard deviations.

In general, smoothness of the panels was improved as heat-treatment temparature increased. The R_a values surface roughness values of all treated MDF panels were lower than control MDF panels. Panels treated at 225°C for 30 min had the smoothest surface with an R_a value of 3.82 μm while the roughest surface was found for the control panels having an R_a value of 5.38 μm (Fig. 1). The average surface roughness value of panels treated at 225°C for 30 min was 29% lower than control samples, followed by panels treated at 200°C for 30 min (23% lower) and 175°C for 15 min (10% lower). This was in agreement with the results of previous studies in solid wood and veneer sheets [12–14, 21]. The R_y and R_z values of the panels also decreased with increasing heat-treatment temperature. When compared to control MDF, all surface roughness parameters (R_a, R_y, and R_z values) for the panels were significantly improved by heat-treatment, but no significant differences were noted between groups heat-treated at temperatures ≥200°C (Table 1). 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.

During heat-treatment, physical and chemical processes occur in layers near the surface that result in a modified surface with new characteristics. After the glass transition temperature (160°C) is achieved, plastification of lignin start to affect the surface characteristics of wood [22]. The heat treatment apparently resulted in such a plastification on the MDF surfaces. High temperatures above 160°C probably caused lignin to reach a thermoplastic condition and thus densify panel surface. Better surface quality of the heat-treated MDF can also be related to this additional surface densification on the face of the MDF panel. The surface-densified MDF samples exhibited a glossy and smooth appearance after heat treatment. Moisture in the manufactured MDF panel is transformed to steam when hot press platens contact to the panel surfaces. This steam tends to soften the fibers near the surface layers also plays a part in MDF surface compaction and plasticization which improves the surface smoothness.

3.2. Wettability

The contact angle values of the heat-treated MDF samples are presented in Table 1. Significant differences ($pr < 0.01$) between all groups were found to exist as determined by Duncan's multiple-comparison tests (Table 1). The contact angle values for all treated panels increased after the heat treatment. The contact angle measurements showed that post heat-treatment had a significant influence on the surface wettability of the treated MDF samples. The average contact angle value of panels treated at 175°C for 15 min was 20% higher than control controls, followed by panels treated at 200°C for 30 min (28% higher), and 225°C for 30 min (37% higher).

The increase in contact angle may be interpreted as a decrease in hydrophilicity [23]. The surface of heat-treated wood is less polar and thus repels water, resulting in a lower wettability than in the case of untreated wood. The hydrophilic character of wood is strongly affected by heat-treatment [1]. The sorption and diffusion properties of the heat-treated MDF panels decreased after heat exposure. In a previous study, wettability decrease was due to the degredation of the most hygroscopic compounds, hemicelluloses, and amorphous cellulose, but also to dehydratation reactions during heat treatment was reported [24]. Plastification of lignin starts affecting particularly the hydrophilic properties of the wood [3]. Peculiar behavior of wettability in relation to heat treatment for the test panels may be explained at the cellular level. Hemicelluloses are hydrolyzed during heat treatment, and this decreases the hygroscopity of heat-treated wood [25]. Exposure duration and temperature are two important factors affecting hemicelluloses degradation. Cumulative heat exposure in the hot-press alters the hemicellulose structure because arabinan and galactan, each a side-chain component of the hemicellulose, tend to be more degraded as both temperature and press duration increase. At very high temperatures, the hemicelluloses may be changed to furfural polymers, which are less hygroscopic. In addition, at high temperatures moisture content strongly catalyzes the depolymerization processes of wood constituents.

Heat-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 [2]. A wood surface, which 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 [26]. Similar results showing negative effect of heat treatment on wood wettability were also found by many authors [2, 3, 9, 24]. Wettability is directly related to the oxygen:carbon (O/C) ratio and inversely related to the C1/C2 ratio [23]. 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.

3.3. Adhesive Bonding Strength between MDF Surface and Veneer Sheet

Adhesive bonding strength of the heat-treated MDF panels decreased with increasing contact angle value. Significant differences between treatment groups were determined individually by Duncan's multiple-comparison tests (Table 1). All treatment groups were significantly different from each other and control group. The test values of all treated panels were between 6 to 20% lower than the average of the control control panels. The highest adhesive bonding strength was of 2.30 N/mm^2 for the control, and the lowest was of 1.85 N/mm^2 for the panels treated 225°C for

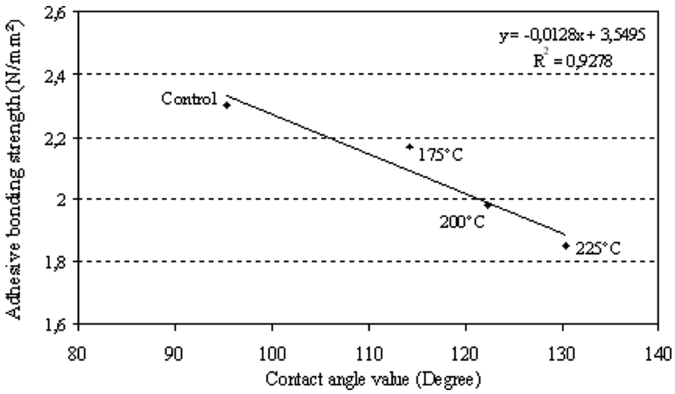

FIGURE 2.—Linear relationship between adhesive bonding strength (delamination of the UF resin bondline between MDF-panel surface and a sheet of beech veneer) and contact angle values.

30 min. A linear relationship ($R^2 = 0.92$) between contact angle and adhesive bonding strength values (delamination strength) was found (Fig. 2). It is evident that the bonding strength of the heat-treated panels was decreased with increasing contact angle. This result was consistent with previous studies [26, 27]. For MDF, this data shows that both changes in the contact angle values and in the rate of change in the contact angle values were a clear indication of how strongly adhesion would develop between surfaces.

The results obtained from our study indicated that the hydrophobic character of the heat-treated MDF could diminish the ability of waterborne thermoset adhesives (aminoplasts) such as urea-formaldehyde and melamine/urea formaldehyde 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 acheive adequate bonding and to then develop bonds. However, its wetting capability was influenced by a loss in the wettability of the fiber resulting from heat treatment. This is why binding the cell polymers after heat treatment, which are chemically modified compounds, became problematic. Micropore closure affects also 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 [28]. 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 [8] can partially improve adhesion. Surface cleaning and surface removal, for example, by sanding also improve the adhesion between inactivated surfaces.

4. CONCLUSIONS

The following conclusions have been drawn from the results of present work:

1. Post-manufacture heat-treatment was a significant factor influencing of surface roughness, wettability, and bond strength of exterior MDF panels. Heat-induced chemical modification resulted in surface inactivation of fibers in layers near the surface.
2. Increasing the severity of post-manufacture heat-treatment resulted in smoother surfaces.
3. The adhesive bonding performance decreased with the increasing contact angle. The bonding strength between veneer and panel surface was adversely affected by the post-manufacture heat-treatment. A significant relationship ($R^2 = 0.92$) was found to exist between the adhesive bonding strength and the contact angle values. This relationship indicates that contact angle could be an indicator for the degree of adhesive bond strength of MDF.
4. We suggest that MDF manufactures use modified glues, paints, or varnishes adapted to these inactivated MDF surfaces to prevent in-service adhesion problems.

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