MECHANICAL BEHAVIOR OF HYDROTHERMALLY TREATED OIL PALM WOOD IN DIFFERENT BUFFERED PH MEDIA

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(Received November 2015)

Abstract. This study was carried out to determine mechanical properties of oil palm (*Elaeis guineensis*) wood (OPW) in different buffered pH media; alkaline (pH 8), acidic (pH 5), and water at a temperature of 140°C for 120 min. The OPW samples were taken from outer part of trees. The modulus of rupture (MOR), modulus of elasticity (MOE), the compression strength parallel to grain (PC_{II}), and hardness strength (HB) were measured on treated and untreated samples. The different buffer media significantly affected the mechanical properties of OPW. The results showed a significant decrease of MOE, MOR,

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Wood and Fiber Science, 48(3), 2016, pp. 193-201

PC_{II}, and HB of treated specimens compared with untreated specimens. The PC_{II} and HB properties were significantly different between treated samples in water and pH 5 with the treated samples in pH 8 and also untreated samples. It was concluded that the hydrothermal treatment in the buffered medium significantly decreased the mechanical properties of treated specimens of OPW that could be related to the degradation of hemicelluloses. This will highly affect bending and tensile strengths of the OPW.

Keywords: Oil palm wood, mechanical properties, buffered media, thermal modification.

INTRODUCTION

Oil palm (*Elaeis guineensis* Jacq.) is a monocotyledonous with specific structural differences from commercial timbers. It is widely cultivated (about 15 million ha) in a few countries and is also grown primarily for its oil (Lim and Gan 2005). The oil palm has been cultivated in Malaysia for a century and now is the most important economic crop in Malaysia. The oil palm stems (OPSs) are highly available during replanting process after a growing period of 25-30 yr. Approximately 15 million tons of lignocellulosic materials are annually generated in Malaysia (Hartono 2012).

One of the drawbacks for using oil palm wood (OPW) is its naturally poor mechanical strength and the difficulties in drying with no defects. Unfortunately, the scientific information and knowledge "know-how" of oil palm trunk (OPT) is still limited. However, there is no economic value of OPS from structural point of view (Abdul Khalil 2006), and the maximum utilization of OPS is still inhibited with several factors such as variation in density, high MC (Shirley 2002; Choo et al 2010; Loh et al 2011), high extractives content (2-10%), and low dimensional stability (Ibrahim 1989; Abdul Khalil et al 2008; Bakar et al 2008; Bakar et al 2013).

Various techniques have been implemented to enhance the properties of OPS such as polymer injection, thermal treatment, chemical modification, and resin impregnation. The most advantage of buffered solution is its feature as a nonchemical modification that will not pollute the environment (Xie et al 2007; Fukuta et al 2008; Bakar et al 2013).

The hydrothermal treatment method makes some alterations in wood such as removal of extractives, hemicellulose hydrolysis, and change of the lignin and cellulose properties (Garrote et al 1999; Sandberg and Navi 2007). Therefore, this method is another alternative way for treating OPT with usage of preservatives. This method is designed to improve the structural properties of the OPW (Ruyter et al 1995; Dungani et al 2013).

The objective of this study was to investigate the effect of hydrothermal treatment with different buffered media on the OPW strength properties to reduce the degradation effect of acids released from the wood after treatment. This study was a preliminary study to understand the wood structure alteration after a treatment in buffered media.

MATERIALS AND METHODS

Buffer Preparation

Acidic buffer (pH 5), alkaline buffer (pH 8), and tap water (pH 6.70) were selected as media for the hydrothermal treatment. The buffer mixtures were prepared according to Table 1.

Sample Preparation

Matured oil palm trees were randomly harvested from Universiti Putra Malaysia (UPM) oil palm plantation. Three logs with 2 m long were consecutively cut above the tree breast height. This study was based on the outer parts of the trunks due to having similar density (Bakar et al 2008). The logs were then processed and flat sawn into

Table 1. Preparation of the buffered solutions.

pH	Chemical composition
5	257.5 mL $Na_2HPO_4.2H_2O$ (0.2 M) + 242.5 mL $C_6H_8O7.H_2O$ (0.1 M)
8	486.25 mL $Na_2HPO_4.2H_2O$ (0.2 M) + 13.75 mL $C_6H_8O_7.H_2O$ (0.1 M)

Source: Alexeyev (1967)

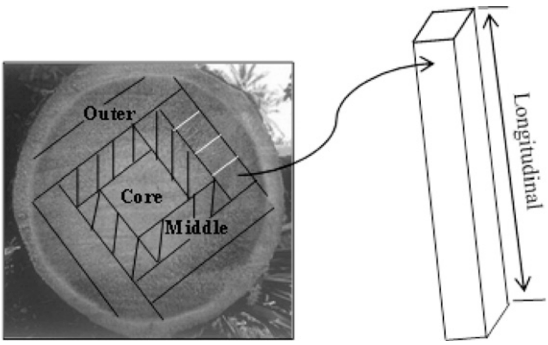

Figure 1. Flat sawn cutting for board preparation.

boards with dimensions of 60 × 5 × 5 cm (length × width × thickness) (Fig 1). To avoid fungus attack and moisture loss, the boards were kept in a cold room at 4°C for further analyses. Afterward, specimens with dimensions of 300 × 20 × 20 mm, 60 × 20 × 20 mm, and 150 × 50 × 50 mm were cut to evaluate mechanical properties such as static bending tests, compression parallel to grain (PC_{II}), and hardness strength (HB).

Heat Treatment

Hydrothermal treatment was carried out using buffered media, ie, pH 5 and 8 and water medium. Tap water with pH 6.7 was used as one of the treatment media. For measuring each mechanical property, 10 green samples (MC approximately 114%) were placed in a laboratory digester (JSR Instruments, Digester 30.4, Uttarakhand, India) and were treated with the aqueous solution of the buffered and unbuffered (control) media. The temperature was gradually increased to 140°C during 35 min and was kept for 120 min. The main role of the buffered media is to keep the pH constant in a certain level (Talaei 2010; Ebadi et al 2015). Untreated samples were used as controls for comparison purposes. After heat treatment, cylinders were gradually cooled down to avoid structural tension and specimens were kept in a control chamber at temperature of 20 ± 2°C and RH of 65% ± 3% to equilibrate them to about 12% ± 2% MC. All of the tests were performed according to British-adopted European standard BS 373 (1957) for static bending tests, PC_{II}, and universal toughness accordance to ASTM (D143-09, 2004) for HB.

The static bending test specimens were made on 20 × 20 × 300 mm. The width and thickness of each specimen were measured at mid length. Center point loading with a span length of 300 mm was used. The specimen was then placed on two supports over a span of 280 mm. The cross-head was continuously applied at mid span of the samples at a constant speed of 6.60 mm/min. The span was established to maintain a minimum span-to-depth ratio of 14. These properties were calculated according to the following equations:

$$\mathrm{MOE} = \frac{\mathrm{PL}^3}{4Dbt^3} \tag{1}$$

$$\mathrm{MOR} = \frac{3P_mL}{2bt^2} \tag{2}$$

where MOE: modulus of elasticity (MPa or N/mm^2); MOR: modulus of rupture (MPa or N/mm^2); P: loading at proportional limit (N); P_m: maximum load and force (N); b: width of specimen (mm); L: span length between specimen support of the span (mm); t: thickness or depth of specimens (mm); and D: increment of deflection at mid length corresponding to P or deflection of the neutral plane at the proportional limit measured at half span (mm).

The PC_{II} test was carried out by samples with dimension of 20 × 20 × 60 mm. The load was applied through a metal bearing plate 50 mm in width placed across the upper surface of the specimen at equal distances from the ends and right angles to the length. The PC_{II} was implemented continuously throughout the test at a rate of motion of the movable crosshead of 0.63 mm/min. This property was calculated as follows:

$$PC_{II} = \frac{P_{max}}{A_O} \tag{3}$$

where PC_{II}: compression strength parallel to grain (MPa or N/mm^2); P_{max}: breaking load or maximum crushing load (N); b: width of the specimen (mm); t: thickness of the specimens (mm); and Ao: ($b \times t$) cross-sectional dimensions of specimens (mm^2).

HB normally determined using Janka hardness test method. The test was carried out based on Brinell-modified method using the modified ball test with a "steel ball" of 11.3 mm (0.444 in.) as an indenter and a force of 3000 kgf (29 kN; 6600 lbf). The predicted area of the ball on the test specimen was about 1 cm^2. The crosshead speed was 6.35 mm/min. This property was calculated based on the following equation:

$$HB = \frac{2P}{\pi D\left(d - \sqrt{D^2 - d^2}\right)} \quad (4)$$

where HB: hardness rate (kg/mm^2); D: ball diameter (mm); π: 3.14; P: ball weight (kg); d: the penetration depth of ball diameter in 30 s (mm).

Statistical Analysis

The results were analyzed by a one-way analysis of variance. The mean values of the properties were evaluated by Duncan's post hoc test (Duncan multiple range test; DMRT) at a 5% confidence level (CI). The normality of collective data was tested for skewness and Kolmogorov-Simrnov (Ho 2006). Pearson's correlation was calculated to determine relationships between wood mechanical properties and buffered media. Furthermore, simple linear regression equations were established to expect wood quality using planting density as predictor variables. The established models were evaluated based on the coefficient of determination (R) and significant levels. Correlation is concerned with the magnitude and direction of the relationship and regression focuses on using the relationship for prediction. Guilford's rule of thumb (1956) was used to describe the magnitude of correlations ($r < 0.2$: negligible relationship; $r = 0.2\text{-}0.4$: low relationship; $r = 0.4\text{-}0.7$: moderate relationship; $r = 0.7\text{-}0.9$: high relationship; and $r > 0.9$: very high relationship).

RESULTS AND DISCUSSION

The results and discussions in this were on the basis of visual observation, laboratory test, and analysis, and afterward the obtained data were analyzed using mathematical calculation and statistical analysis for a better interpretation and easy understanding of the experimental results. Mechanical properties of OPW were MOE (MPa), MOR (MPa), PC_{II} (N/mm^2), and HB (kg/mm^2) for both treated and untreated (control) samples.

Acidity Variation after Hydrothermal Treatment

The acidity of wood during the hydrothermal treatment was determined via measuring the pH of the liquor process before and after each treatment. Figure 2 indicated the level of acidity after the hydrothermolysis in the buffered medium and temperature. The highest and lowest changes between acidity before and after heat treatment (6.67 and 5 vs 3.49 and 5) were in water and buffer 5, respectively. The release of acid caused by hydrothermal treatment is predominantly limited by the process temperature and buffered media.

The increase in wood acidity during a thermal treatment of wood under wet conditions is due to the formation of removed carbonic acids, mainly acetic acid due to breaking of the acetyl groups of particular hemicelluloses. Further, catalyzes of carbohydrates cleavage cause a reduction in polymerization degree of them (Tjeerdsma et al 1998). Therefore, hydrothermal modification method in the buffered media is an effective method to control the destructive effects of acids formed via the degradation of carbohydrates

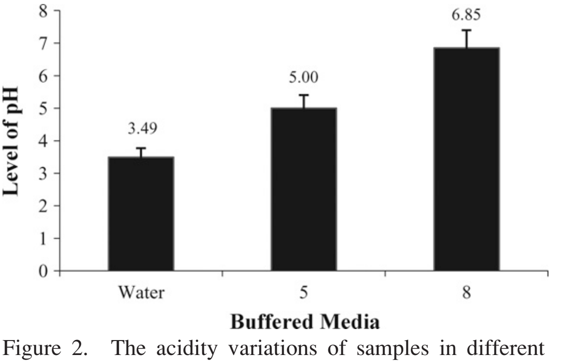

Figure 2. The acidity variations of samples in different buffered pH media after the hydrothermal treatment.

during the process (Talaei and Karimi 2012b; Ebadi et al 2015). In addition, the buffer solution is able to neutralize and control the acidity of the medium (Talaei 2010; Talaei et al 2013).

Mechanical Properties of the OPW Samples

In this study, several mechanical properties such as static bending, MOE, MOR, PC_{II}, and HB were measured. The testing was based on BS EN 373:1957 and ASTM (D143) standards for the mechanical properties evaluation. The analysis of mechanical properties of OPW was particularly examined the effect of the buffer solutions on the outer zone of OPW. The OPW specimens were categorized into two groups: first, the untreated wood (control); and the second, the treated wood within the buffered solutions using heat technique. The various chemical changes regularly lead to the modification of the various physical properties of wood and reduction in strength properties (Welzbacher et al 2011; Talaei and Karimi 2012b). In fact, the loss of strength properties always is one of the main concerns for a commercial utilization of thermally treated wood.

Strength Properties

Percentages of strength loss caused by the heat treatment in buffered media are listed in Table 2. It is difficult to direct comparison of the laboratory heat treatment process used in this study with the full-scale industrial process. However, interesting indications can be outlined. The treatment medium should be buffered in alkaline level to avoid significant decreases in MOE, MOR, PC_{II} and HB compared with control (untreated) samples.

Static Bending

The static bending (MOE, MOR) of the OPW samples were decreased after hydrothermal treatment. There were significant differences between the MOE and MOR of the treated and untreated samples ($p < 0.05$). No significant difference was determined between the treated samples ($p > 0.05$). The mean MOE and MOR before the hydrothermal treatment were 5907 MPa and 39.84 MPa, respectively. The treatment media indicated a decrease of 17.61% and 36.87% in water, 16.79% and 27.98% at a pH of 5, 23.44% and 24.30% at a pH of 8, respectively. The highest reductions in MOE and MOR were observed in tap water as tap water > buffer solution with pH of 5 > buffer solution with pH of 8 in comparison with the treated and untreated samples (Table 2).

The release of acid during hydrothermolysis could be dependent to the used treatment medium. It was concluded that hydrothermal treatment in buffer 8 medium obviously reduced the destructive effect of released acids and led to lessen MOE and MOR comparing with water and acidic media. Thus, there was a remarkably interaction between the media of treatment and MOE and MOR properties of specimens.

At the high temperatures, hemicelluloses are subjected to degradation (Boonstra and Tjeerdsma 2006). As Yildiz and Gümüşkaya (2007) expressed, the width and length of crystalline regions increased by reorientation of cellulose structure. Consequently, development of amorphous regions

Table 2. Mean MOE and MOR values for heat-treated and untreated samples of OPW.

Measured properties (MPa)	Untreated sample (Control)	Treated samples		
		Water	pH 5	pH 8
MOE	5907 (±811)	4866 (±1348)	4915 (±1171)	4522 (±953)
C.V. (%)	13.73	27.71	23.84	21.09
95% CI	5362.47-6452.37	3960.72-5773.05	4128.34-5703.02	3881.88-5163.56
MOR	39.84 (± 5.26)	25.15 (± 7.10)	28.69 (± 8.40)	30.16 (± 6.24)
95% CI	36.30-43.37	20.38-29.92	23.05-34.33	25.97-34.36
C.V.	13.20	28.23	29.27	20.70

C.V., coefficient of variation; CI, confidence interval.
The pH of tap water was 6.67. Values in parentheses indicate standard deviation.

Table 3. Mechanical properties loss of hydrothermally treated OPW in buffered media.

Mechanical properties	Untreated samples	Treated samples (%)		
		Water	Buffer 5	Buffer 8
PC_{CII} (MPa)	31.87 (±s4.33)	−8.75 (±2.27)	−11.38 (±2.79)	−16.05 (±3.47)
C.V.	13.60	25.57	24.53	21.64
CI	28.95-34.78	7.24-10.25	9.50-13.25	13.72-18.38
HB (kg/mm$^{2)}$	6.67 (±0.90)	−0.70 (±0.25)	−1.25 (±0.33)	−1.03 (±0.57)
C.V.	13.52	36.71	26.43	5.53
CI	6.07-7.28	0.53-0.87	1.03-1.48	0.99-1.07

has a considerable influence to the decrease of the MOE.

Compression Strength Parallel to Grain

According to Table 3 and Fig 3, PC_{II} of heat-treated specimens significantly decreased in all media. The release of acid during hydrothermolysis was dependent to the treatment medium and temperature applied.

The PC_{II} to grain of the treated OPW samples decreased. On the other hand, there was significant differences between the heat-treated samples in water and pH of 5 against the pH of 8 ($p < 0.05$). The mean PC_{II} before the hydrothermal treatment was 31.87 MPa, and the treatment media indicated a decrease of 72.58% in water and 64.32% in pH 5, and 49.64% in pH 8. The order of reduction in PC_{II} was observed in specimens treated in water > buffer 5 > buffer 8 (Fig 3).

Hardness Strength

According to Table 3 and Fig 4, the hardness of heat-treated specimens remarkably decreased in all media. There were significant differences between the treated samples in water with the pH of 5 ($p < 0.05$).

In addition, release of acid during hydrothermolysis was found to be dependent to the treatment medium and temperature applied. The average HB before the hydrothermal treatment was 6.67 kg/mm^2, and the treatment media indicated a decrease of 89.50% in water and 84.56% in pH 5, and 81.26% in pH 8. The order of HB reduction was observed in specimens treated in water > buffer 8 > buffer 5.

As Tjeerdsma and Militz (2005) stated, the reason for reduction in the strength properties of heat-treated wood samples could be related to cleavage of glycoside bonds of cellulose and shortening the cellulose chains initiated from the influence of acid on cell wall structure. This is in agreement with Talaei and Karimi (2012b).

Relationships between Buffered Media vs Mechanical Properties

The correlation between aqueous media and the mechanical features in hydrothermal treatment

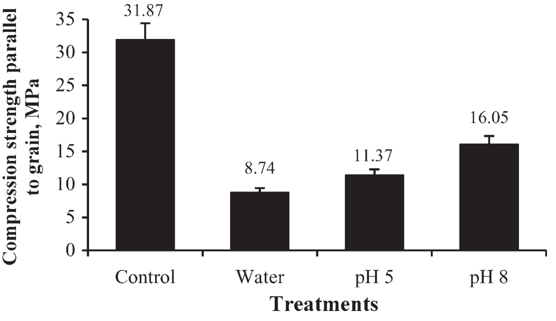

Figure 3. Compression strength parallel to grain of hydrothermally treated OPW in buffered media.

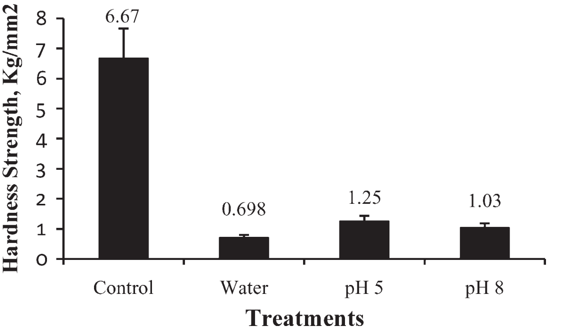

Figure 4. Hardness of hydrothermally treated OPW in buffered media.

method was examined for the OPW samples. Generally, the mechanical properties showed various levels of correlations with different pHs. The averages of the MOE, MOR, PC_{II}, and HB were negatively significantly correlated to the pH as −0.435, −0.533, −0.762, and −0.910, respectively. The highest degree of deacetylation occurs in acidic medium (buffer 5) that is probably due to acidification of the treatment medium during the process. Therefore, with releasing larger amounts of organic acids after thermal treatment, the mechanical properties will be decreased (Talaei 2010). On the other hand, the buffer 8 is representative of the neutralization of acids released by buffers, so that the reduction of the mechanical properties in buffer 8 is lower than the control. Furthermore, the effect of buffer 8 on controlling the destructive effects of released acids during the process is greater than the buffer 5. In addition, buffering the heat treatment medium at alkaline level can effectively control the negative effect of released acids on strength properties of wood (Talaei 2010).

The regression equations for the prediction of mechanical properties as a function of pH are illustrated in Fig 5a-d. The coefficient of determination (R^2) values for MOE, MOR, PC_{II}, and HB (Fig 5a-d) were 0.19, 0.28, 0.57, and 0.83, respectively. According to the R^2 values, moderate and high amount of variations in the properties were accounted for by the effect of pH. Therefore, most of the reduction values in OPW mechanical properties were described by variation in pH.

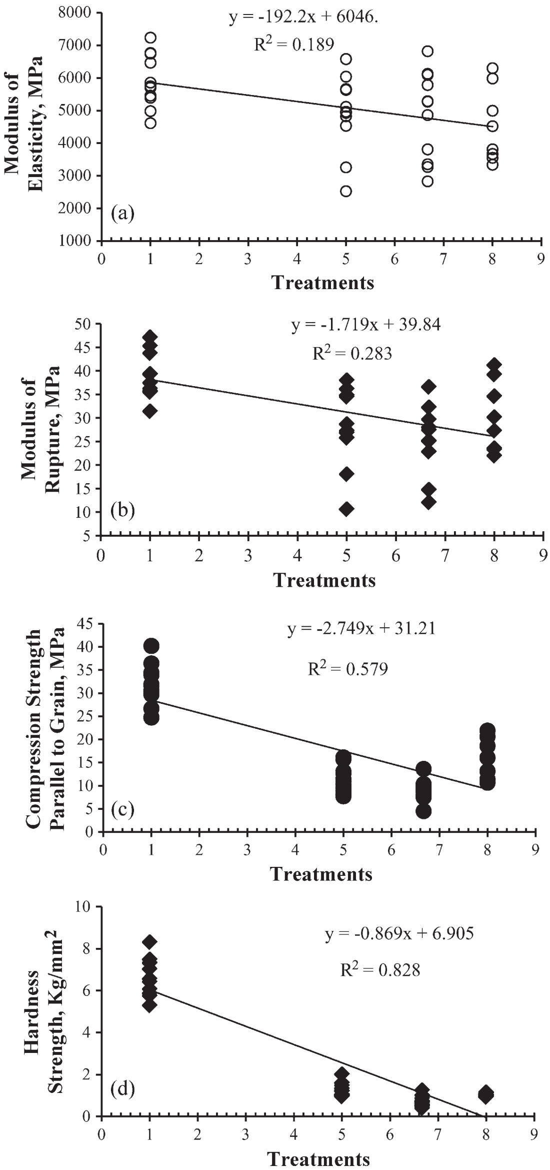

Figure 5. Relationship between treatments and mechanical properties (a) MOE, (b) MOR, (c) PC_{II}, and (d) HB. Note: Order of treatments from left to right: control, pH 5, tap water (pH 6.7), and pH 8.

CONCLUSIONS

The overall objective of this research was focused on the experimental investigations of the mechanical behaviors of OPW in various buffered media during hydrothermal treatment process. This idea was originated from the inherent flaws of the OPW. From the results of the present study, it can be concluded that

1. The acidity of the treated medium was increased during hydrothermal process. It seems that buffer medium can reduce the destructive effect of acid released during the heat treatment on the wood structure.
2. Acidic and alkaline buffers (5 and 8) neutralized the released acids and kept pH constant in certain level (about neutral).

3. The mechanical properties of OPW were reduced due to the effect of heat treatment on wood component.
4. Buffering hydrothermal treatments in the range of weak alkaline media is suggested to prevent a decline in mechanical strength.
5. Increasing the acidity of the treatment media significantly affected the mechanical properties of OPW.

ACKNOWLEDGMENT

The researchers would like to thank Universiti Putra Malaysia for providing the research grant that has made this work possible.

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