Physical Behavior of Hydro-thermally Treated Oil Palm Wood in Different Buffered pH Media

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This study investigated changes in the physical properties of oil palm *(Elaeis guineensis)* wood (OPW) using various buffered media for the hydrothermal treatment process. The buffered media were prepared separately for three different treatment conditions: pH of 8, pH of 5, and tap water. These treatments were compared with unbuffered, control samples. The OPW samples were taken from the outer part of the trees. The OPW samples were treated with the buffered media at a temperature of 140 °C for 120 min. The parameters evaluated were wood density (ρ), equilibrium moisture content (EMC), mass loss (ML), water absorption (WA), volumetric swelling (S_V), anti-swelling efficiency (ASE), and water repellent efficiency (WRE), for both treated and untreated samples. The buffered media significantly affected the EMC (%), ρ (g/cm^3), ML (%), and WA (%), with no significant effects on the ASE (%) and WRE (%). It was concluded that the hydrothermal treatment in the buffered medium with a pH of 8 had the most significant effect on the physical properties of OPW.

Keywords: Oil palm wood; Physical properties; Buffered mediums; Thermal modification

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INTRODUCTION

Oil palm *(Elaeis guineensis)* is a major agricultural commodity in Malaysia and generates more than 90 million tons of waste annually from empty fruit bunches, oil palm trunks (OPT), and oil palm fronds during the replanting process (Abdul Khalil *et al*. 2011). Oil palm wood (OPW) has several inherent defects, such as a high moisture content, low dimensional stability, low wood density, and a high proportion of parenchyma tissue, that affect its utilization. These defects increase the cost of processing and manufacturing. However, some of these weaknesses can be improved with appropriate treatment and conditioning; OPW has both exterior and interior uses (Kamarulzaman *et al*. 2004; Razak *et al*. 2008). However, disposing of oil palm stems by burning them pollutes the air and the environment, and it is considered an irrational action (Bhat *et al*. 2010).

Hydrothermal treatment is considered to be an eco-friendly method for wood modification without any usage of chemicals (Borrega and Kärenlampi 2009). Heat treatment can play an important role to increase the competitiveness of fast growing plantation wood by overcoming some of its weaknesses (Borrega and Kärenlampi 2009). Hydrothermal treatment is a cost-effective approach to enhance the inferior properties of

OPW. Moreover, the presence of water affects the chemical structure of thermally modified wood, and heat is easily transferred into the wood. Wood properties can be improved considerably by converting hydrophilic OH- groups into more hydrophobic groups. Heat treatment of wood reduces its hygroscopicity, which leads to less swelling and enhanced fungal resistance compared with untreated wood (Tjeerdsma *et al*. 1998; Kamden *et al*. 1999).

Acidic conditions and high temperatures can degrade wood by hydrolysis. During hydrothermal treatment, acidic conditions is produced by forming acids from the wood itself. Formic and acetic acids are produced during the heating process, especially at high temperatures (Tjeerdsma *et al*. 1998). The degradation rate of carbohydrates is high under acidic conditions, which is promoted by the high availability and low crystallinity of hemicelluloses (Theander and Nelson 1988).

Previous studies have revealed that only the outer parts of the mature OPT can be used as solid wood. These sections of the trunk produce the best quality OPW (Ratanawilai and Kirdkong 2006; Bakar *et al.* 2008; Rahayu 2001). Even so, the inherent defects mentioned earlier are still high in wood obtained from these parts. As a result, comprehensive treatment must be carried out to solve these problems. Treatment with a modified compression method has proven to be effective in solving the first three unfavorable characteristics: high moisture content, low dimensional stability, and low wood density (Ratanawilai and Kirdkong 2006; Bakar *et al*. 2008). During the hydrothermal process, the pH level of a hydrothermal medium may become more acidic. Buffered mediums can be used to maintain less change in pH level during hydrothermal treatment to control the destructive effects caused by the released acids. According to this, hydrothermal process can be applied to the OPW, determining how buffered mediums would affect the physical properties that need to be evaluated.

The objectives of this research are to determine the physical behaviors of oil palm wood when heated in three different buffered mediums as suitable hydrothermal treatments: alkaline buffer with pH 8, acidic buffer with pH 5, and water medium.

EXPERIMENTAL

Materials and Methods

Sample preparation

Three mature oil palm trees were randomly harvested at the Universiti Putra Malaysia's oil palm plantation. Three logs, 2 m in length, were taken above the tree breast height. The logs were then processed and flat sawn into boards with dimensions of 60 × 5 × 5 cm (length × width × thickness) (Fig. 1). The outer OPW timbers were selected based on similar wood densities. To avoid losses from fungus and moisture, the boards were kept in a cold room at 4 °C.

Hydrothermal treatment was carried out in three buffered mediums, *i.e.*, pH 5 and 8 and water medium, at 140 °C for 120 min. Tap water with pH of 6.7 was used as one of the treatment media. For measuring the mass loss, the sample was first oven-dried and then put on the digester. For other measurments, green samples with the moisture of about 114% were placed in a digester (JSR Instruments, Digester 30.4, Uttarakhand, India) and treated with the three buffered media. Thus, all OPW samples were under buffered and unbuffered

(control) hydrothermal treatments. To prevent changes in the pH during the heating process, buffered media were used to keep the pH constant (Talaei 2010).

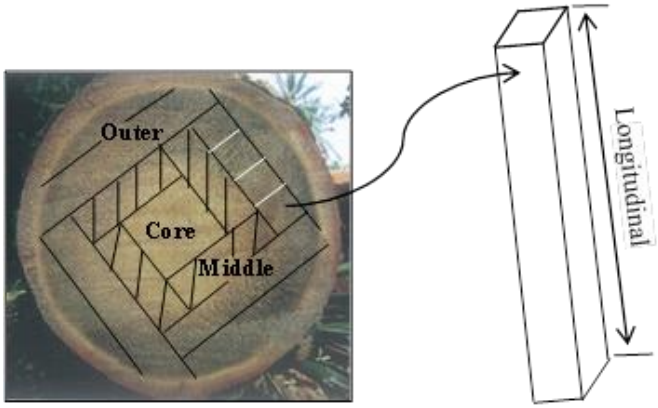

Fig. 1. Flat sawn cutting for board preparation

After the hydrothermal treatments, the samples were gradually cooled to room temperature before they were cut into 20 × 20 × 20 mm specimens, which were used to evaluate their physical properties. About 10 samples were prepared for each determined property. Untreated samples were used as the controls for comparison purposes. The samples were then transferred into a conditioning room with a temperature of 20 ± 2 °C and a relative humidity (RH) of 65 ± 3 % until they reached a constant weight, *i.e.*, equilibrium moisture content (EMC, 12 ± 2%). Once all the samples reached a constant weight, the percent moisture content (MC, %), EMC (%), mass loss (ML, %), water absorption (WA, %), anti-swelling efficiency (ASE, %), water repellent efficiency (WER, %), and oven-dry wood density (ρ, g/cm^3) were calculated. All of the tests were conducted in accordance with the procedures specified by the British-adopted European standard test methods for small, clear samples of timber, BS 373 (1957). These properties were calculated as follows:

$$MC\% = \left(\frac{W_1 - W_{OD}}{W_{OD}}\right) \times 100 \quad (1)$$

$$EMC\% = \left(\frac{W_{EMC\%} - W_{OD}}{W_{OD}}\right) \times 100 \quad (2)$$

where, MC% is the initial moisture content of the green samples (%), EMC% is the equilibrium moisture content (12 ± 2%), W_1 is the initial weight (g), W_{OD} is the oven-dry weight (g), and $W_{EMC\%}$ is the weight for the equilibrium moisture content state (g),

$$\rho = \frac{W_{OD}}{V_{OD}} \quad (3)$$

where, ρ is the wood density (g/cm^3), W_{OD} is the weight after oven-dry (g), and V_{OD} is the oven-dry volume (cm^3);

$$ML\% = \left(\frac{W1_{OD} - W2_{OD}}{W2_{OD}}\right) \times 100 \quad (4)$$

where, ML% is the mass loss (%), W_{1OD} is the oven-dry weight of the samples before being treated (g), and W_{2OD} is the oven-dry weight of the samples after treatment (g),

$$WA\% = (\frac{W_t - W_{OD}}{W_{OD}}) \times 100 \quad (5)$$

where, WA% is the water absorption (%), W_t is the weight of the wet samples after immersion in distilled water (g), and W_{OD} is the weight of the oven-dry samples (g),

$$S_V\% = (\frac{V_2 - V_1}{V_1}) \times 100 \quad (6)$$

where, Sv% is the volumetric swelling coefficient (%), V_2 is the volume of the saturated samples or the sample's volume after immersion in distilled water (mm^3), V_1 is the volume of the oven-dried samples before saturation (mm^3). V is the volume calculated from the longitudinal (L), tangential (T), and radial directions,

$$ASE\% = (\frac{S_u - S_t}{S_u}) \times 100 \quad (7)$$

where, ASE% is the anti-swelling efficiency (%), S_t is the volumetric swelling coefficient of the treated samples (%), and S_u is the volumetric swelling coefficient of the untreated samples (%); and

$$WRE\% = (\frac{WA_1 - WA_2}{WA_1}) \times 100 \quad (8)$$

where, WRE% is the water repellent effectiveness (%), WA_1 is the rate of water absorption of the untreated samples, and WA_2 is the rate of water absorption of the treated samples.

Buffered solutions were prepared in the laboratory as shown in Table 1.

Table 1. Preparation of the Buffered Solutions

pH	Chemical Composition
5	257.5 mL $Na2HPO_4.2H_2O$ (0.2 M) + 242.5 mL $C_6H_8O7.H_2O$ (0.1 M)
8	486.25 mL $Na2HPO_4.2H_2O$ (0.2 M) + 13.75 mL $C_6H_8O_7.H_2O$ (0.1 M)

Source: Alexeyev (1967)

Statistical Analysis

The results were analyzed using a one-way analysis of variance (ANOVA). The mean values of the determined properties were evaluated using the Duncan's post-hoc test (Duncan Multiple Range Test; DMRT) at a 5% confidence level.

RESULTS AND DISCUSSION

Variation of pH after Hydrothermal Treatments

Wood acidity after hydrothermolysis was determined by measuring the process liquor. Figure 2 shows the pH level achieved after hydrothermal treatment in the two buffered mediums and water (pH 6.7). During the hydrothermal process, acid that was released into the solution produced a decrease in pH and hemicellulose acetylation.

Hydrothermal modification method in the buffered media is an effective method to control the destructive effects of acids formed *via* the degradation of carbohydrates during the process (Talaei 2010, 2013). During thermal treatment process, the formation of acetic acid resulting from hemicellulose acetyl groups increases the acidity (pH) of treatment medium (Tjeerdsma and Militz 2005; Sundqvist *et al.* 2006). Heat treatment in alkaline and acidic buffered mediums decreases the thermally induced degradation in wood. In addition, the acidic buffer solution increase crystallinity index and higher rate of lignin. Meanwhile, the alkaline buffer considerably controls the carbohydrate degradation in the buffered treatment (Talaei *et al.* 2012a,b; Talaei *et al.* 2010, 2011; Taghiyari *et al.* 2011). Therefore, the buffer solutions can neutralize and control the the acidity of the medium.

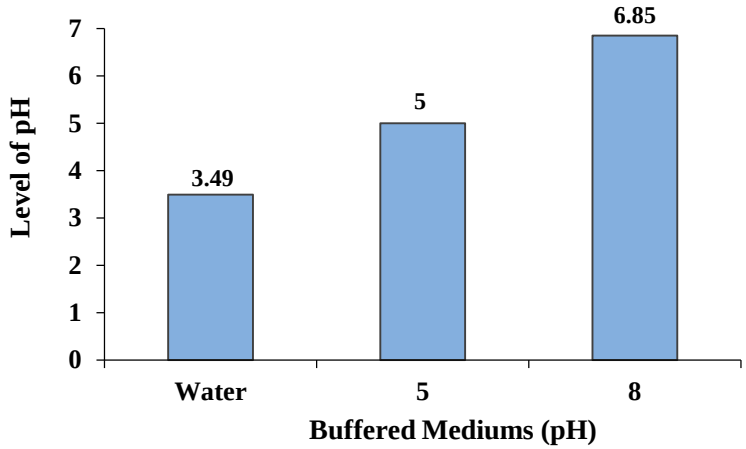

Fig. 2. Variations of pH of samples in buffered mediums and water, after the hydrothermal treatment

Physical Properties of the OPW samples

The MC of the green OPW samples ranged from 100 to 500% (Kilmann and Lim 1985; Bakar *et al*. 2008). Table 2 shows the initial MC and EMC at different buffered solutions of treated and untreated OPW. The control samples of OPW had a green MC of 114% because of the removal of the extractives from the cellular structure of OPW and the loss in hydroxyl groups (-OH) post-treatment (Tjeerdsma *et al*. 1998; Sandberg *et al*. 2013; Talaei *et al.* 2013).

Table 2. Moisture Contents of OPW Samples in Different Solution Before and After Treatment

Properties	Untreated samples (Control)	Treated specimens in the buffer solutions		
		Tap-water	pH 5	pH 8
Initial MC	114.00	116.33	112.22	115.38
EMC	14.71	10.27	12.45	11.94

MC, moisture content; EMC, equilibrium moisture content; The pH of tap water was 6.67

There were significant differences between the EMC of the heat-treated samples and that of the untreated samples ($p < 0.05$). Untreated samples had an EMC of 14.7%, and showed a decrease of 30.36 % in water, 13.63% at a pH of 5, and 18.79% at a pH of 8. The EMC of wood was expected to decrease because of the reduction in the number of -OH

groups, which participate in hydrogen bonding with water molecules (Sandberg *et al*. 2013). However, the EMC of wood reached a saturation point when the reaction was at equilibrium. Therefore, EMC reduction is caused by the increased crystallinity of cellulose, which reduces the availability of hydroxyl groups to water molecules (Salim *et al*. 2010; Yuliansyah and Hirajima 2012).

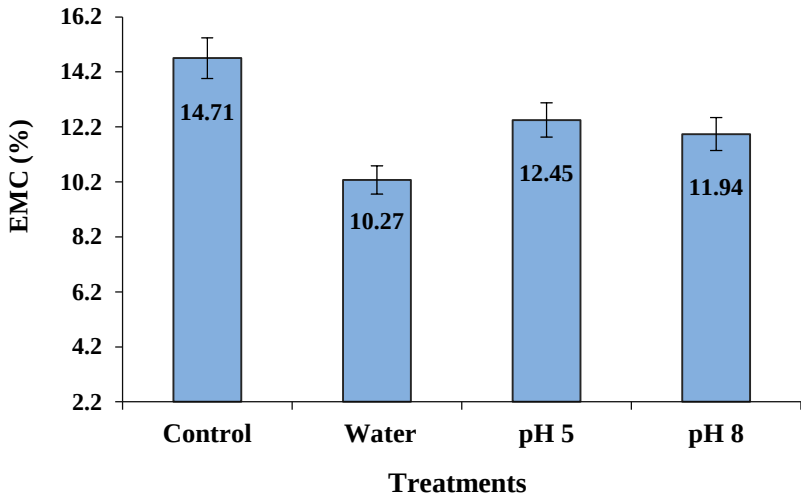

Fig. 3. Variation in EMC (%) for treated and untreated OPW samples [The confidence intervals (CI) of treatments were 14.37-15.04; 9.82-10.62; 12.23-12.39; 11.78-12.09, respectively from left to right].

The -OH groups that were present in the hemicellulose component absorbed water. The hydrothermal treatments achieved some modifications in the OPW samples. However, the modifications that occurred in the chemical composition of hemicelluloses resulted in the degradation of -OH groups. This is because hemicellulose can be thermally degraded at a lower temperature than cellulose (Wikberg and Maunu 2004). Hence, the fact that treated OPW samples had a lower EMC than the control OPW samples was attributed to the degradation of hemicellulose during the hydrothermal treatment. Hemicellulose is more hygroscopic; its structure contains a larger number of -OH groups compared to cellulose and lignin. Therefore, cellulose and lignin contribute minimally to the hygroscopic properties (Khalil *et al*. 2007). Furthermore, the cellulose crystallinity is increased due to the degradation of amorphous areas, which decreases the availability of hydroxyl groups to water molecules and also reduces the (EMC) (Wikberg and Maunu 2004; Bhuiyan and Hirai 2005; Boonstra and Tjeerdsma 2006).

The density of all OPW samples had been decreased after the hydrothermal treatment. Although there were differences in the densities of the treated and untreated samples, they were not significantly different ($p > 0.05$). The mean density before the hydrothermal treatment was 0.57 g/cm^3, with a decrease of 1.07% in water, 1.27% at a pH of 5, and 4.5% at a pH of 8.

Some studies have indicated that thermal treatment results in a decline in density (Tjeerdsma and Militz 2005). The main reason behind this may be related to the instability of hemicelluloses with heat. These components easily decompose at high temperatures in water-soluble and carbohydrate compounds (Yildiz *et al*. 2003). During the hydrothermal process, degradable materials are gradually emitted from the cell walls and are carried into the medium. This process causes a decline in density. Furthermore, this method influences

the removal of extractives, hemicellulose hydrolysis, and changes in the lignin and cellulose components (Garrote *et al*. 1999; Sandberg and Navi 2007). At higher temperatures, the degradation rates are accelerated and accompanied by a decrease in density.

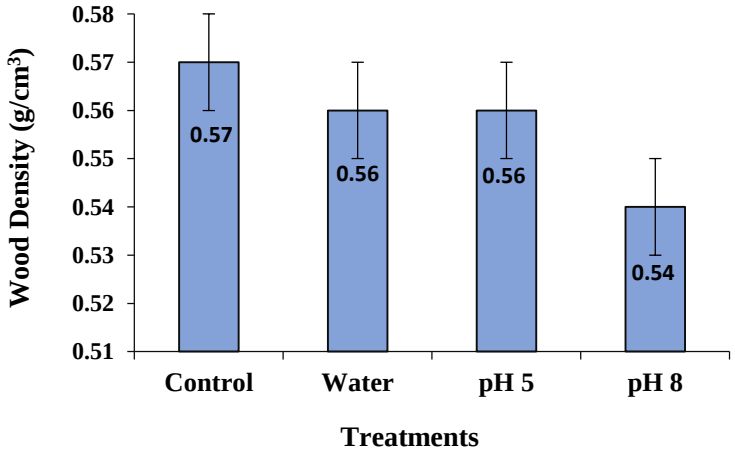

Fig. 4. Variation of wood density for treated and untreated OPW sample [CI of treatments were 0.53-0.63; 0.52-0.61; 0.53-0.64; 0.52-0.60, respectively from left to right].

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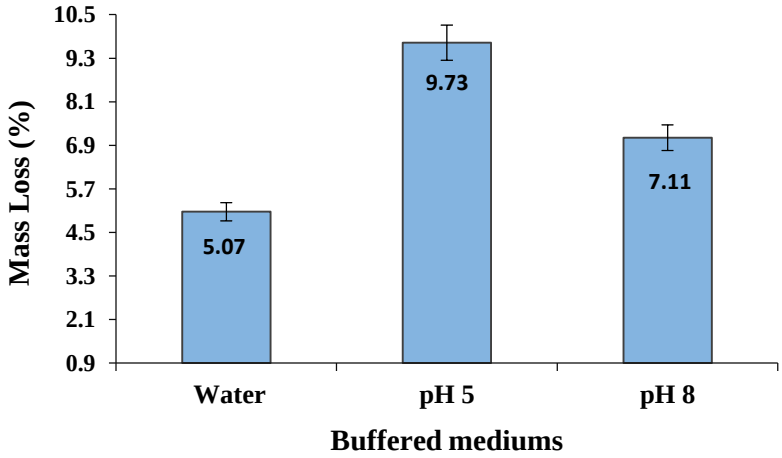

Fig. 5. Mass loss percentage of the treated samples in different aqueous pH buffers (The bars show standard deviations; CI of treatments were 5.44-7.17; 9.38-12.44; 3.88-10.34, respectively from left to right).

The ML at a pH of 5 revealed considerable degradation of the wood contents (the exractives, starch, and the cell wall of parancyma tisse). Therefore, acidic conditions may significantly increase the degradation of wood polymers *via* acidic hydrolysis at different hydrothermal treatments ($p < 0.05$) (Tjeerdsma *et al*. 1998). More specifically, in hyrothermal treatment, the exception of hydrogen (H^+) and hydroxide ($-OH^-$) ions, the rest of ions will not change the pH of the treated medium (especially in 140 °C). Therefore, a high amount of H^+ and OH^- will result into high acidic and alkaline pH values, respectively. Moreover, pH of the treated medium using the buffered solutions is kept constant at a certain level of pH and also the degradation effect of the released acids can be controled in the process of hydrothermal (Talaei 2010). Consequently, the heat treatment in buffered

mediums decreases the thermally induced degradation in the wood (Talaei *et al*. 2012a,b; Talaei *et al.* 2011; Taghiyari *et al.* 2011). On the contrary, in the conditions with no buffering medium (tap water), the acidity will highly increase as observed in water condition with a pH of 3.49.

According to the results, differences in the ML between treatment groups were significant ($p < 0.05$). In addition, the dry weight of the samples decreased after the hydrothermal treatment. The ML exhibited a decrease of 6.31% in water, 10.91% at a pH of 5, and 7.11% at a pH of 8.

The main reason for these declines may be related to the removal of extractives, starch, and degradation of paranchyma cells. Therefore, according to removal of extractives and starch from the wood structure, their high dissolvability in water and aqueous solutions, ML, and decreasing wood density seemed to be reasonable (Talaei and Yaghoobi 2009; Talaei *et al*. 2013).

Volumetric Swelling and Water Absorption

Based on the analysis, a significant difference in the S_V at 2, 24, and 48 h occurred between buffered and unbuffered (control) samples ($P < 0.05$; Fig. 6). The mean volumetric shrinkage showed no significant difference among treated samples. Furthermore, the maximum and minimum values were in the buffered medium with pH 8 and in control samples, respectively. The S_V of the control samples at 2, 24, and 48 h were approximately 6.90, 7.77, and 7.95%, respectively. The S_V showed an increase of 83.38, 76.77, and 75.76% in water; 92.33, 77.92, and 79.19% at pH 5; and 106.15, 86.12, and 85.45% at pH 8, respectively.]

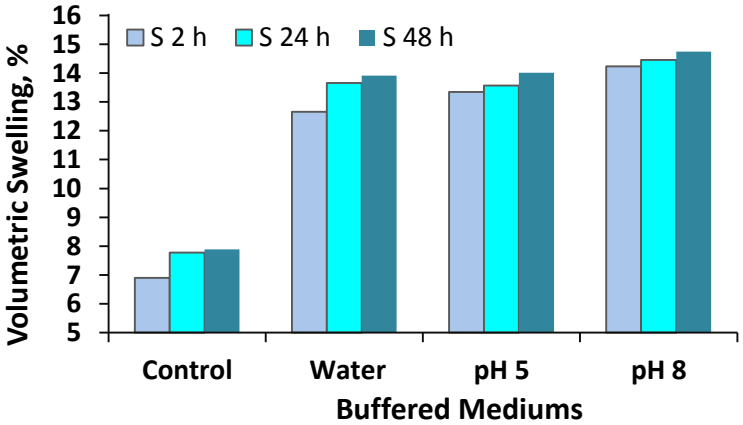

Fig. 6. Volumetric swelling at 2, 24, and 48 h for the treated and untreated OPW samples

The cell wall components can be destroyed during the thermal treatment. So that, except for the removal of extractives and starch, a great volume of cell walls of parenchyma cells may also be destroyed, resulting in the significant increase of volumetric swelling (Bourgois *et al.* 1989 and Garrote *et al*. 1999).

Figure 7 shows that the maximum rate of WA occurred after the first 15 min of soaking in distilled water. There were significant differences ($p < 0.05$) between untreated samples and samples treated at a pH of 5, in comparison with samples treated at a pH of 8 and in water.

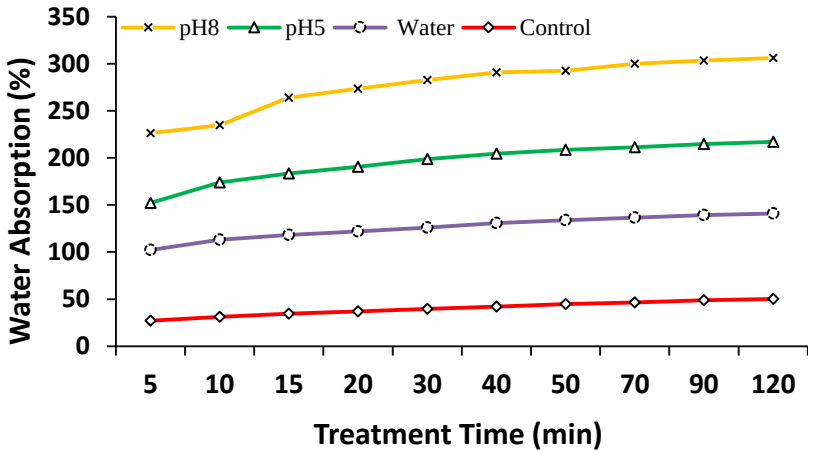

Fig. 7. Variation in water absorption during 120 min of soaking for treated and untreated OPW samples in distilled water

Concerning the WA at 2 and 24 h, significant differences ($p < 0.05$) were observed between the treated samples in the buffered medium for water and a pH of 8, compared with the untreated (control) samples and samples treated at a pH of 5 (Fig. 8) ($p < 0.05$). There were also significant differences in water absorption at 48 h between the samples treated in water and those treated at a pH of 5, compared to the untreated samples ($p < 0.05$). No significant differences occurred between the treated samples at a pH of 8 and the samples in treated water and at a pH of 5 ($p < 0.05$). However, there was a significant difference between treated samples at a pH of 8 and the untreated samples. Furthermore, the WA of the untreated samples at 2, 24, and 48 h was 49.24, 63.40, and 65.69%, respectively. Results from Fig. 8 demonstrate an increase in the WA for treatment groups: 81.35, 57.28, and 59.16% for water; 49.63, 31.43, and 32.30% at a pH of 5; and 80.29, 55.78, and 55.91% at a pH of 8, respectively.

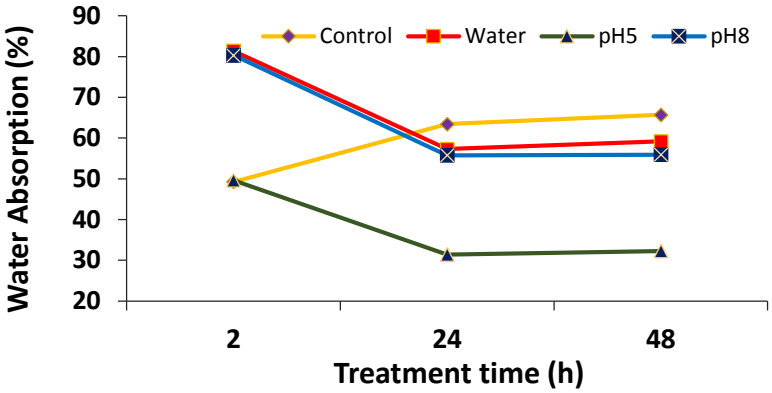

Fig. 8. Variations of water absorption at 2, 24, and 48 h

It appears that the chemical and cellular structure, *i.e.* extractives (up to 9.8%), abundant amount of the main free sugars such as sucrose, glucose, and fructose (10%), and great content of starch (25%) in the parenchyma cell walls of the OPW were significantly degraded and removed after the hydrothermal treatment (Khalil *et al.* 2007). Thus, the porosity and water absorption were increased. Therefore, this response could have been

due to the high percentage of contents removed from cell walls (Erakhrumen and Olukayode 2009).

Table 3. Mean ASE (%) and WRE (%) Values for Heat-Treated and Untreated OPW Samples

Properties	Time (h)	Water	pH 5	pH 8
ASE%	2	-89.14 (±61.88)	-98.81 (±64.20)	-116.59 (±40.15)
	24	-85.44 (±72.88)	-80.59 (±60.74)	-100.84 (±48.56)
	48	-85.80 (±73.63)	-82.46 (±61.47)	-101.44 (±45.85)
WRE%	2	-79.77 (±42.88)	-48.54 (±34.02)	-77.32 (±32.63)
	24	-55.11 (±25.58)	-30.12 (±27.22)	-54.09 (±16.11)
	48	-58.43 (±32.48)	-34.14 (±28.02)	-53.91 (±22.23)

ASE%: anti-swelling efficiency; WRE%: water repellent efficiency. Values in parentheses indicate standard deviation.

According to Table 3, the ASE for treated samples at 2, 24, and 48 h showed a decline of 89.14, 85.44, and 85.80% in water, 98.81, 80.59, and 82.46% at a pH of 5, and 116.59, 100.84, and 101.44% at a pH of 8, respectively. This showed that the highest ASE% was obtained at a pH of 8.

The WRE was lower for treated than untreated samples after 2, 24, and 48 h, with declines of 79.77, 55.11, and 58.43% in water, 48.54, 30.12, and 34.14% at a pH of 5, and 77.32, 54.09, and 53.91% at a pH of 8, respectively (Table 3). The thermal treatment in the buffered mediums actively prevent the degradation of cellulose and hemicellulose (Talaei 2010).

The alterations of the chemicals in the structure of wood can be a main reason for the more porous nature, the decline of ASE and WRE, and also the high absorbency rate (Husin *et al*. 1985; Tomimura 1992; Siti Norbaini 2009). Zaihan *et al.* (2011) reported that the water absorbed is more placed within the starch than the cell wall. In addition, high absorbency rate was observed in treated wood due to the thin-walled of the parenchyma tissues and more porosity (Paridah *et al.* 2006).

CONCLUSIONS

This study has provided an account of the effects of hydrothermal modification on the physical properties of OPW in the various buffered mediums. The main findings of the present study are as follows:

1. The acidity of the treated medium was increased after hydrothermal process. It follows that buffer can decrease and control the destructive effect of acid on the wood structure.

2. The EMC was reduced due to the effect of heat treatment. Thus, this reduction affected the wood quality by increasing of the cellulose crystalinity and also degradation of the amorphous part.
3. The wood density was somehow reduced at pH 5 and pH 8 with no differences between them. Furthermore, the reduction of weight loss was highly related to the removal of high percentage of extractives, starch, and parenchyma tissue from the palm wood.
4. Due to the neutralization effect of buffer solution, the acidity of medium and the rate of carbohydrates degradation was decreased. This reaction may chemically restrain and control the destructive effects of hydrothermal treatment on some structural properties of the wood.

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