Characterization of Particleboard Made from Oil Heat-Treated Rubberwood Particles at Different Mixing Ratios

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Particleboard was produced by mixing oil heat-treated rubberwood particles at different ratios, with the goal of achieving high dimensional stability. Rubberwood particles were soaked in palm oil for 2 h and heat treated at 200 °C for 2 h. The treated particles were soaked in boiling water for 30 min to remove oil and were tested for chemical alteration and thermal characterization *via* Fourier-transform infrared spectroscopy and thermogravimetric analysis. Particleboard was fabricated by mixing treated rubberwood particles (30%, 50%, and 70%) with untreated particles (70%, 50%, and 30%, respective to previous percentages) and bonded with urea-formaldehyde (UF) resin. The results revealed that oil-heat treated particles had greater thermal stability than the untreated particles. The addition of oil heat treated particles improved the physical properties of the particleboard with no significant reduction in mechanical strength. However, this was only valid for ratios of 70% untreated to 30% treated and 50% untreated to 50% treated. When a ratio of 70% oil heat treated particles was used, both the physical and mechanical properties were reduced drastically, due to bonding interference caused by excessive oil content. Particleboard made with a ratio of 5:5 (treated to untreated) exhibited the best physical and mechanical properties.

Keywords: Oil heat treatment; Particleboard; Rubberwood; Palm oil; Green treatment

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INTRODUCTION

Urea formaldehyde (UF) is the most prevalent binding agent used for the manufacturing of particleboard. However, the aminomethylene linkage in UF resin is susceptible to hydrolysis, making it unstable in humid and hot conditions (Dunky 1998). Thermal treatment has long been recognized as an effective treatment method that improves the dimensional stability and biological durability of wood and wood-based products (Cui and Matsumura 2019). Research on post-heat treatments has been conducted in order to improve the dimensional stability of the particleboard (Lee *et al.* 2015). However, the study revealed that post-heat treatment adversely affected the strength of the particleboard. As UF resin is not heat resistant and will be degraded when subjected to high temperature, post-heat treatment is not suitable for enhancing the dimensional stability of UF-bonded particleboards (Zorba *et al.* 2008). Therefore, pre-heat treatment should be applied to the particles before being formed into particleboards. Vegetable oils have been widely used as the treating medium for wood in the heat treatment process due to its environmentally friendly and non-toxic nature (Dubey *et al.* 2016). The oils are also effective media for heat treatment. Application of vegetable oils during the heat treatment process could transfer the heat more evenly and readily to the wood (Cheng *et al.* 2014). In addition, it forms a protective layer on the wood, which inhibits water uptake (Wang and Cooper 2005).

Oil heat treatment on wood-based composites have been reported in various previous studies (Lee *et al.* 2017, 2018a,b, 2020). Lee *et al.* (2020) applied a two-step post heat treatment using palm oil on particleboards made from oil palm trunks and rubberwood, and the changes in colour and any improvements in fungal resistance were recorded. Lee *et al.* (2020) also revealed that the resistance of the oil heat treated particleboard against white rot fungi is improved. Another study by Lee *et al.* (2018c) also reported that oil palm trunk particleboards that underwent a two-step oil heat treatment using palm oil had enhanced termite resistance and dimensional stability. Nevertheless, the mechanical strength of the treated particleboard was severely degraded. As a result, the authors had to incorporate rubberwood (in ratios of 30% to 70%) into the oil palm trunk particleboard in order to enhance the strength of the particleboard. The reduction of the strength was primarily attributed to the thermal degradation of the UF resin, which was used as a binding agent for the particleboard in the study (Lee *et al.* 2017).

Most previous studies were focused on post-treatment, where the particleboard samples were thermally treated after the fabrication process. The drawback of post-treatment is that it caused severe degradation of the UF resin, which has low thermal resistance. Therefore, pre-treatment of the particles before the production of the particleboard would be a feasible option. A preliminary study on the performance of particleboards fabricated from palm oil-treated rubberwood particles was performed by Paiman *et al.* (2019). However, compared to the aforementioned studies, the temperature used by Paiman *et al.* (2019) was low (50 °C). The particles were immersed in boiling water for 30 min to remove the excess oil on the particle surfaces prior to particleboard fabrication. As a result, the total thickness swelling and water absorption of the particleboard made from oil treated particles was reduced, which indicated better dimensional stability. However, the bending strength of the particleboard was adversely affected, particularly the internal bonding strength, which experienced a 92.5% reduction in comparison to the untreated particleboard. Lower wettability or hydrophobic characteristics of the particles as a result of oil treatment was one of the reasons that inhibited effective spreading of the UF resin and subsequently led to inferior mechanical strength (Shi and Gardner 2001). Therefore, in this study, the feasibility of mixing oil heat-treated rubberwood particles with untreated rubberwood particles at different mixing ratios was investigated. The purpose of the study was to produce particleboards with high dimensional stability and minor or no adverse effects on the mechanical strength.

EXPERIMENTAL

Materials

Rubberwood (*Hevea brasiliensis*) particles were obtained from HeveaBoard Berhad, a particleboard manufacturing plant located in Gemas, Johor, Malaysia. The obtained particles were stored in a conditioning room and were dried to 3% moisture content prior to the oil heat treatment. Type E1 urea formaldehyde (UF) resin, with a solids content of 65%, was purchased from Aica Malaysia Sdn. Bhd., a glue production plant located in Senawang, Selangor, Malaysia. Edible palm oil with the brand name Vesawit was purchased from a local grocery store and was used as the treating medium for the rubberwood particles. Ammonium chloride was used as a hardener in this study.

Oil Heat Treatment of the Rubberwood Particles

Rubberwood particles were dried at 103 °C ± 2 °C in a laboratory oven for 24 h. The moisture content of the rubberwood particles was recorded. After being oven dried, the particles were soaked in palm oil for 2 h. After soaking, the particles were treated in a laboratory oven at 200 °C for 2 h. Next, the heat-treated particles were taken out of the oven and immersed in a pail filled with boiling water for 30 min. The purpose of this step was to reduce the amount of oil on the particles. After immersion, the particles were dried in a laboratory oven set at 103 °C ± 2 °C for 24 h. The dried particles were then conditioned in a conditioning room, which had a temperature of 20 °C ± 3 °C and a relative humidity of 65% ± 5% for 1 week prior to particleboard production.

Characterization of the Oil Heat Treated Particles

Fourier-transform infrared spectroscopy (FTIR) analysis

Both the treated and untreated rubberwood particles were ground into a powder. Then the powders were scanned using a Perkin Elmer FTIR instrument (with a 1 cm^{-1} resolution, 16 scans, KBr method) at the Laboratory of Biocomposite, located at the Institute of Tropical Forestry and Forest Products (INTROP), Universiti Putra Malaysia. The scanning region was between 500 cm^{-1} and 4000 cm^{-1}.

Thermogravimetric (TGA) analysis

Approximately 8 mg of powdered treated and untreated rubberwood particles were subjected to TGA analysis under a nitrogen atmosphere using a TGA Q500 (V20.13 Build 39), at a temperature between 30 °C and 600 °C (at a heating rate of 10 °C min^{-1}). The TGA device was located at the Laboratory of Biocomposite, Institute of Tropical Forestry and Forest Products (INTROP), Universiti Putra Malaysia.

Production of the particleboards

Single layer particleboards were fabricated from the treated and untreated particles, respectively. Four different ratios (on weight basis) of untreated particles to treated particles were used: 100% untreated, 70% untreated to 30% treated, 50% untreated to 50% treated, and 30% untreated to 70% treated. These were denoted as the control, 7:3, 5:5, and 3:7, respectively. Particleboards with dimensions of 340 mm x 340 mm x 12 mm (length by width by thickness) and a target density of 800 kg/m^3 were produced. A 10% UF resin, based on the oven-dried weight of rubberwood particles, was used (170 g at 65% solid content). The UF has a gelation time of 65 s at 100°C, viscosity of 220 cP, and pH of 5.5. A 1% hardener (ammonium chloride (NH_4Cl)), based on the solid weight of the UF resin (4.4 g at 25% solid content), and a 0.5% wax (9.3 g at 60% solid content), based on the oven dried weight of the rubberwood particles, was added to the UF resin.

The resin admixture was sprayed onto the rubberwood particles and blended evenly in a blending machine for 5 min. After the blending process, the resinated wood particles were poured into a wooden mould to form a mat and a pre-press was applied to compact the mat. The mat was then hot-pressed at 180 °C for 270 sec with a pressure of 100 bar. After pressing, the board was conditioned for a week in a conditioning room at a temperature of 20 °C ± 3 °C and relative humidity of 65% ± 5%. After the constant mass was reached, the boards were trimmed and cut into testing dimensions (as denoted above) for both the mechanical and physical properties evaluation. For each ratio, 2 boards were produced. A total of 8 boards were manufactured in this study.

Evaluation of the Physical and Mechanical Properties of the Particleboard

Physical properties

The testing procedure for determining the water absorption (WA) and thickness swelling (TS) were conducted in accordance with Japanese industrial standard (JIS A 5908 2003). Particleboard samples with a width and length of 50 mm x 50 mm were immersed in water, and the thickness and weight of the samples were weighed after immersion for 2 h and 24 h. The changes in thickness and weight before and after immersion were recorded and were used to compute the WA and TS values. The equilibrium moisture content (EMC) and moisture excluding efficiency (MEE) of the samples were determined according to Lee *et al.* (2017). The equations for EMC and MEE are shown in Eq. 1,

$$\text{Equilibrium moisture content (\%)} = 100\,(W_2 - W_1)/W_1 \qquad (1)$$

where W_1 is the oven dried weight (g) and W_2 is the constant weight after reconditioning (g), and in Eq. 2,

$$\text{Moisture excluding efficiency (\%)} = 100\,(\text{EMC}_\text{u} - \text{EMC}_\text{t})/\text{EMC}_\text{u} \qquad (2)$$

where EMCu is the EMC of the untreated samples (%) and EMCt is the EMC of the treated samples.

The anti-swelling efficiency (ASE) and water repellence efficiency (WRE) of the samples after immersion in water for 24 h were determined according to Paiman *et al.* (2019). The formula is shown in Eq. 3,

$$\text{Anti-swelling efficiency (\%)} = 100\,(S_\text{u} - S_\text{t})/S_\text{t} \qquad (3)$$

where S_u is the volumetric swelling of the untreated samples (%) and S_t is the volumetric swelling of the treated samples (%), and in Eq. 4,

$$\text{Water repellence efficiency (\%)} = 100\,(W_\text{u} - W_\text{t})/W_\text{t} \qquad (4)$$

where W_u is the water uptake by the untreated specimens (%) and W_t is the water uptake by the treated samples (%). Five samples were used for each type of testing procedure.

Mechanical properties

The mechanical properties, which included the internal bonding strength (IB) and static bending (modulus of rupture (MOR) and modulus of elasticity (MOE)) were evaluated according to the procedure stipulated in standard (JIS A 5908 2003). Five samples were tested for each testing procedure.

Statistical Analysis

The collected data were analyzed using the Statistical Analysis System (SAS) procedure for the analysis of variance (ANOVA) at a 95% confident level (*p*-value was less than or equal to 0.05). Tukey’s Honest Significant Difference (HSD) test was used to further determine the significance level between each mixing ratio.

RESULTS AND DISCUSSION

Fourier-transform Infrared Spectroscopy (FTIR) Analysis

Figure 1 illustrates the changes in the chemical properties of the control and the oil heat treated rubberwood particles at 200 °C. New peaks were observed at 2919, 2850, and 1740 cm^{-1} for the oil heat treated rubberwood particles. The peaks corresponded to the =C-H stretching, CH_2 asymmetric and symmetric bending, and the -C=C stretching of ester carbonyl groups of triglycerides, respectively; these peaks are typical characteristics of palm oil. The peak near 1740 cm^{-1} corresponds to the carbonyl group (Tjeerdsma and Militz 2005). After exposure to high temperatures, this peak should disappear or decrease in intensity, due to the cleavage of the acetyl groups, *i.e.*, deacetylation. However, as shown in Fig. 1, the intensity of the peak increased. The increased intensity could be attributed to ester groups from the palm oil retained in the rubberwood particles (Pena *et al.* 2014). Another band that corresponded to the palm oil characteristics was a sharp peak in the 1454 cm^{-1} region, which corresponded to the CH_2 bending, -C-O stretching vibration, and -CH_2 bending (cis -CH=CH-bending) of palm oil (Sadrolhosseini *et al.* 2017). This peak might also correspond to the -C=C- of furfural, which results from the degradation of polysaccharides (Tjeerdsma and Militz 2005). The peak near the 1154 cm^{-1} region corresponded to the fatty acids and glycerols (C-O ester groups) generated from the hydrolysed triglycerides of the palm oil at elevated treatment temperatures (Sim *et al.* 2014).

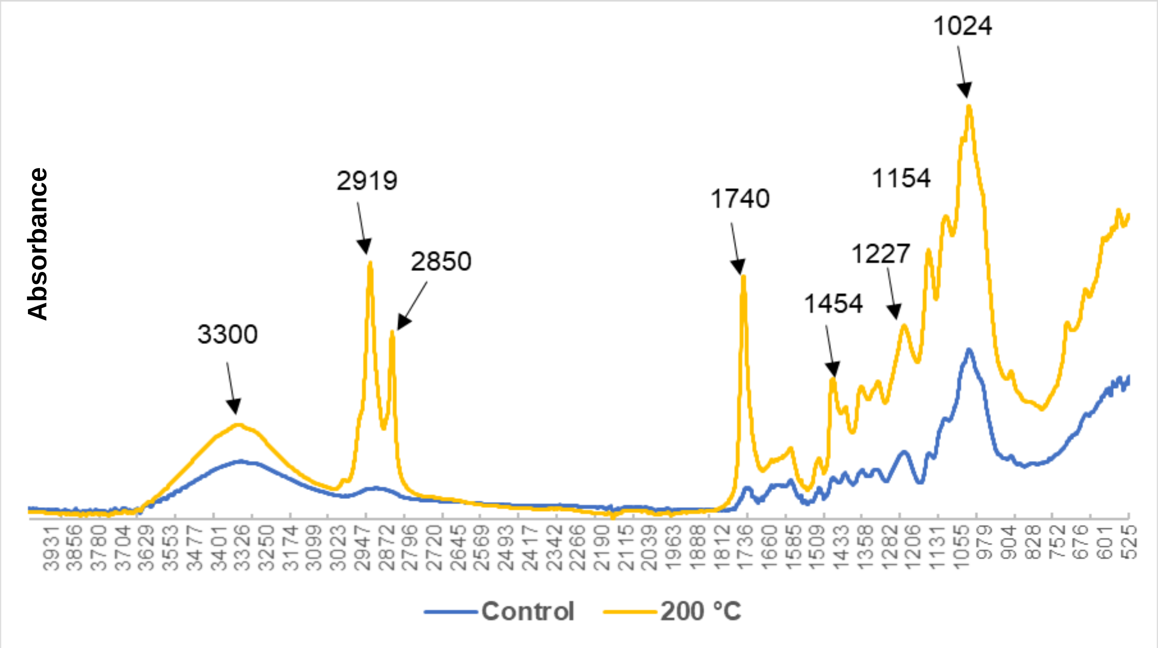

Fig 1. FTIR spectra of control rubberwood particles and oil heat treated particles at 200 °C

Bands at the 3300 cm^{-1} regions could be attributed to the O-H stretching vibration from alcohols/phenols (Esteves *et al.* 2013). Higher peak intensity for oil heat-treated particles compared to untreated particles was observed at this region. It is known that thermal treatment reduces the accessibility of the OH groups *via* carbohydrate dehydration and therefore the intensity in this region should decrease. However, the O-H groups from the phenolic groups in lignins increased at the same time the carbohydrates degraded and subsequently led to such observations (Esteves *et al.* 2011; Kubovský *et al.* 2020). Bands observed near the 1227 cm^{-1} region could be attributed to the syringyl ring breathing and C-O stretching in the lignins (Colom *et al.* 2003). The peak near 1024 cm^{-1} corresponded to the C–O stretching in celluloses and lignins (Traoré *et al.* 2016). These peaks increased as the lignin percentage increased, due to the degradation of polysaccharides. Chemical constituents of treated and untreated rubberwood particles were determined using TAPPI standards. The cellulose and hemicellulose content of rubberwood particles were reduced from 43.1% and 27.9% to 38.2% and 21.7%, respectively, after oil heat-treated at 200°C. Contrarily, lignin content increased from 23.1% to 34.2% after oil heat treatment.

Thermogravimetric Analysis (TGA)

The thermogravimetric (TG) curves and derivative thermogravimetric (DTG) curves of the oil heat treated rubberwood particles (at 200 °C) are shown in Figs. 2 and 3. As shown in Fig. 3, two mass loss peaks were observed, namely the dehydration stage at a temperature range of 20 to 120 °C and the polysaccharides decomposition stage, which started at greater than 200 °C (Xu *et al.* 2019). The maximum mass loss of the untreated rubberwood particles was reached at 351 °C, while the maximum mass loss for the oil treated particles was reached at 360°C.

At temperatures below 100 °C, the untreated rubberwood particles lost 10.2% of their mass, which could be primarily attributed to the loss of water and some volatile organic compounds (Korošec *et al.* 2017). However, at the same temperature (below 100 °C), the oil heat treated particles only lost 2.35% of their mass, which indicated that the hygroscopicity of the treated particles had been greatly reduced.

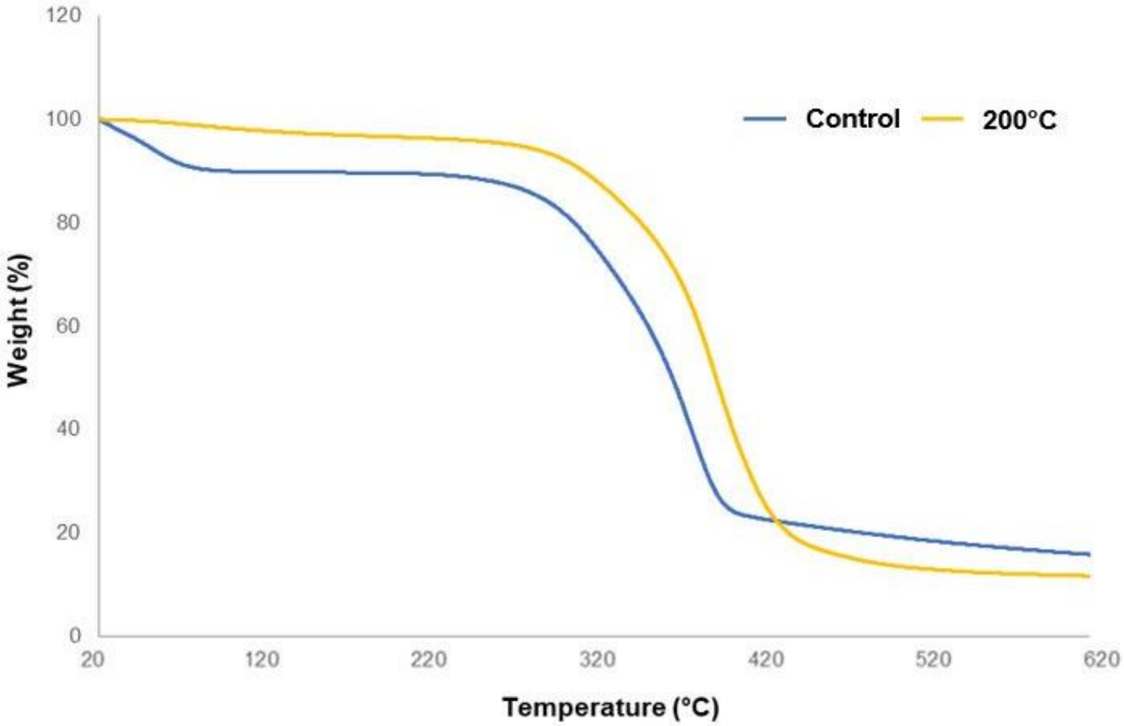

Fig. 2. Thermogravimetric (TG) curves of the control rubberwood particles and oil heat treated particles (at 200°C)

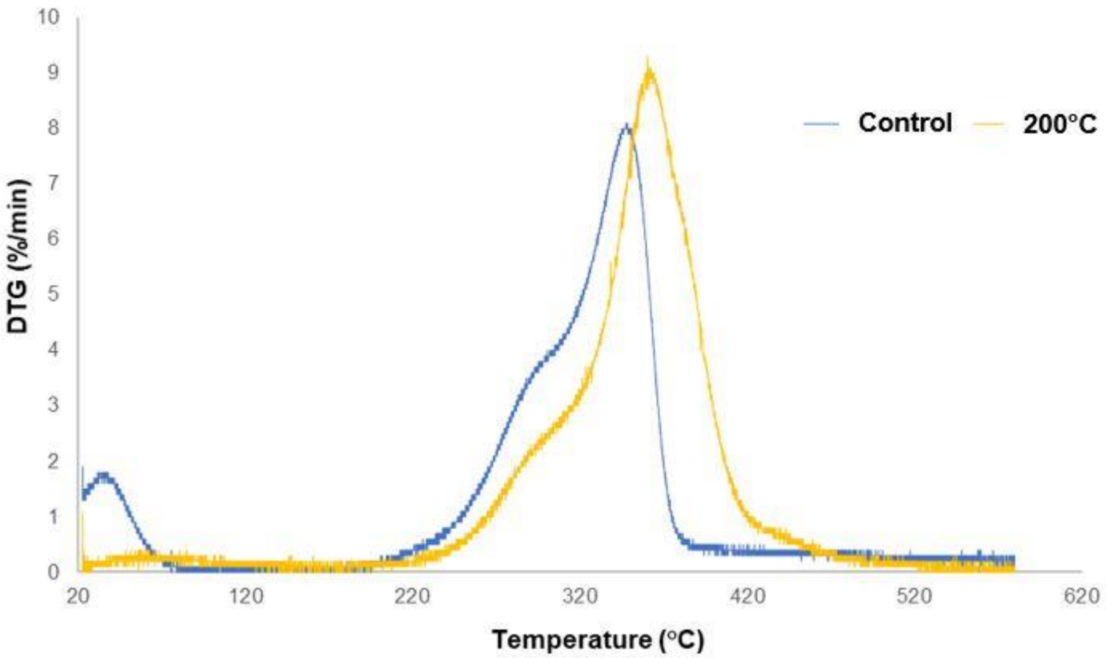

Fig. 3. Derivative thermogravimetric (DTG) curves of the control rubberwood particles and oil heat treated particles (at 200 °C)

The highest mass loss for both treated and untreated particles were recorded within a temperature range of 250 °C to 400 °C, where more than half of their mass was lost. The mass loss of the samples was primarily caused by the decomposition of the chemical constituent of the wood itself at high temperatures. Sinoven *et al.* (2002) stated that hemicelluloses and amorphous cellulose start to decompose at 180 °C. However, lignins start to degrade at a slower rate (Alen *et al.* 1995). As the temperature continue to rise to approximately 300 °C, the crystalline celluloses started to decompose (Kim *et al.* 2001). At temperatures higher than 380 °C, the decomposition rate started to slow down and leave behind residues, which contained primarily carbon and partially decomposed lignin (Korošec *et al.* 2017).

Oil heat-treated rubberwood particles exhibited greater thermal stability compared to the untreated rubberwood particles. For untreated rubberwood particles, the highest mass loss (33.9%) was observed within a temperature range of 300 to 350 °C. However, the oil heat treated rubberwood particles displayed its highest mass loss (40.5%) at a higher temperature range, *i.e.*, 350 °C to 400 °C. This phenomenon could be explained by the fact that the oil heat treated particles contained a higher lignin content and a lower holocellulose content than the untreated particles. Promising features of the thermally modified wood include that it has a lower amount of hydroxyl groups but a higher cellulose crystallinity and greater number of cross-linking reactions in its lignins (Esteves and Pereire 2009). Therefore, a higher mass loss was observed in the oil heat treated samples at 350 to 400 °C as lignin generation was reported to typically take place at temperatures greater than 350 °C (Gronli *et al.* 2002).

Physical Properties of Particleboard

The equilibrium moisture content (EMC) of the particleboards made from different mixing ratios of untreated and oil heat-treated particles are shown in Fig. 4. The EMC of the control particleboard was 4.02%. As shown in Fig. 4, the EMC of the particleboards decreased after being admixed with oil heat treated particles. The EMC of the treated samples were reduced to 3.35% (7:3), 2.47 % (5:5), and 2.93 (3:7). The reduction in EMC is a prominent characteristic of heat treatments due to the diminished hydrophilic hydroxyl groups and increased cellulose crystallinity (Boonstra and Tjeerdsma 2006). Reduction of hemicellulose after treatment also contributed to this phenomenon.

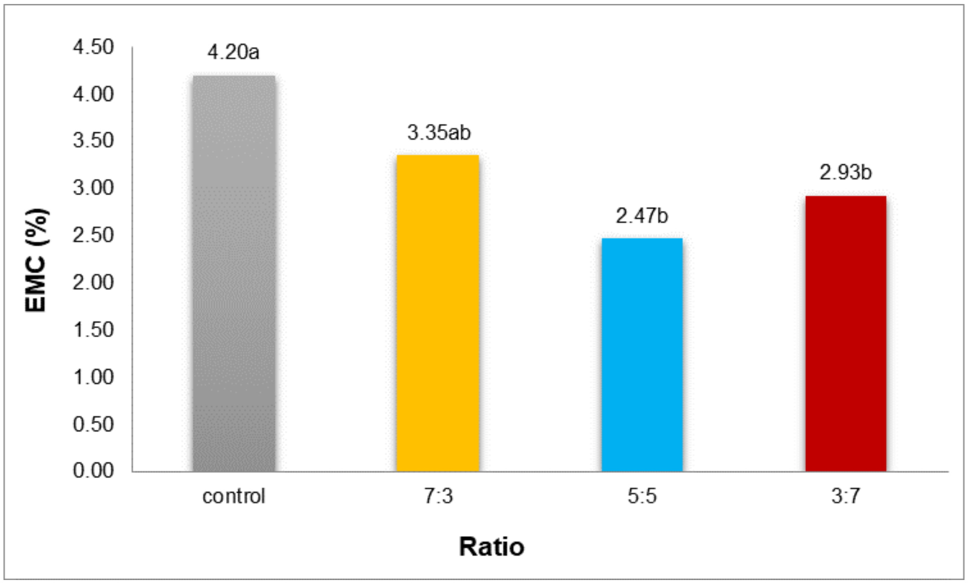

Fig. 4. Equilibrium moisture content (EMC) of the particleboards produced from treated particles at different mixing ratios

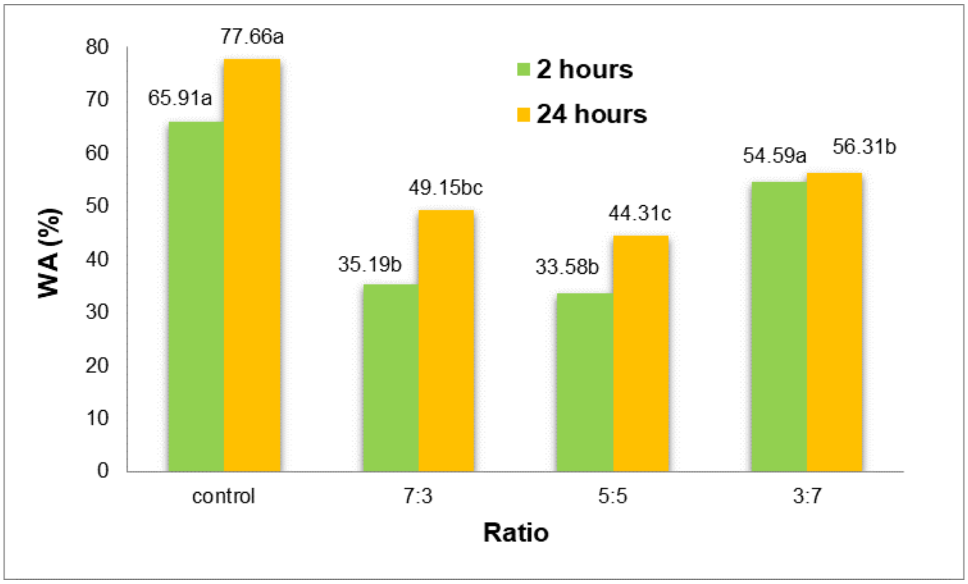

Fig. 5. Water absorption (WA) of the particleboards produced from treated particles at different mixing ratios

Figure 5 illustrates the average values of the WA_{2h} and WA_{24h} of the control and particleboards made from different mixing ratios. The control particleboard values recorded for the WA_{2h} and WA_{24h} were 65.9% and 77.7%, respectively. After mixing with the oil heat treated rubberwood particles, the WA value of the particleboard was significantly ($p \leq 0.05$) reduced. When the mixture included 30% oil heat treated particles (7:3), the WA_{2h} and WA_{24h} values were significantly ($p \leq 0.05$) reduced (35.2% and 49.2%, respectively). A higher reduction was observed when the mixture included 50% oil heat treated particles (5:5). However, when the amount of oil heat treated particles was increased to 70% (3:7), the WA values started to increase.

A similar trend was observed for the TS, as shown in Fig. 6. For the control particleboard, the TS_{2h} and TS_{24h} values were 30.4% and 38.8%, respectively. Similar to WA, the lowest TS value was recorded in the particleboard fabricated from a mixing ratio of 5:5. When the mixture included 70% oil heat treated particles, the TS value of the particleboard increased to an extent that had no statistical difference ($p \leq 0.05$) from the control particleboard.

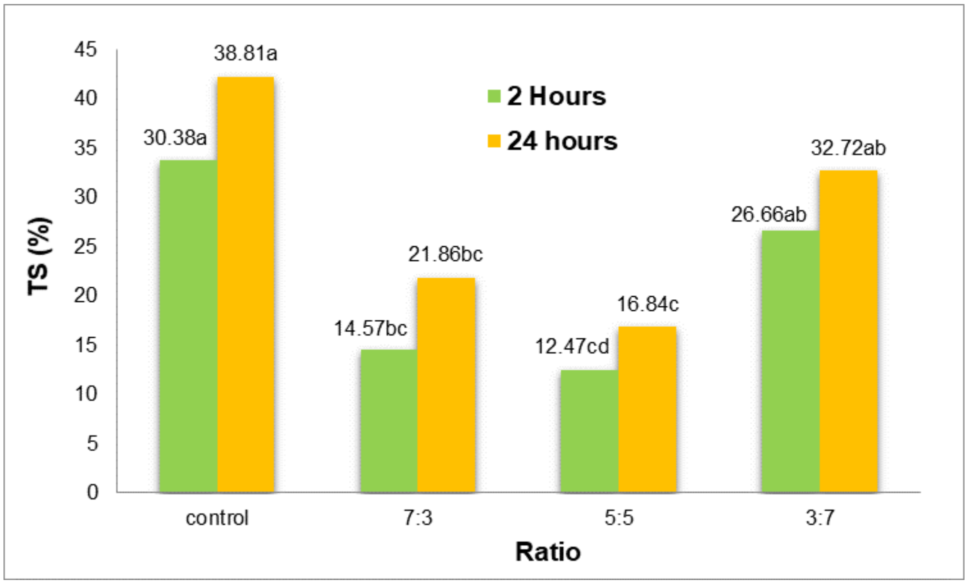

Fig. 6. Thickness swelling (TS) of the particleboards produced from treated particles at different mixing ratios

Table 1. Anti-swelling Efficiency (ASE), Water Repellent Efficiency (WRE), and Moisture Excluding Efficiency (MEE) of the Particleboards Made From Different Ratios of Treated and Untreated Particles

Ratio (U:T)	ASE (%)	WRE (%)	MEE (%)
7:3	22.59 ± 2.63^{a}	60.41 ± 1.31^{b}	37.86 ± 1.34^{b}
5:5	26.79 ± 3.32^{a}	76.14 ± 3.44^{a}	44.14 ± 3.94^{a}
3:7	15.32 ± 3.23^{b}	35.78 ± 2.61^{c}	24.56 ± 1.64^{c}

Note: within the same column, mean values followed by the same letter a, b, and c are not statistically different at $p \leq 0.05$.

The anti-swelling efficiency (ASE), water repellent efficiency (WRE), and moisture excluding efficiency (MEE) of the particleboards made from different ratios of treated and untreated particles are listed in Table 1. The positive ASE and WRE values indicated the effectiveness of the treatment. Positive ASE values of 15.3% to 26.8% were recorded in this study, which indicated that the dimensional stability of the particleboard had been improved. It should be noted that the particleboards made from 50% untreated and 50% oil heat treated particles (5:5) had the highest ASE value (26.8%) and WRE value (76.1%). The particleboards made from 30% untreated and 70% treated particles (3:7) had the lowest ASE and WRE values, 15.3% and 35.8%, respectively. Moisture excluding efficiency values of 24.6% to 44.1% were recorded, which suggested that the hygroscopicity of the particleboard had been reduced due to hemicellulose reduction.

As mentioned above, the reduction in EMC due to the heat-induced reaction on the wood constituents enhanced the dimensional stability of the particleboard. In addition to that, the intake of oil into the wood cell walls as well as the formation of a barrier on the rubberwood particles improved the dimensional stability of the particleboard to a greater extent due to a synergetic effect between the heat and oil (Hyvonen *et al.* 2007). Overall, a ratio of 50% treated to 50% untreated particleboard bestowed the best physical properties to the particleboard. When a mixture of 70% oil heat treated particles were used, excessive oil might have prevented the particleboard from obtaining sufficient bonding. Consequently, the holding ability between the particles became weaker and easily broke down when immersed in water, which led to higher rates of swelling of thickness and water absorption.

Mechanical Properties of Particleboard

The mean value for the modulus of rupture (MOR), modulus of elasticity (MOE), and internal bonding (IB) strength of the particleboards made from different mixing ratios are depicted in Figs. 7, 8, and 9.

The MOR of the particleboards made from 100% untreated particleboard (control) had slightly higher values compared to the other ratios, as shown in Fig. 8. The particleboard made from made from a mixture of 70% untreated to 30% treated (7:3) and 50% untreated to 50% treated (5:5) particles had a slightly higher MOE in comparison to the control particleboard, as shown in Fig. 9.

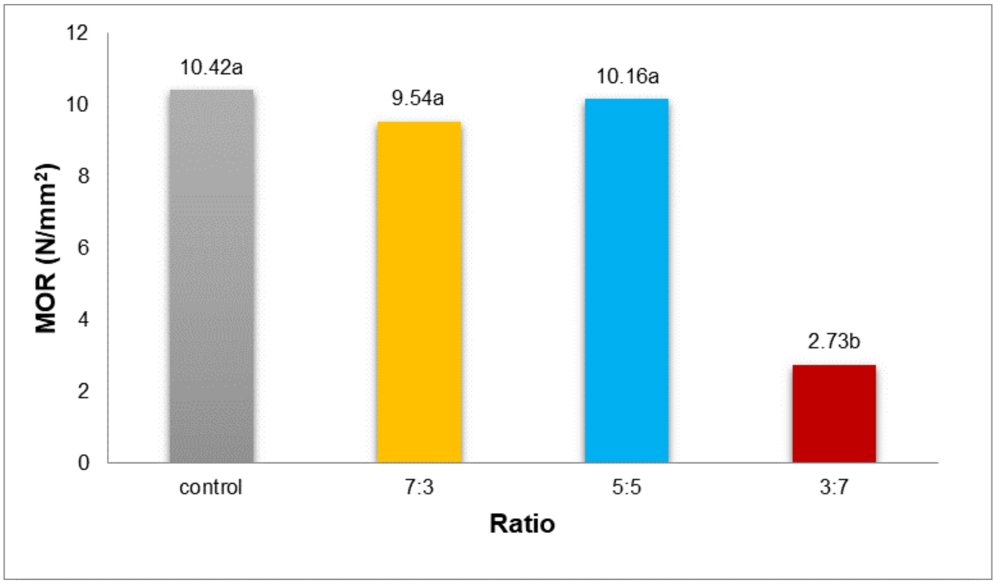

Fig. 7. Modulus of rupture (MOR) of the particleboards produced from treated particles at different mixing ratios

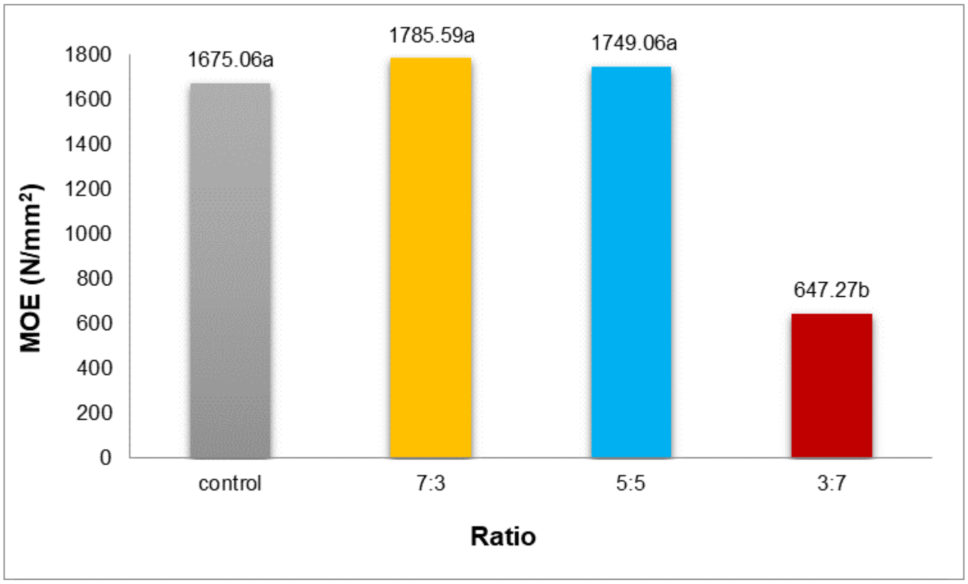

Fig. 8. Modulus of elasticity (MOE) of the particleboards produced from treated particles at different mixing ratios

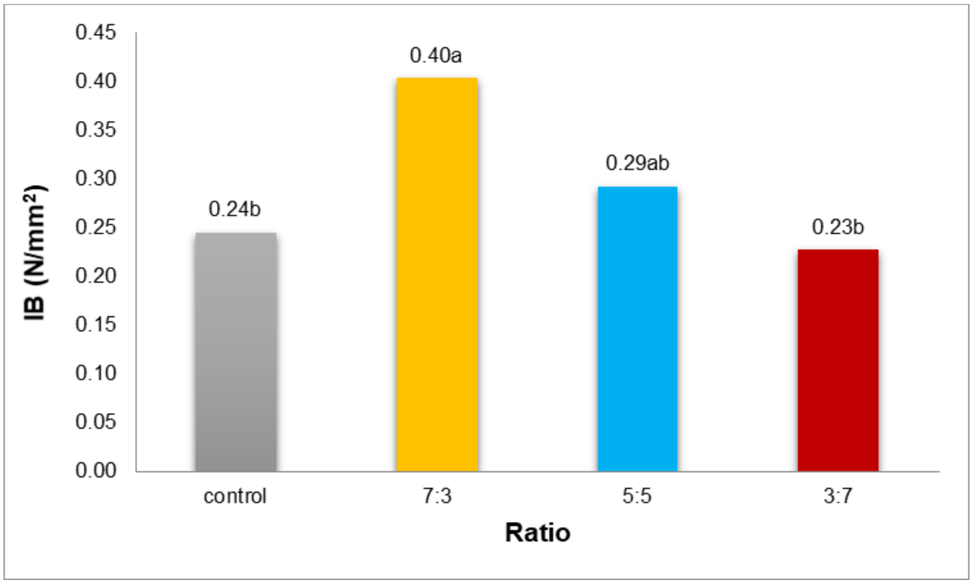

Fig. 9. Internal bonding (IB) of the particleboards produced from treated particles at different mixing ratios

However, no significant difference ($p \leq 0.05$) in terms of the MOR and MOE were detected between the control particleboards and the two aforementioned ratios. Nevertheless, when a 70% oil heat treated particle mixture (3:7) was used in particleboard production, both the MOR and MOE values were severely reduced in comparison to the control particleboard. A reduction of 74% in the MOR and 62% in the MOE was detected. As for IB, the particleboards made from a 70% untreated to 30% treated (7:3) and 50% untreated to 50% treated (5:5) particle ratio exhibited a higher value than the control particleboards, as shown in Fig. 10. Similarly, the IB value for the particleboards made from 70% oil heat treated particles had the lowest values.

Oil heat-treated rubberwood particles have lower wettability due to the formation of an oily hydrophobic layer and resulted in poor resin spreading (Shi and Gardner 2001). Consequently, good particle-particle bonding was inhibited and led to lower mechanical properties. However, when a lower ratio of oil heat treated particles, *e.g.*, 30% and 50%, were added, the presence of oil might aid the UF resin in spreading more rapidly and evenly during pressing. As a result, the bonding between the particles improved. Nevertheless, when the ratio of oil heat treated particles was increased to 70%, the excessive oil might interfere with the curing system of the UF resin and negatively affect the mechanical properties of the particleboard.

CONCLUSIONS

1. FTIR analysis showed peaks that corresponded to the presence of the palm oil used in this study, as well as peaks that corresponded to the alteration of the chemical composition of the rubberwood particles after oil heat treatment.
2. Oil heat-treated rubberwood particles also had a higher thermal stability when compared to the untreated rubberwood particles, as shown by the TGA analysis.
3. Mixing oil heat-treated particles significantly ($p \leq 0.05$) improved the physical properties of the resultant particleboards and did not greatly affect the mechanical properties. This observation was also prominent even when using a lower ratio of oil heat treated particles (30%).
4. Generally, particleboards made from mixture of 50% untreated and 50% oil heat treated rubberwood particles (5:5) possessed the highest modulus of elasticity (MOE) and internal bond (IB) values in comparison to the other mixing ratios and the control. The modulus of rupture (MOR) values of the oil heat treated particleboards were less than control particleboard, but no statistical different ($p \leq 0.05$) was observed.
5. The particleboards made from 50% untreated and 50% oil heat treated rubberwood particles (5:5) had the lowest thickness swelling (TS) and water absorption (WA) values, which indicated the greatest dimensional stability. However, when the amount of oil heat treated particles was increased to 70%, all of the properties of the particleboard were adversely affected. Therefore, the ratio of 5:5 was judged to be the best mixing ratio for producing particleboard with high dimensional stability and good mechanical properties.

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