EFFECT OF POST-THERMAL TREATMENT ON THE DENSITY PROFILE OF RUBBERWOOD PARTICLEBOARD AND ITS RELATION TO MECHANICAL PROPERTIES

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The objective of the study was to investigate the effect of heat treatment on the changes in density profile of rubberwood particleboard samples. The relationship between density profile and mechanical properties of particleboard samples was determined using regression analysis. Single layer particleboard made from rubberwood particles with dimensions of 340 mm × 340 mm × 12 mm and targeted density of 700 kg m^{-3} was heat-treated using oven and hot press at three different temperature levels, namely, 100, 150 and 200 °C for 30 min. All density profile attributes were significantly affected by heat treatment. Bending properties, internal bond strength and hardness were influenced by treatment temperature. However, samples treated using hot press had better mechanical properties compared with those treated using oven. Mean and peak densities exerted profound effects on mechanical properties of the samples.

Keywords: Heat treatment, compression, bending properties, internal bonding, Brinell hardness

INTRODUCTION

Modification of wood changes its chemical constituents and effects its properties. Of the various wood modification techniques, thermal modification is the most well-known and commercialised (Norimoto & Gril 1993). It improves dimensional stability and durability of the wood and wood-based products. Dimensional stability and fungal resistance of oil–heat-treated rubberwood were improved by up to 60 and 36% respectively (Umar et al. 2016).

Apart from wood, heat treatment is also applied to particleboard to improve its dimensional stability (Lee et al. 2015). However, when heated under high temperature some adverse effects on mechanical properties such as reduction in bending and internal bonding strengths of the particleboard have been reported (Carvalho et al. 2015, Lee et al. 2015). In order to maintain the mechanical properties of heat-treated wood, emphasis should be given to the treatment method. In Brazil, post heat-treatment, whereby panels are pressed several times in hot press after conditioning, improved dimensional stability and mechanical properties of the wood (Okino et al. 2007). Modification method involving combination of heat treatment and compression is another effective way to enhance dimensional stability and mechanical properties of wood materials (Wang et al. 2014).

Re-pressing panels during heat treatment causes changes in the physical properties, especially density of the panels. In the manufacture of composite boards such as particleboard, uneven density distribution occurs along the thickness when the board is hot pressed. This density distribution is known as vertical density profile and normally resembles a U shape (Wong 1999). The formation of density profile is highly dependent on the furnish characteristics as well as pressing conditions such

as pressing temperature, duration and pressure (Kollmann et al. 1975). Vertical density profile has been reported to have significant effects on properties of composite boards. High density in the surface layers results in good bending strength and surface finishing characteristics (Chapman 2006). Meanwhile, higher density in the surface layer is accompanied by lower density in the core region, i.e. 60–70% of the former.

Changes in wood–water relationship, temperature and moisture profiles of wood during heat treatment has been studied by several researchers (Almeida et al. 2009, Cermák et al. 2014). However, to our knowledge, information about effect of heat treatment on the density profile and its influence on mechanical properties such as bending strength, internal bond strength and hardness of rubberwood particleboard is scarce. Vertical density profile of medium density fibreboard (MDF) panels manufactured from heat-treated fibres showed no significant difference with the MDF panels made from untreated fibres (Garcia et al. 2006). The relationship between density profiles and mechanical properties of the post heat-treated wood-based products has not yet been studied. Therefore, the main objective of the present study was to evaluate the changes in the density profile and mechanical properties of particleboard induced by heat treatment ranging from 100 to 200 °C in hot press and oven. This study also investigated the specific effects of density profile on mechanical properties of the particleboards.

MATERIALS AND METHODS

Rubberwood core particles with final moisture content of 3% were obtained from Heveaboard Berhad, a particleboard production factory in Gemas, Negeri Sembilan, Malaysia. Single layer particleboards (340 mm long × 340 mm wide × 12 mm thick) with targeted density of 700 kg m^{-3} were produced at the Faculty of Forestry, Universiti Putra Malaysia. Melamine-fortified urea formaldehyde resin with solids content of 60–65% was sprayed during the blending process. The resin concentration used was 8% based on the dry particle weight. Other additives such as wax (0.5% based on dry resin weight) and hardener (ammonium chloride, 1%) were incorporated into the resin prior to blending. Panels were hot-pressed at 180 °C under 4 MPa pressure for 270 s. After pressing, the boards were conditioned for 7 days at a temperature of 20 ± 2 °C and relative humidity of 65 ± 5%. Prior to heat treatment, test samples were prepared according to EN (1993a, b, 2010).

Two types of heat treatment methods, namely, oven method and hot press, were used in the present study. In the oven method, particleboard samples were heated in the laboratory oven at 100, 150 or 200 °C for 30 min. In the hot press method, particleboard samples were treated using hot press at three temperature levels mentioned above for 30 min and pressure of 4 MPa was applied to re-press the samples. A set of untreated panels served as control. A total of 14 particleboards, two for each combination plus two control boards, were made and evaluated in the present study.

After heat treatment, treated samples were reconditioned at 20 ± 2 °C and relative humidity 65 ± 5% prior to testing. Mechanical properties such as modulus of rupture (MOR), modulus of elasticity (MOE), internal bonding strength and Brinell hardness were evaluated according to the European Standards. Five samples from each experimental combination were tested for MOR and MOE in accordance to EN 310 (EN 1993a). MOR and MOE were determined using the equations below:

$$\text{MOR (N mm}^{-2}) = 3P_bL/2bh^2 \quad (1)$$

$$\text{MOE (N mm}^{-2}) = P_{bp}L^3/4bh^3Y_p \quad (2)$$

where P_b = maximum load (N), P_{bp} = load at the proportional limit (N), b = width of sample (mm), h = thickness of sample (mm), Y_p = deflection corresponding to P_{bp} (mm), and L = span (mm).

Internal bonding values were determined in accordance to EN 319 (EN 1993b) using five samples from each experimental combination. The internal bonding strength (IB) was calculated using equation 3:

$$\text{IB (N mm}^{-2}) = P_s/bl \quad (3)$$

where P_s = rupture load (N), b = width of sample (mm), and l = length of sample (mm). Brinell hardness was calculated according to EN 1534 (EN 2010). Resistance to indentation was determined by applying a loaded indenter to the face of the sample. Using a measurement rig, diameter of the residual indentation was

determined to an accuracy of ± 0.1 mm. Brinell hardness (BH) was calculated to two significant digits according to the following formula:

$$BH\ (N\ mm^{-2}) = 2F/[g \times \pi \times D\ (D - \sqrt{D^2 - d^2})] \quad (4)$$

where g = acceleration of gravity (m s^{-2}), π = 3.14, F = max load applied force (N), D = diameter of the indentation ball (mm) and d = diameter of the residual indentation (mm).

Density profiles of the samples were determined using x-ray laboratory density analyser. X-ray was transmitted through the sample along the thickness at 0.2 mm intervals. Particleboard samples (50 mm × 50 mm × 12 mm) were used to determine the density distribution in the samples. The data were analysed statistically using analysis of variance (ANOVA) computed by Statistical Analysis System (SAS) software to verify the significance of the variables. Regression analysis was performed to assess significance of relationship of relevant variables. Duncan's multiple range tests were then used to further determine the levels of significance of average values for each treatment.

RESULTS AND DISCUSSION

Density profile

Figure 1 exhibits the density profiles of the particleboard samples treated using different

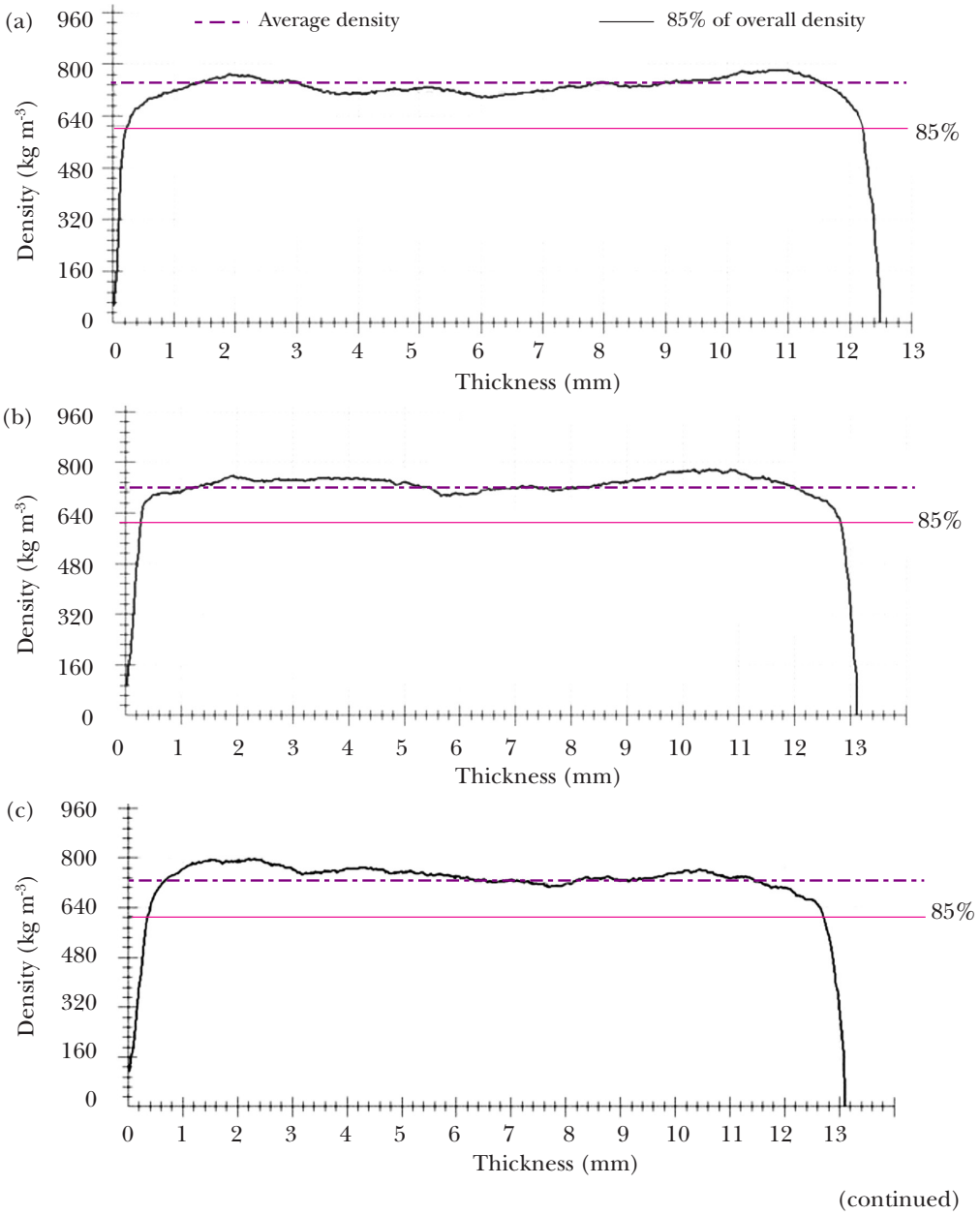

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Figure 1 (continued)

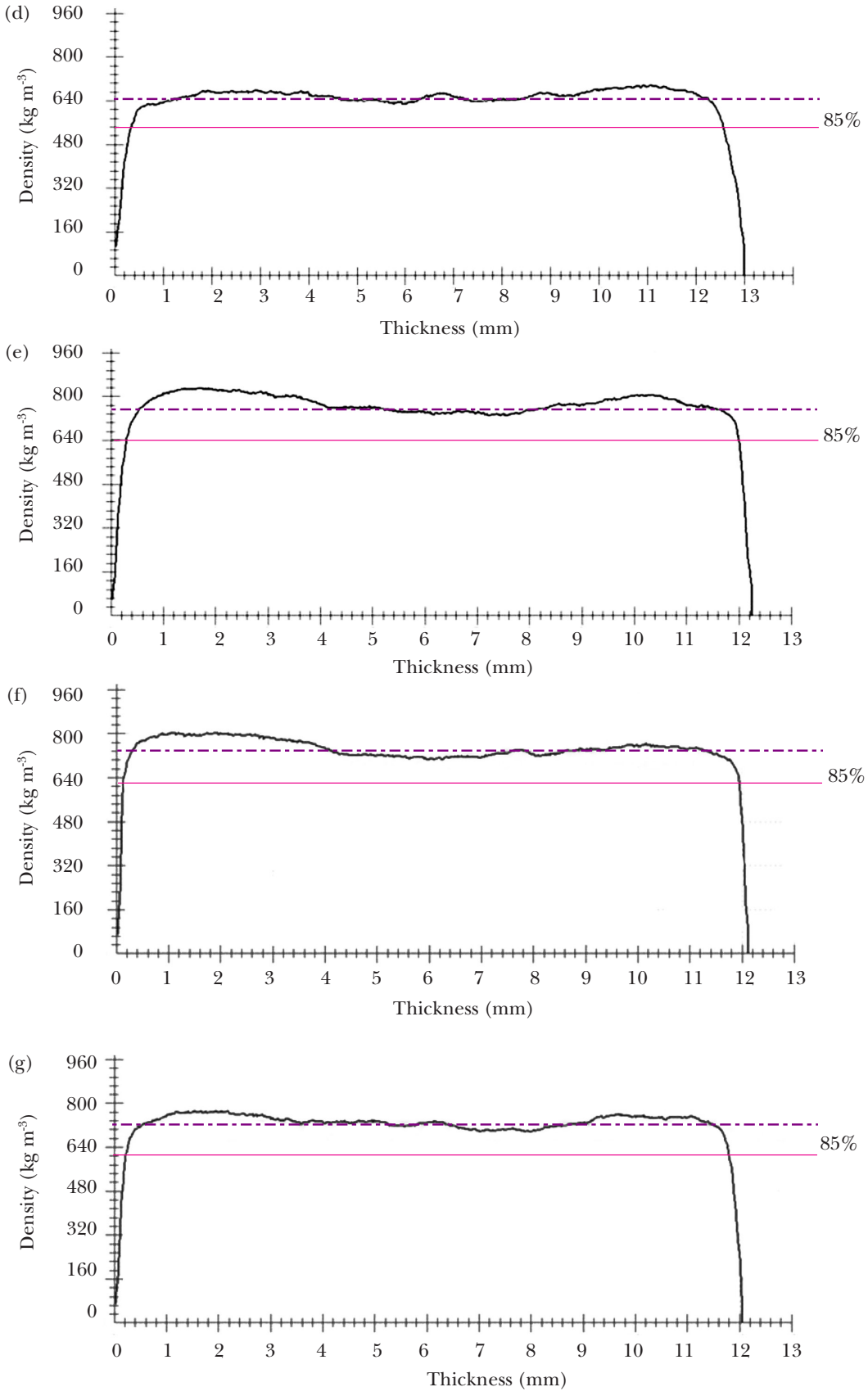

Figure 1 Typical density profile of particleboard sample: (a) control, (b) oven, 100 °C, (c) oven, 150 °C, (d) oven, 200 °C, (e) hot press, 100 °C, (f) hot press, 150 °C and (g) hot press, 200 °C

methods and temperatures. The samples had U-shaped density profiles which indicated regular density gradient across the panel thickness. Lowest density was observed in the core layer while two peak densities were found in both outer layers. Peak density refers to the mean of highest density measured within each half of the profile while mean density refers to the average density of the sample and peak distance denotes the distance of peak density from the board surface (Wong 1999).

Summary of ANOVA results of treatment method and effects of temperature on peak density, mean density, peak distance and mass loss are given in Table 1. Treatment method significantly affected all density profile attributes as well as mass loss. On the other hand, treatment temperature exerted significant influence on all dependent variables with peak distance as an exception.

Average values of peak density, mean density and peak distance of the samples treated using different methods and temperatures are shown in Table 2. Mean density of the samples treated using hot press increased in comparison with control samples. Particleboard samples treated at 100 and 150 °C using hot press showed increased peak density and a slight reduction was observed when the samples were treated at 200 °C. Peak density and mean density of the samples treated using oven increased slightly when treated at 100 °C but started to reduce when treatment temperature was elevated to above 150 °C. Peak distance decreased when samples were hot pressed. Mean density and peak density were higher in the samples treated using hot press. Increasing treatment temperature reduced mean density and peak density values of samples. The highest reduction in mean density was observed in samples treated at 200 °C, i.e. 10.6% reduction compared with control.

Increment in volume was the main reason that led to reduced mean density in the samples treated in oven. Final thickness of the oven-treated samples was higher than that of control and hot pressed (Figure 1). An increase in volume meant a decrease in density. Heat-induced mass loss also reduced mean density of the samples. Loss of mass and density increased with increasing treatment temperature (Table 3). This finding was in agreement with Rusche (1973) who suggested that treatment method, temperature and time were crucial factors that decided the extent of reduction in mass during heat treatment. Samples treated using oven experienced higher mass loss although the differences between these two methods were not significant. Increase in mean density was observed in the samples treated using hot press. Volumetric shrinkage due to reduction in thickness of samples treated using hot press compensated the mass loss induced by heat treatment. Therefore, reduction in volume increased the final density of samples. Mean density and peak density of samples treated using hot press increased when subjected to 100 and 150 °C (Table 2). However, as the treatment severity increased (200 °C), more mass was lost and consequently lower peak density was observed in comparison with control samples.

Treatment method significantly affected peak distance of the samples. Samples treated using hot press had peak distance closer to the board surface compared with those treated using oven. Thickness of samples expanded after treatment in oven due to spring back which pushed peak densities farther from the board surface. On the contrary, re-pressing samples during the treatment in hot press reduced thickness of the samples and brought the peak densities closer to the board surface.

Mechanical properties as function of heat treatment

MOR was significantly affected by treatment method and temperature (Table 4). Only a small change in MOR was observed in wood steamed at 180 and 200 °C, while MOE experienced greater reduction when subjected to the same treatment temperature. Mechanical properties of untreated and treated particleboard samples are presented in Table 5. Internal bonding of the samples treated using oven was significantly decreased with increased temperature. No reduction in internal bonding was observed in the samples treated using hot press. The improvement in internal bonding of the samples treated using hot press was probably caused by re-pressing or re-consolidation during treatment. Internal bonding is directly proportional to core density of the board, which is equivalent to mean density in homo-profile boards with a uniform density profile (Wong 1999). Therefore, increase in mean density due to re-pressing resulted in better internal bonding.

Table 1 Analysis of variance of the treatment method and temperature effects on peak density, mean density, peak distance and mass loss of particleboard samples

Source	df	Pr > F					
		PD (L)	PD (R)	MD	Pdi (L)	Pdi (R)	Mass loss
Method	1	0.000**	0.000**	0.000**	0.001**	0.049**	0.009**
Temperature	2	0.000**	0.000**	0.000**	0.081	0.985	0.000**
Method × temperature	2	0.018**	0.001**	0.001**	0.197	0.420	0.924

** = significant at $p \leq 0.05$, df = degree of freedom; PD (L) = peak density (left), PD (R) = peak density (right), MD = mean density, Pdi (L = peak distance (left), Pdi (R) = peak distance (right)

Table 2 Average values of peak density, mean density and peak distance as functions of treatment method and temperature

Treatment method	Temperature (°C)	PD (L) (kg m^{-3})	PD (R) (kg m^{-3})	MD (kg m^{-3})	Pdi (L) (mm)	Pdi (R) (mm)
Control	0	783.86 bc	769.46 b	715.98 b	1.90 b	2.06 ab
		(13.25)	(19.87)	(21.66)	(0.43)	(1.01)
Oven	100	796.85 bc	764.34 b	722.16 b	2.22 bc	2.50 ab
		(15.39)	(26.62)	(19.82)	(0.68)	(0.61)
Oven	150	781.19 bc	758.04 b	715.96 b	1.94 b	2.32 ab
		(11.00)	(18.89)	(17.03)	(0.74)	(0.96)
Oven	200	697.13 d	678.93 c	640.08 c	2.90 c	2.63 b
		(29.81)	(20.56)	(20.79)	(0.66)	(0.91)
Hot press	100	831.85 a	807.55 a	762.69 a	1.86 b	1.96 ab
		(25.26)	(10.68)	(18.08)	(0.69)	(0.70)
Hot press	150	805.54 b	763.11 b	737.46 b	1.04 a	1.30 a
		(10.61)	(20.04)	(6.17)	(0.32)	(0.19)
Hot press	200	774.02 c	761.95 b	719.44 b	1.54 ab	1.96 ab
		(20.98)	(22.81)	(15.58)	(0.36)	(1.23)

Numbers in parentheses are standard deviation values, means followed by the same letter in the same column are not significantly different at $p \leq 0.05$; PD (L) = peak density (left), PD (R) = peak density (right), MD = mean density, Pdi (L) = peak distance (left), Pdi (R) = peak distance (right)

Table 3 Mass changes of the samples after heat treatment

Treatment method	Temperature (°C)	Mass loss (%)
Oven	100	0.96 a (0.17)
Oven	150	2.81 b (0.38)
Oven	200	3.76 c (0.32)
Hot press	100	0.68 a (0.48)
Hot press	150	2.45 b (0.15)
Hot press	200	3.36 c (0.37)

Numbers in parentheses are standard deviation values, means followed by the same letter in the same column are not significantly different at $p \leq 0.05$

Table 4 Analysis of variance (ANOVA) of the treatment method and temperature effects on internal bond (IB), bending strength and hardness of particleboard samples

Source	df	Pr > F			
		IB	MOR	MOE	BH
Method	1	0.002**	0.206	0.048**	0.001**
Temperature	2	0.007**	0.152	0.037**	0.000**
Method*Temperature	2	0.432	0.700	0.589	0.027**

** = significant at $p \leq 0.05$, df = degree of freadom; MOR = modulus of rupture, MOE = modulus of elasticity, BH = Brinell hardness

Table 5 Average values of internal bond (IB), modulus of rupture (MOR), modulus of elasticity (MOE) and Brinell hardness (BH) as functions of treatment method and temperature

Treatment method	Temperature (°C)	IB (N mm^{-2})	MOR (N mm^{-2})	MOE (N mm^{-2})	BH (N mm^{-2})
Control	0	1.15 b	13.36 a	2180.60 ab	0.33 bc
		(0.24)	(1.98)	(195.30)	(0.05)
Oven	100	1.29 ab	14.00 a	2203.40 ab	0.44 b
		(0.36)	(1.63)	(241.39)	(0.12)
Oven	150	1.14 b	13.68 a	2160.60 ab	0.32 bc
		(0.26)	(1.76)	(215.98)	(0.09)
Oven	200	0.78 c	13.10 a	2056.40 b	0.26 c
		(0.11)	(2.22)	(130.61)	(0.02)
Hot press	100	1.61 a	15.45 a	2440.00 a	0.82 a
		(0.40)	(0.69)	(100.35)	(0.27)
Hot press	150	1.33 ab	14.56 a	2320.00 ab	0.46 b
		(0.21)	(2.12)	(237.69)	(0.07)
Hot press	200	1.29 ab	13.24 a	2110.40 b	0.32 bc
		(0.12)	(1.57)	(210.91)	(0.02)

Numbers in parentheses are standard deviation values, means followed by the same letter in the same column are not significantly different at $p \leq 0.05$

Values of MOR decreased as treatment temperature increased (Table 5). However, no significant reduction was observed in heat-treated samples compared with control. MOR values of samples treated at 200 °C using oven and hot press reduced by 1.95 and 0.90% respectively. Similar to MOR, MOE values were significantly influenced by the increasing treatment temperature. Great reduction was observed when treatment temperature increased from 150 to 200 °C. This behaviour can be related to the cellulose in the samples. Mechanical property of cellulose microfibrils was the main factor that affected MOE. Cellulose is not degraded at low temperatures, and less or no change in MOE is observed up to 160 °C (Kollmann & Fengel 1965).

Hardness of samples decreased as the treatment temperature increased (Table 5). Reduction in hardness can be related to the increase in brittleness due to chemical modification induced by heat treatment (Bakar et al. 2013). In general, mechanical properties of particleboard samples degraded after heat treatment. Heat treatment induces degradation of hemicellulose which leads to loss of mechanical strength (Boonstra & Tjeerdsma 2006). Overall, samples treated using hot press had better mechanical properties compared with samples treated using oven. Re-pressing improved mechanical properties of samples because it increased the density of the samples and counterbalanced the loss of strength caused by reduction in mass and density after

heat treatment. During heat treatment in oven, cleavage of the carbohydrates could be the main reason that led to reduction in board density and shear strength of wood particles, which subsequently resulted in strength loss in treated particleboard (Boonstra et al. 2006).

Mechanical properties as function of density profile

Peak density and mean density are regarded as the dominant factors that influence the properties of particleboard (Wong et al. 1998). Thus, regression analysis was carried out to determine the correlation between peak and mean densities and mechanical properties of the particleboard. Internal bonding was linearly correlated to peak density and mean density (Figure 2). Peak and mean densities were directly proportionate to internal bonding value.

The general correlations between internal bonding (IB) and peak density (PD) and mean density (MD) can be expressed as in equations 5 and 6 respectively.

$$IB = 0.006PD - 3.598, r^2 = 0.594 \quad (5)$$

$$IB = 0.007MD - 4.102, r^2 = 0.741 \quad (6)$$

Unlike other mechanical properties, internal bonding had higher tendency to be affected by mean density compared with peak density (r^2 = 0.741 and 0.594 respectively). Internal bonding

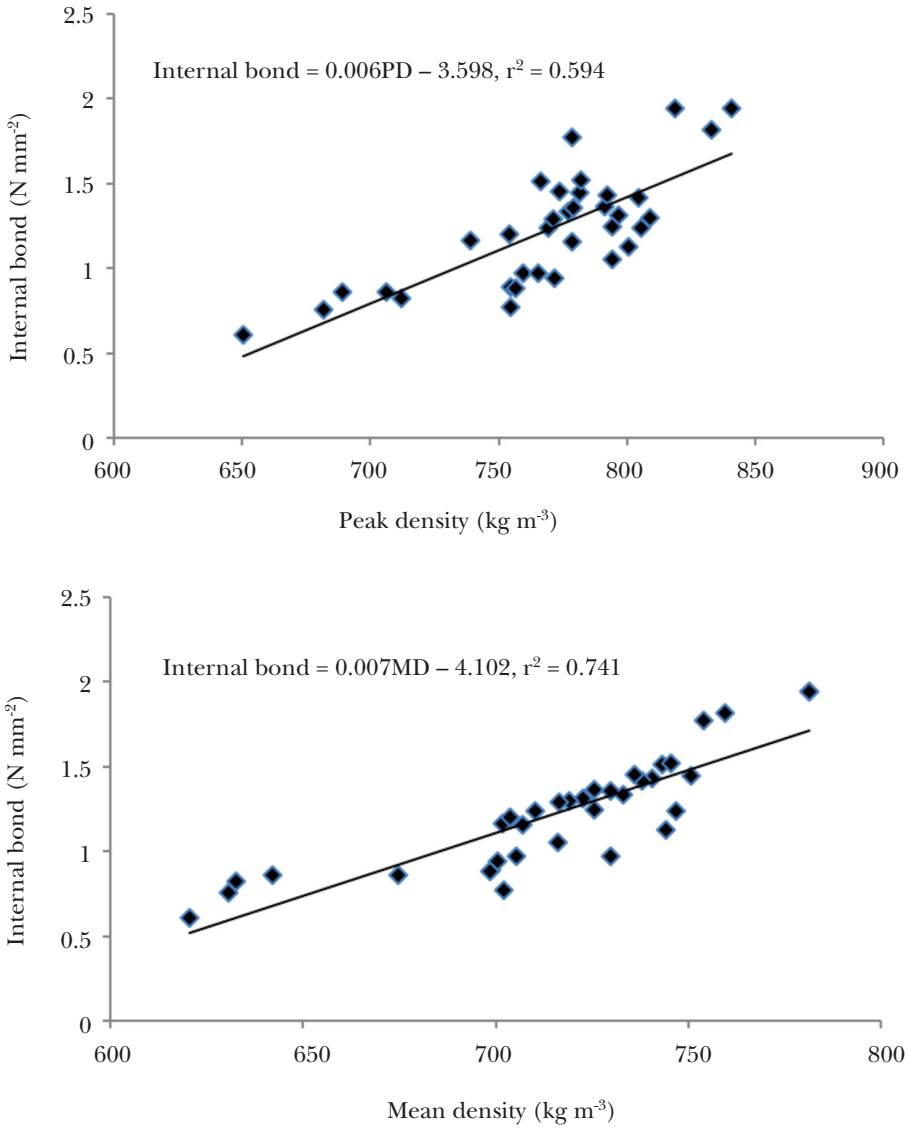

Figure 2 Correlation between internal bond and (a) peak density and (b) mean density

is highly dependent on core density of the board (Wong 1999). Therefore, most failures occur in the lower density core region during vertical tensile test. In fact, in homo-profile boards with uniform density profile, core density is equivalent to mean density which explained why internal bonding was more dependent on mean density compared with peak density (Wong 1999). Based on the internal bonding–mean density regression established in equation 6, an increment of 47% in internal bonding could be expected when mean density increased from 700 to 800 kg m^{-3}.

Weak linear relationships were observed between MOR and both peak and mean densities (Figure 3). The correlations between MOR and peak (PD) and mean (MD) densities can be expressed as in equations 7 and 8 respectively.

$$MOR = 0.027PD - 7.181,\ r^2 = 0.396, \quad (7)$$

$$MOR = 0.023MD - 2.291,\ r^2 = 0.239, \quad (8)$$

Although relationships were weak, based on the r^2 values, peak density exerted greater effect on MOR compared with mean density. Based on the equations above, an increase in peak density from 700 to 800 kg m^{-3} could produce 27% increment in MOR.

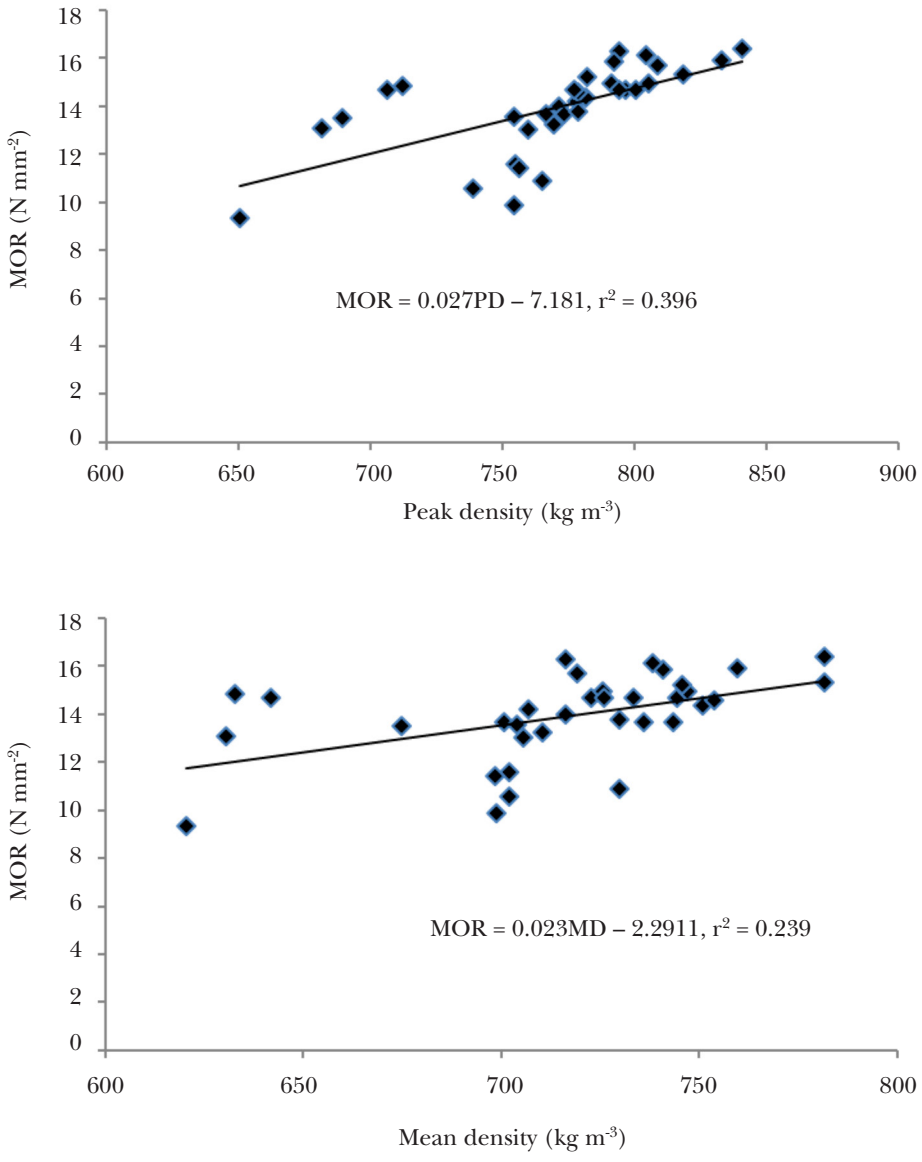

Figure 3 Correlations between the modulus of rupture (MOR) and (a) peak density and (b) mean density

Figure 4 exhibits the curvilinear relationship between MOE and peak (PD) and mean (MD) densities and the following equations were derived from the relationships:

$$MOE = 17081.57 - 43.88PD + 0.032PD^2, \quad r^2 = 0.642 \quad (9)$$

$$MOE = 14763.24 - 39.45MD + 0.031MD^2, \quad r^2 = 0.415 \quad (10)$$

Similar to MOR, peak density had more acute effect on MOE compared with mean density (Figure 5). These findings are in agreement with Wong et al. (2003) who reported that MOE increased proportionally with increasing peak density.

Highly positive quadratic term or convex curve was formed between Brinell hardness (BH) and peak (PD) and mean (MD) densities. Their relationships can be expressed as in equations 11 and 12 respectively.

$$BH = 26.261 - 0.073PD + 5.114E - 5PD^2, \quad r^2 = 0.845 \quad (11)$$

$$BH = 22.275 - 0.066MD + 4.992E - 5MD^2, \quad r^2 = 0.643 \quad (12)$$

Peak density had greater influence on the Brinell hardness of the particleboard samples compared with mean density. In comparison with internal bonding, peak density exerted greater effects on MOR, MOE

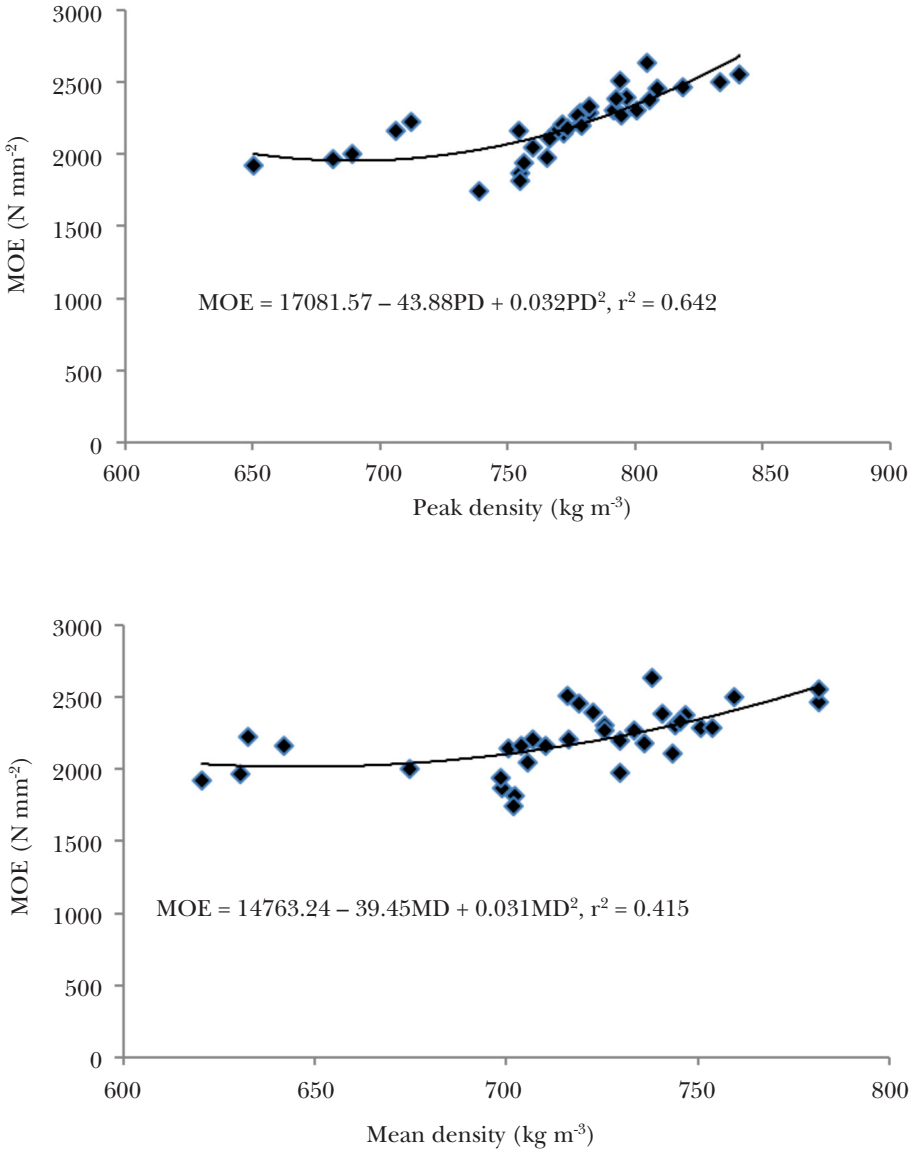

Figure 4 Correlation between modulus of elasticity (MOE) and (a) peak density and (b) mean density

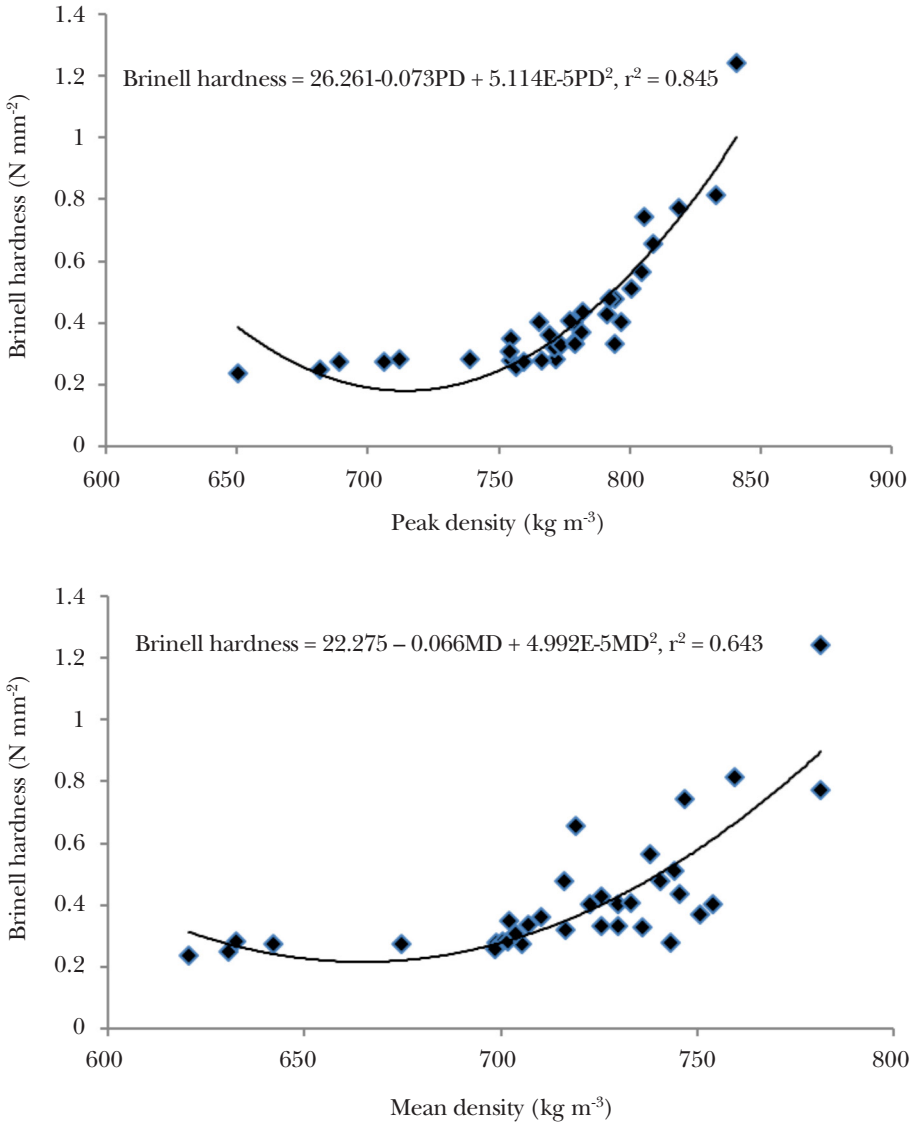

Figure 5 Correlations between the Brinell hardness and (a) peak density and (b) mean density

and Brinell hardness. During the flexural test, where the boards were bent, the concave and convex faces experienced stress at maximum compressive and tensile stress respectively. Core density exerted least or no influence on the compressive and tensile strength during the test. Thus, higher peak density, i.e. higher density in the surface layers correspondingly generated greater MOR, MOE and Brinell hardness.

CONCLUSIONS

Density profile was highly affected by treatment method. Treatment temperature significantly affected peak density and mean density but not peak distance. Mechanical properties of the particleboard samples decreased with increasing treatment temperature. However, samples treated using hot press had better mechanical properties after re-pressing which increased the density of the samples and counterbalanced the loss of strength. Internal bonding was highly affected by mean density while MOR, MOE and Brinell hardness were dependent on peak density. Heat treatment altered the density profile of the samples and these changes altered the mechanical properties of treated particleboard samples.

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