

# HYDROTHERMAL TREATMENT OF OIL PALM WOOD: EFFECT OF TREATMENT VARIABLES ON DIMENSIONAL STABILITY USING RESPONSE SURFACE METHODOLOGY

MUHAMMAD ANUAR RIDZUAN SALIMAN\*; ZAIDON ASHAARI\*; EDI SUHAIMI BAKAR\*;  
 LEE SENG HUA\*; PARIDAH MD TAHIR \*\*; JULIANA ABDUL HALIP\*\* and NABIL FIKRI LEEMON\*\*

## ABSTRACT

Oil palm wood (OPW) extracted from felled oil palm trunks during replanting could be a source of replenishment for the depleted timber sources. Nevertheless, dimensional instability of OPW is one of the drawbacks that need to be addressed before it can be utilised efficiently. Therefore, to improve its dimensional stability, hydrothermal treatment using different buffered media was employed in this study and the optimal treatment condition for OPW were investigated. Response surface methodology (RSM) models for treatment temperature, treatment duration and buffered media of hydrothermally treated OPW were developed. OPW samples were hydrothermally treated with different buffered media (pH 4 to pH 9), treatment temperature (60°C to 160°C) and time (13 min to 147 min). Water absorption (WA) and thickness swelling (TS) of the treated samples were determined. It is demonstrated that hydrothermal treatment has improved the dimensional stability of treated samples with treatment temperature appearing to be the most influential factor. Samples treated in an acidic media displayed lower WA mainly due to the fact that higher amount of hydrophilic hemicellulose was degraded under acidic condition. However, lower TS was observed in the samples treated in alkaline media, probably caused by the removal of lignin that increased the porosity of the OPW.

**Keywords:** buffered media, central composite design, hydrothermal treatment, thickness swelling, water absorption.

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## INTRODUCTION

In 2015, the coverage of oil palm plantations in Malaysia has reached a new high of around 17% of the total land area covered by oil palm. There are 5.64 million hectares of oil palm plantations in 2015,

an increase of 4.6% against the 5.39 million hectares recorded in the previous year (MPOB, 2015). Replanting practice at the end of their economic life span has generated a huge amount of biomass. Bakar *et al.* (2013) reported that an estimated 230-250 m<sup>3</sup> of oil palm stems per hectare could be obtained during replanting. Oil palm wood (OPW) extracted from these felled oil palm stems is an ideal substitution for the depleting timber resource. Utilised effectively, alternate wood products derived from OPW could potentially alleviate the deforestation rate in Malaysia which has seen its forested area plummeting from 68.11% in 1990 to 61.73% in 2012. Oil palm trunks and fronds have been used for particleboard and slow release fertiliser production (Lee *et al.*, 2015; Nur Nabilah

\* Department of Forest Production,  
 Universiti Putra Malaysia,  
 43400 UPM Serdang,  
 Selangor, Malaysia.  
 E-mail: zaidon@upm.edu.my

\*\* Laboratory of Biocomposite Technology,  
 Institute of Tropical Forestry and Forest Products,  
 Universiti Putra Malaysia,  
 43400 UPM Malaysia,  
 Selangor, Malaysia.

*et al.*, 2015). Unfortunately, the poor dimensional stability of OPW as reported by Bakar *et al.* (2013) has consequently inhibited OPW from being utilised effectively to substitute wood. The main concern of the present study is to improve the dimensional stability of OPW using an effective treatment.

Hydrothermal treatment is one of the effective thermal treatment methods that have been implemented by several researchers (Endo *et al.*, 2016; Tjeerdsma and Millitz, 2005). The efficiency of hydrothermal treatment is highly dependent on treatment temperature and time. Apart from that, the heating media also play an important role. Hydrothermal treatment of beech wood has been conducted in acidic, neutral and alkaline buffered solutions by Talaei and Karimi *et al.* (2015) and varied mechanical properties were observed in the wood treated using different buffered media. To the authors' knowledge, reports on the influence of hydrothermal treatment using different buffered media on the dimensional stability of OPW are scarce in the literature. Therefore, the present study aimed to improve the dimensional stability of OPW using hydrothermal treatment. Different buffered solutions were used as heating media to investigate the relationship of buffered solutions and dimensional stability. Optimisation models for hydrothermal conditions using response surface methodology (RSM) were also developed in the present work. The current work served as a preliminary study to understand the behaviour of OPW treated in buffered media for potentially further investigation.

## MATERIALS AND METHODS

### Materials Preparation

Twenty-five-year old oil palm (*Elaeis guineensis*) were harvested at the University Agriculture Park, Universiti Putra Malaysia (UPM), Selangor, Malaysia. The harvested oil palm were then flat sawn into dimensions of 60 cm × 5 cm × 5 cm in the Forest Research Institute Malaysia (FRIM), Kepong, Selangor, Malaysia. All the samples were stored in cold rooms in UPM at a temperature of 4°C to prevent fungal attack and prevent moisture loss.

### Response Surface Methodology (RSM) and Central Composite Design

Central composite design (CCD) using RSM was used in the present study to investigate the effects of treatment variables on the dimensional stability of oil palm wood and bamboo. Three independent variables, namely, buffered media (pH), treatment temperature (°C) and treatment time (min) were selected and the response variable were thickness swelling (TS) and water absorption (WA). The CCD was conducted using Design Expert software (State Ease, Design Expert 9). A 20-run CCD using RSM was developed and the ranges of the variables are shown in *Table 1*. Each of the independent variable was coded by five different levels as shown in *Table 1*, where the pH, treatment temperature and treatment times ranged from 4°C to 9°C, 60°C to 160°C and 13 to 147 min, respectively. The experimental conditions of coded and actual values developed using RSM with central composite design are shown in *Table 2*.

### Hydrothermal Treatment Schedule

Hydrothermal treatment was carried out by boiling the samples in different buffered media ranging from pH 4 to 9 above the atmospheric pressure by using twin digesters. Twenty OPW with dimensions of 60 cm × 5 cm × 5 cm, one for each run, were placed into twin digester. Buffered solution with pH values ranging from pH 4 to 9 were poured into the twin digester until the samples were fully immersed. Buffered solution was subsequently heated to the required temperature ranging from 60°C-160°C as suggested by RSM models. After the respective required treatment times (13 to 147 min), the treated samples were removed from the digester and cut into 50 mm × 50 mm × 10 mm followed by dried in the oven at 103 ± 2°C for 18 hr prior to dimensional stability evaluation.

### Dimensional Stability Evaluation

WA and thickness swelling TS of treated OPW samples were calculated based on 24 hr submersion in distilled water. Five oven-dried samples for each run were immersed in distilled water in a beaker

TABLE 1. THE RANGE AND LEVELS OF THE VARIABLES

Factor	Variable	Units	Range and level of actual and coded values				
			- $\alpha$	-1	0	1	$\alpha$
X <sub>1</sub>	pH	-	4	5	6.5	8	9
X <sub>2</sub>	Temperature	°C	60	80	110	140	160
X <sub>3</sub>	Time	min	13	40	80	120	147

TABLE 2. EXPERIMENTAL DESIGN USING CENTRAL COMPOSITE DESIGN

Run	Coded factor			Actual factor		
	X <sub>1</sub>	X <sub>2</sub>	X <sub>3</sub>	pH	Temp (°C)	Time (min)
1	-1	-1	-1	5	80	40
2	1	-1	-1	8	80	40
3	-1	1	-1	5	140	40
4	1	1	-1	8	140	40
5	-1	-1	1	5	80	120
6	1	-1	1	8	80	120
7	-1	1	1	5	140	120
8	1	1	1	8	140	120
9	-α	0	0	4	110	80
10	α	0	0	9	110	80
11	0	-α	0	6.5	60	80
12	0	α	0	6.5	160	80
13	0	0	-α	6.5	110	13
14	0	0	α	6.5	110	147
15	0	0	0	6.5	110	80
16	0	0	0	6.5	110	80
17	0	0	0	6.5	110	80
18	0	0	0	6.5	110	80
19	0	0	0	6.5	110	80
20	0	0	0	6.5	110	80

and placed in a vacuum apparatus for 15 min evacuation. After evacuation, the samples were left immersed in the water at atmospheric pressure for 24 hr. The weights and thickness before and after the soaking process were determined.

RESULTS AND DISCUSSION

Regression and Adequacy of the Model

From the experimental result, two linear equations were obtained. The final equations in terms of actual factors are shown below:

$$WA = 18.30 + 0.44pH + 0.14Temperature + 0.03Time$$

$$TS = 6.38 - 0.08pH - 0.02Temperature - 0.004Time$$

To ensure the fitted model gave a sufficient approximation of the results obtained in the experimental conditions, the adequacy of the model was evaluated. Insignificant lack-of-fit values of 0.8152 and 0.6296 for WA and TS, respectively, suggested that the model was statistically appropriate for further investigation. The fit of the model was evaluated using coefficient of multiple regression (r<sup>2</sup>) and adjusted r<sup>2</sup> was used for confirmation of the model adequacy. Based on the analysis, r<sup>2</sup> values of 0.9333 and 0.9005 for the WA and TS, respectively, indicated high fitness of the model. The adequacy of the model was further proved by high adjusted r<sup>2</sup> of 0.9208 and 0.8818, respectively.

Effects of Hydrothermal Treatment on Dimensional Stability of the Samples

The effects of variables (pH, treatment temperature and time) on the response of WA and TS of the hydrothermally treated samples are presented in Figures 1 and 2. When the OPW samples were treated at 160°C for 80 min, the lowest TS of 2.59% were obtained. On the contrary, the highest TS of 4.60% was recorded with the samples treated at 60°C for 80 min. Lower TS could be obtained when treating the samples at higher temperature even when shorter time was used. For example, OPW samples treated at 140°C for 40 min resulted in a TS of 2.76%, which was slightly lower than the TS of OPW samples (2.87%) that subjected to lower temperature but longer time (110°C for 147 min). From the given example, it could be seen that treatment temperature is a more influential factor than that of treatment time. The findings suggested that 107 min of treatment time could be cut off when the treatment temperature was raised by 30°C. The WA pattern mirrored that of TS as lower WA obtained when subjected to higher treatment temperature. The obtained results were comparable to the findings reported by other researchers who treated OPW with different methods. Maminski *et al.* (2016) impregnated OPW with 1,3-dimethylol-4,5-dihydroxyethyleneurea and found that the TS and WA of the treated OPW was 1.3%–2.5% and 19%–27%, respectively. Another modification method called furfurylation was carried out by Szymona *et al.* (2014) on OPW and the TS values of 1.1%–1.4% and WA values of 30%–45% were recorded. Destruction of several hydroxyl groups has reduced the affinity of the wood to water and therefore led to improvement in dimensional stability (Weiland and Guyonnet, 2003). The inaccessibility of the hydroxyl group to water molecules due to the increase of cellulose crystallinity during heat treatment has also imparted better stability in dimension to the treated wood (Boonstra and Tjeerdsma, 2006).

On the other hand, the pH values of the heating media exerted influence when both treatment temperature and time remained constant. Treated at the same level of treatment temperature and time, samples treated in the alkaline media (pH > 6.5) displayed better TS compared to samples treated in an acidic media. The findings were in agreement with Ebadi *et al.* (2015) who reported that the highest anti-swelling efficiency (ASE) was attained in the samples treated in a buffered media with pH of 8. Nevertheless, contrary observation for WA was obtained as higher WA was found in the samples treated using alkaline media. Formic and acetic acids are formed during heat treatment and these released

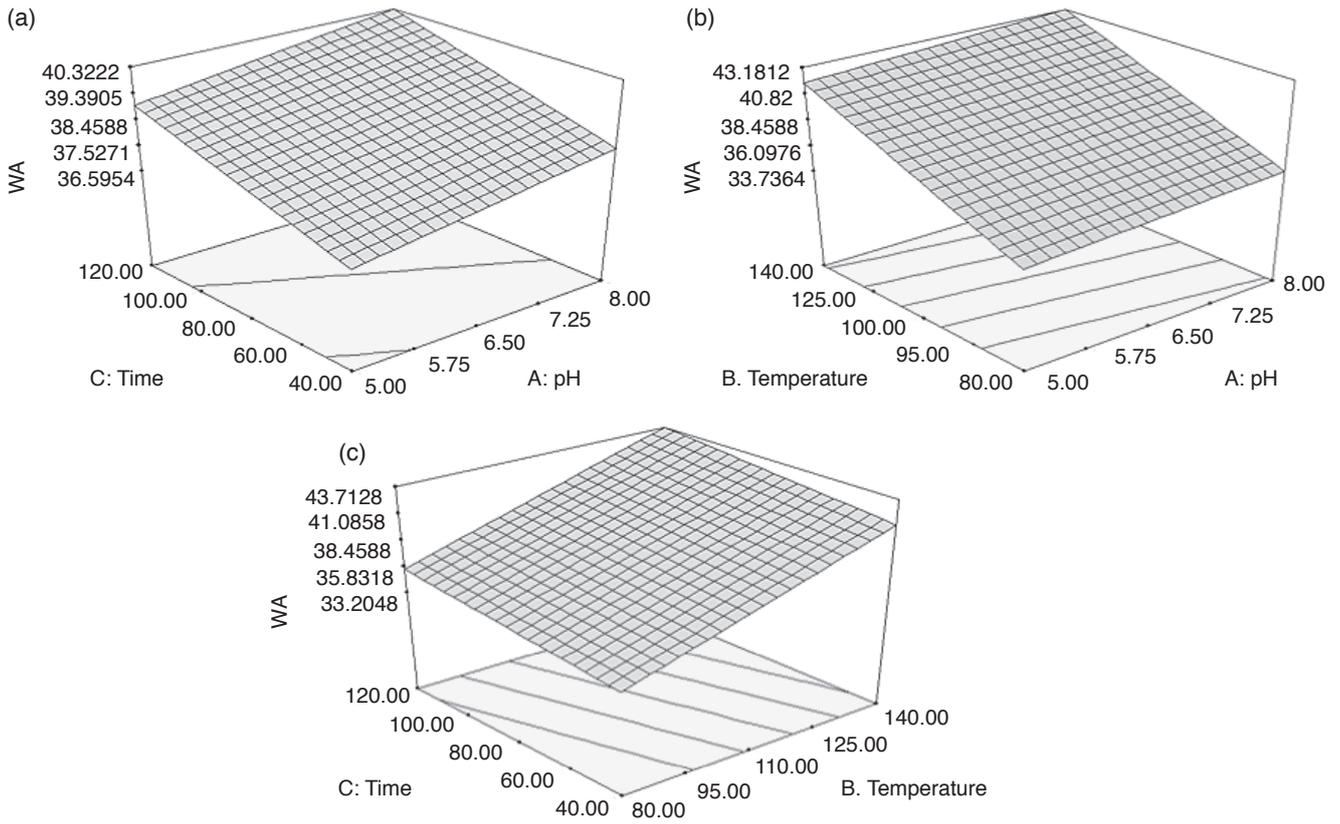


Figure 1. The 3D-surface plots of water absorption (WA) as function of (a) time and pH, (b) temperature and pH and (c) time and temperature.

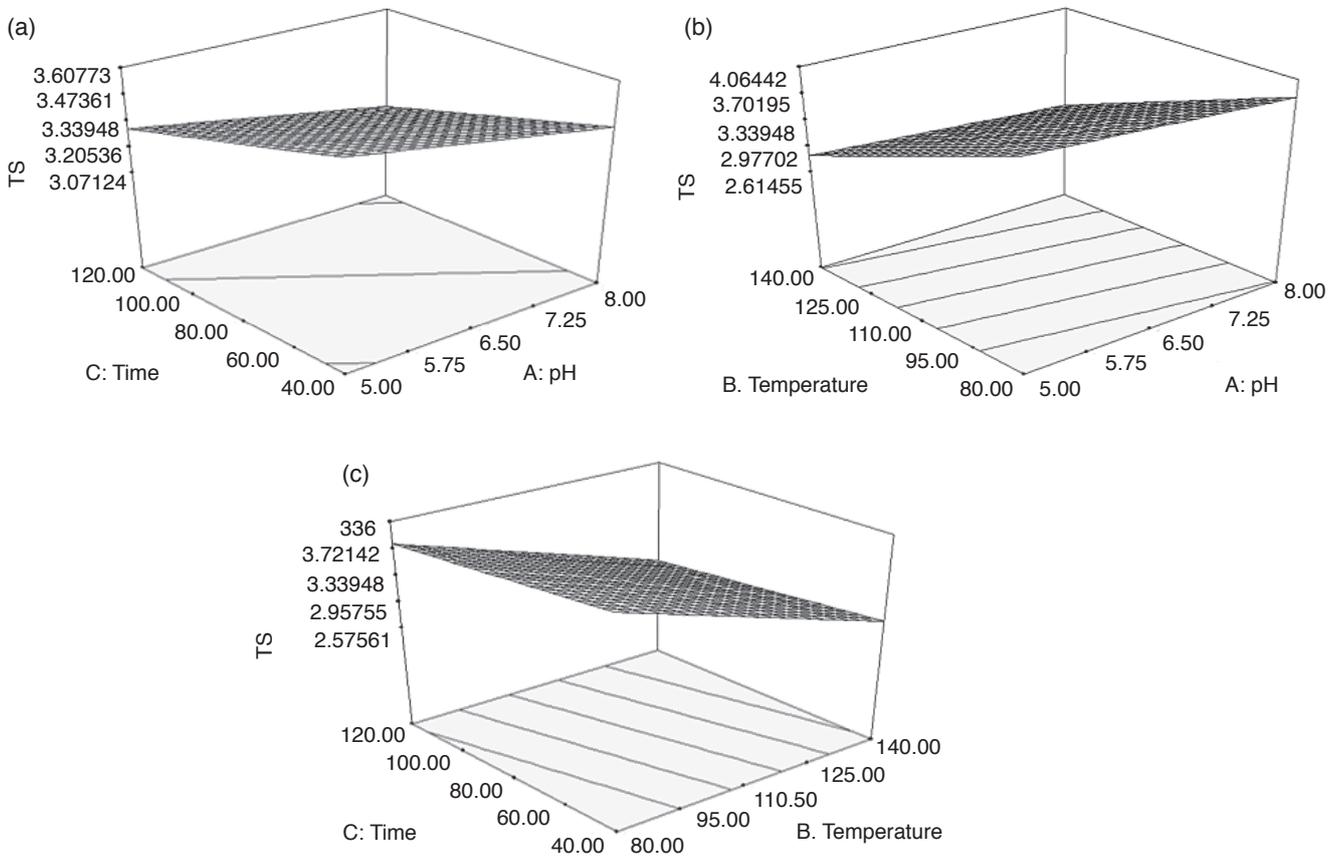


Figure 2. The 3D-surface plots of thickness swelling (TS) as function of (a) time and pH, (b) temperature and pH and (c) time and temperature.

acids are responsible for the degradation of the carbohydrates of wood, especially hemicellulose (Tjeerdsma *et al.*, 1998). Therefore, a higher amount of hemicellulose was removed under acidic condition where the degradation is more severe. However, alkaline hydrolysis was found to be able to remove lignin from the wood. Hence, the removal of hemicellulose together with lignin in alkaline media accounted for a higher loss of wood constituents and may have rendered higher porosity to OPW. Therefore, a higher amount of water was able to be retained within the wood and led to higher WA. Consequently, ensuing swelling was partially extended into the existing voids and this led to a lower swelling of thickness.

Apart from improved dimensional stability, hydrothermal treatment altered the chemical composition in the wood considerably. This alteration could have in turn bestowed several advantageous attributes to the treated OPW, for example, resistance to termites. Hemicellulose, cellulose and lignin compose the woody cell wall. Being the less thermally stable constituent, hemicellulose degrades first during hydrothermal treatment followed by cellulose and lignin (Kamdem *et al.*, 2002). Termites rely on the celluloses derived from wood as their food source. The degradation of carbohydrate during hydrothermal treatment inflicted great loss of favourable food sources for termites and hence, partially prevented OPW from being attacked by termites. Furthermore, the resultant reduction in hygroscopicity of OPW by hydrothermal treatment might be deemed to be another reason that enhance its resistance against termites. Subterranean termites, the most aggressive termite species responsible for 95% of termite damage in Malaysia, require high amounts of moisture to survive and are therefore attracted to wood with a high moisture content. Hydrothermal treatment could reduce the equilibrium moisture content (EMC) of OPW by decreasing the accessibility of the hydroxyl groups in hemicellulose to water. In addition, the formation of less hygroscopic furfural polymer from the hydrolysis of hemicellulose under high temperature has also contributed to the reduction of EMC in wood (Tjeerdsma and Militz, 2005). Consequently, lower wood moisture content could repulse the invasion of termites and confer better termite resistance to the OPW.

## CONCLUSION

Based on the findings, it can be concluded that the dimensional stability of oil palm wood was enhanced by hydrothermal treatment implemented in the present work. The following are the summary

of conclusions according to the presented study:

1. RSM models suggested that the treatment temperature influence both TS and WA of the OPW to a great extent as the dimensional stability improved along with increasing temperature.
2. Treatment time is a less crucial factor in the dimensional improvement of OPW compared to treatment temperature. Better dimensional stability could be observed in the samples treated at higher temperature even when exposed to a shorter time.
3. The pH value of the buffered media also exerted significant influence as hydrothermal treatment conducted in alkaline media resulted in lower TS but higher WA. Removal of lignin in alkaline media needs to be further investigated and studied for better understanding to its influence on the dimensional stability of treated OPW.
4. Apart from that, hydrothermal treatment could have conferred better biological durability to OPW which is worth investigating in future studies.

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