Journal of Tropical Forest Science 31(2): 151–161 (2019)
https://doi.org/10.26525/jtfs2019.31.2.151161

EVALUATION OF BENDING STRENGTH OF HYDROTHERMALLY TREATED OIL PALM WOOD IN VARIOUS BUFFERED MEDIA USING RESPONSE SURFACE METHODOLOGY

Ebadi SE[1, *], Ashaari Z[2], Late Masoumi HRF[3], Soltani M[1], Naji HR[4] & Vaysi R[1]

[1]*Department of Wood and Paper Science and Technology, Chalous Branch, Islamic Azad University, Chalous, 46619-61367 Iran*
[2]*Department of Forest Production, Faculty of Forestry, Universiti Putra Malaysia (UPM), 43400 Serdang, Selangor, Malaysia*
[3]*Department of Biomaterials, Iran polymer and Petrochemical Institute, Tehran, 14977-13115 Iran*
[4]*Department of Forest Sciences, Ilam University, Ilam, 69315-516 Iran*

**ebadi_es@iauc.ac.ir*

Submitted November 2017; accepted January 2019

This study was focused on the effect of hydrothermal treatment in buffer solutions to improve mechanical properties of oil palm wood (OPW). To control the destructive effects of the released acids caused by the degradation of acetyl groups of the hemicelluloses, the hydrothermal treatment in buffered media was conducted using surface methodology (RSM). Central composite design (CCD) is a useful empirical design in RSM. The CCD and RSM were also applied to optimise the hydrothermal treatment variables (buffer solutions, temperature and time) as effective actual variables with 20 experiments. The results showed that the samples treated in neutral media and low temperatures displayed higher modulus of rupture (MOR) (MPa) and modulus of elasticity (MOE) (MPa) due to control of the medium acidity with low degradation of hemicelluloses. Furthermore, the treatment in buffered media significantly affected on mechanical properties of hydrothermally treated OPW. The effect of the temperature on bending strength were more notable than buffer solutions and time. The actual response according to predicted treatment conditions (pH = 7.12, temperature = 110 °C and time = 120 min) explained closest agreement with the predicted value with residual standard error (RSE) of less than 5%.

Keywords: Response surface methodology, central composite design, hydrothermal treatment, buffered media, bending strength, oil palm wood

INTRODUCTION

Oil palm (*Elaeis guineensis*) is a permanent crop commonly grown in the humid tropics. Malaysia is well known for its potential in renewable resources of lignocellulosic materials. Oil palm has an economic life span of 25–30 years (Hartley 1977). Thereafter, replanting is usually associated with large volumes of logs at any economic life span. The huge amount of residual is a big environmental problem. In industrial applications, oil palm wood (OPW) as a raw material has several drawbacks in physicomechanical and durability properties. To solve the problem, some studies were conducted using phenol formaldehyde, bio-resin, oil heat treatment and other chemical materials by impregnation/compregnation process. (Shams & Yano 2004, Wang & Cooper 2005, Bezerra et al. 2008, Erwinsyah 2008, Amarullah et al. 2010, Abdullah et al. 2012, Widiarti et al. 2015, Zaidon et al. 2015, Endo 2016)

On the other hand, there is great interest in using OPW as an alternative for some wood products in structural applications (Saliman et al. 2017). Mechanical properties of modulus of rupture (MOR) and modulus of elasticity (MOE), as common mechanical properties, are important in various applications of wood. The MOR and MOE will also be influenced by species, treatment conditions and methods (Poncsak et al. 2006).

Hydrothermal treatment, an appropriate method for treating wood, is eco-friendly, non-chemical and an effective thermal treatment technique to enhance and improve wood properties (Boonstra et al. 1998, Oltean et al. 2007, Talaei et al. 2010, 2013). The hydrothermal treatment effects the removal of extractives,

© *Forest Research Institute Malaysia*

hemicellulose hydrolysis and change of lignin and cellulose properties (Sandberg & Navi 2007, Gündüz et al. 2009, David & Madison 2010). One of the most important drawback of hydrothermal treatment is the increase of acidity in the treatment medium (Tjeerdsma & Militz 2005, Boonstra et al. 2007a, Talaei & Karimi 2015, Saliman et al. 2017). In addition, during hydrothermal treatment, acetic acid formation caused by acetyl functional groups of the hemicellulose increases the acidity of treatment medium (Tjeerdsma & Militz 2005, Hill 2007, Talaei et al. 2014).

The buffer solution as a medium can be used to solve the drawbacks in wood during hydrothermal treatment (Talaei 2010, Talaei et al. 2013). Furthermore, the buffer solution is an aqueous solution that can control and neutralise the acidity of medium in a specified pH level (Talaei 2010, Talaei et al. 2014). Hence, hydrothermal treatment in buffered media can be considered a hydrothermal modification method, known as an effective technique for controlling the effects of destructive acids through destruction of carbohydrates during treatment (Talaei 2010, Talaei et al. 2014, Talaei & Karimi 2015). Buffered solutions may also offer an alternative treatment media in wood modification industries (Talaei 2010). Furthermore, the heating media also play an important role to transfer heat into the wood under treatment. Hydrothermal treatment of beech wood has been performed in different buffered media using acidic, neutral and alkaline buffered solutions (Talaei 2010, Talaei & Karimi 2015).

The OPW, a new raw material in the field of timber utilisation, is not easy to process and work on. It also has certain characteristics that offers a wide range of problems, rarely encountered in conventional and commercial timber (Loh et al. 2011, Bakar et al. 2013). Reports on the influence of hydrothermal treatment using different buffered solutions on OPW properties are infrequent in literature. Ebadi et al. (2016) reported that the hydrothermal treatment in buffered media at temperature 140 °C for 120 min significantly decreased the mechanical properties of the treated OPW related to the degradation of hemicelluloses. Ebadi et al. (2015) reported that the buffered media at temperature of 140 °C for 120 min significantly affected the equilibrium moisture content (EMC) (%), mass loss (ML%) and water absorption (WA%), however, there were no significant effects on the anti-swelling efficiency (ASE%) and water repellent efficiency (WRE%). It was concluded that the hydrothermal treatment in the buffered medium of weak alkaline had the most significant effect on the physical properties of OPW. Hydrothermal treatment can improve the dimensional stability of treated samples, and treatment temperature being the most influential factor (Talaei 2010, Saliman et al. 2017). In addition, the treated samples in an acidic media displayed lower water absorption. However, lower thickness swelling was observed in the samples treated in alkaline media, probably caused by the removal of lignin that increased the porosity of the OPW.

To maximise the OPW properties by buffered solutions, the response surface methodology with central composite design (CCD) were used as a statistical design for optimisation of hydrothermal treatment process. Central composite design was an identification method to predict more accurate value of the actual response (Myers & Montgomery 2002, Bezerra et al. 2008, Khuri & Mukhopadhyay 2010).

The effects of independent variables on important mechanical properties of OPW, as well as optimisation models for hydrothermal terms, were evaluated in this study using response surface methodology (RSM). The present study was set up as a preliminary study to understand the behavior of treated OPW in buffered media. Therefore, the aim of the study was to determine and evaluate the effect of buffer solutions on the mechanical properties of hydrothermally treated OPW, using RSM method.

MATERIALS AND METHODS

Samples preparation

Three thirty-years-old oil palm trees were randomly harvested at the Agricultural Park, Universiti Putra Malaysia (UPM), Malaysia. The age of the trees was derived from the initiative plan of the University's botanic garden. They were then flatten sawn from the outer part of the logs into dimensions of 60 cm × 5 cm × 5 cm. Sspecimens based on BS 373: 1957 (1986) with dimensions of 300 mm × 20 mm × 20 mm were cut to evaluate the static bending tests including MOR and MOE. To prevent fungal attack and moisture loss, all samples were stored in the cold room at 4 °C.

Response surface methodology and central composite design

The RSM was used to determine the effect of the independent variables on the responses. In the present study, CCD using RSM was applied to investigate the effects of the treatment variables on bending properties (MOR and MOE) of treated OPW. Therefore, the experiments were designed using CCD. The CCD was evaluated based on the influence of independent variables as impressive actual variables in modeling of MOR and MOE, as a function of respective selected variables. The bending properties such as MOR (MPa) and MOE (MPa) were selected as responses and dependent variables.

Design matrix was generated and the results were statistically analysed by Design Expert software 8.04. Design Expert software was used to obtain the effect of treatment that illustrated the response surface model. A 20-runs CCD using RSM was developed and the ranges of variables were identified based on literature review and preliminary tests (Table 1). Each independent variable was coded at five different levels. Experiments were randomly run in order to avoid questionable variability that affect the outcome of the responses according to unnecessary factors. The centre of the experimental field was performed six times for CCD. The design variables were buffer solutions (X_1, pH), temperature (X_2, °C) and time (X_3, mins), while response variables were MOR and MOE. The coded level of variables included low (5) and high (8) range of pH variables, as well as -α and +α as minimum and maximum of CCD levels, determined by design expert software with lower and higher limits than the pH variables.

The experimental terms of coded and actual values developed using RSM with CCD are shown in Table 2. All the points in the design region were at identical distance from the center. The results in distribution of errors amongst all points were in equal manner.

Hydrothermal treatment was performed by heating and impregnating the samples in various buffered media with pH range 5–8 under atmospheric pressure (~ 110 ± 5 °C) in a laboratory digester. The hydrothermal treatment variables (buffered solutions, temperature and time) were designed using CCD and analysed by RSM. For the measurement of mechanical properties (MOR and MOE), 10 green samples with moisture content of 114% were placed into the digester. The samples were hydrothermally treated in buffered media using the suggested buffer solutions (with different pH), treatment temperature and time by CCD. After hydrothermal treatment, the treated samples were removed from the digester and kept in a conditioned room at temperature 20 ± 2 °C with relative humidity of 65 ± 3% to reach moisture content of about 12 ± 2%.

Evaluation of MOR and MOE

The tests of MOR and MOE were performed by Instron universal testing machine according to British-adopted European standard BS EN 373: 1957 (1986). The width and thickness of each specimen was measured at mid length. Loading span and support were used in center loading with a span length of 300 mm. The specimens were 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^{-1}. The span was established in order to maintain a minimum span-to-depth ratio of 14. These properties were calculated according to the following equations:

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

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

Table 1 Experimental range and levels of the variables

Symbol	Impressive actual variable	Coded level of variables				
		-α	Low (-1)	Middle (0)	High (+1)	+α
X_1	Buffer solutions pH	3.98	5.0	6.5	8.0	9.02
X_2	Temperature (°C)	59.55	80.0	110.0	140.0	160.45
X_3	Time (min)	12.73	40.0	80.0	120.0	147.27

Table 2 Central composite design experiments

Run	Type	Coded variable			Actual variable		
		X_1	X_2	X_3	pH	Temperature	Time
1	Factorial	-1	+1	-1	5.00	140.00	40.00
3	Factorial	+1	-1	+1	8.00	80.00	120.00
4	Factorial	+1	+1	+1	8.00	140.00	120.00
9	Factorial	+1	-1	-1	8.00	80.00	40.00
13	Factorial	-1	-1	-1	5.00	80.00	40.00
14	Factorial	-1	+1	+1	5.00	140.00	120.00
15	Factorial	-1	-1	+1	5.00	80.00	120.00
16	Factorial	+1	+1	-1	8.00	140.00	40.00
7	Axial	-1.682	0	0	3.98	110.00	80.00
11	Axial	+1.682	0	0	9.02	110.00	80.00
17	Axial	0	+1.682	0	6.50	160.45	80.00
2	Axial	0	-1.682	0	6.50	59.55	80.00
18	Axial	0	0	-1.682	6.50	110.00	12.73
5	Axial	0	0	+1.682	6.50	110.00	147.27
6	Central	0	0	0	6.50	110.00	80.00
8	Central	0	0	0	6.50	110.00	80.00
10	Central	0	0	0	6.50	110.00	80.00
12	Central	0	0	0	6.50	110.00	80.00
19	Central	0	0	0	6.50	110.00	80.00
20	Central	0	0	0	6.50	110.00	80.00

X_1 = buffer solution pH, X_2 = temperature, X_3 = time

where, MOE is modulus of elasticity (MPa or N mm^{-2}), MOR is modulus of rupture (MPa or N mm^{-2}), P is loading at proportional limit (N), Pm is maximum load and force (N), b is width of specimen (mm), L is span length between specimen support of the span (mm), t is thickness of specimens (mm), and D is deflection of the neutral plane at the proportional limit measured at half span (mm).

Statistical analysis

Analysis of variance (ANOVA) was carried out to determine the significant differences between the independent variables. Statistically significant independent variables ($p < 0.05$) were considered in the reduced model. Multiple regressions were applied in analysing the experimental data to predict the coefficients of the fitted second-order polynomial model.

RESULTS AND DISCUSSION

Analysis of experimental data and prediction of performance

Experimental data and the predicted values for the dependent variables are presented in Table 3. Three different tests i.e. as sequential F-test (or sequential model sum of squares), lack-of-fit and model summary statistics were employed to decide about the adequacy of various models.

Figure 1 indicates the predicted value vs. actual value of the responses. The coefficient of determination (R^2) of MOR and MOE responses were 0.997 and 0.998, respectively. The predicted values were achieved from fitting techniques of model and were sufficiently correlated with the actual values.

Table 3 Central composite design matrix, actual and predicted values of responses

Run	X_1	X_2	X_3	MOR		MOE	
				Actual	Predicted	Actual	Predicted
1	5.00	140.00	40.00	43.44	43.67	6012.18	6009.42
2	6.50	59.55	80.00	41.88	42.00	5680.78	5680.71
3	8.00	80.00	120.00	29.61	29.43	5144.81	5148.48
4	8.00	140.00	120.00	30.55	30.50	4820.12	4819.76
5	6.50	110.00	147.27	43.49	43.46	5876.80	5882.13
6	6.50	110.00	80.00	40.35	40.46	5550.71	5553.42
7	3.98	110.00	80.00	26.36	26.16	5026.01	5021.18
8	6.50	110.00	80.00	26.21	25.90	4685.52	4692.47
9	8.00	80.00	40.00	35.09	35.16	5623.88	5627.36
10	6.50	110.00	80.00	33.50	33.54	5075.70	5074.53
11	9.02	110.00	80.00	44.09	43.80	6078.43	6074.91
12	6.50	110.00	80.00	19.18	19.58	4630.63	4626.98
13	5.00	80.00	40.00	42.36	42.25	5457.54	5457.99
14	5.00	140.00	120.00	37.99	38.20	5249.22	5243.91
15	5.00	80.00	120.00	39.79	39.06	5350.08	5350.95
16	8.00	140.00	40.00	39.36	39.06	5349.67	5350.95
17	6.50	160.45	80.00	38.35	39.06	5349.63	5350.95
18	6.50	110.00	12.73	39.72	39.06	5348.60	5350.95
19	6.50	110.00	80.00	38.55	39.06	5348.62	5350.95
20	6.50	110.00	80.00	38.60	39.06	5359.99	5350.95

X_1 = buffer solution pH, X_2 = temperature, X_3 = time (min), MOR = modulus of rupture, and MOE = modulus of elasticity

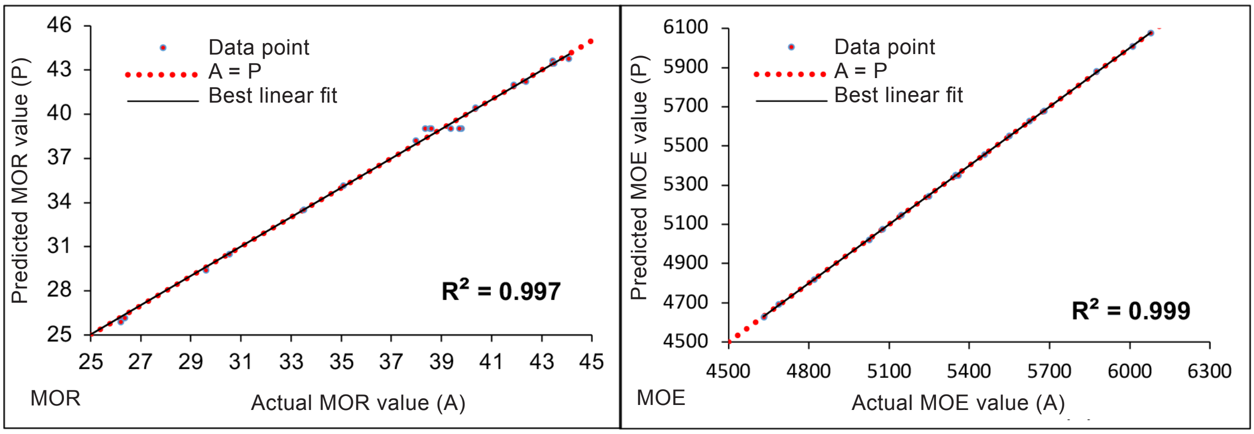


Figure 1 Scatter plot of predicted MOR and MOE value vs. actual MOR and MOE value from CCD

Regression and adequacy of the model

Various factors on MOR and MOE, obtained experimentally based on CCD, are tabulated in Table 3. The predicted values were in agreement with the experimental values in almost all cases. Prediction of MOR and MOE values as response variable was based on experimental data. The data was used to compute the coefficients of the quadratic and linear polynomial equations for MOR and MOE respectively, as shown below:

$$\begin{aligned} Y\,(MOR) = {} & +39.06-0.48X_1-7.20X_2-1.20X_3 \\ & +0.69X_1X_2-0.33X_1X_3-0.76X_2X_3 \\ & -1.67X_1^2-2.61X_2^2+0.41X_3^2 \end{aligned} \quad (3)$$

$$\begin{aligned} Y\,(MOR) = {} & +5350.95-164.36\,X_1-430.47\,X_2 \\ & -63.65\,X_3 \end{aligned} \quad (4)$$

where, X_1 = buffer solution with different pHs, X_2 = temperature, X_3 = time

ANOVA results of the quadratic and linear models for MOR and MOE are tabulated in Table 4. To ensure the fitted model gives an adequate approximation of the results from the experimental terms, the adequacy of the model was evaluated. For any given conditions in the model, high F-value and small p-value would demonstrate more significant effect on the respective response variables. Therefore, the variable with the highest effect on MOR and MOE of treated OPW was temperature, while buffered solutions and treatment time showed lower significant effect. The p-value was smaller than 0.05 which demonstrated that most conditions of the models were still remarkable. Pure errors such as experimental errors were minimal, as the value of lack-of-fitness was insignificant for both responses.

Estimated regression coefficients of standard deviation, the prediction residual error sum of squares (PRESS), R^2, the predicted-R^2, adjusted-R^2 and adequate precession were associated to the effect of independent variables. Table 4 displays regression coefficients for optimisation of process conditions. The fit of the model was evaluated using coefficient of multiple regression (R^2). The adjusted R^2 was used for confirmation of the model adequacy. The R^2 values were 0.997 and 0.998 for the responses of MOR and MOE, respectively. The adequacy of the model was further proved by high adjusted-R^2 of 0.994 and 0.997 for the MOR and MOE, respectively. The analysis showed that MOE had the highest coefficient value, followed by MOR value, and designs fitted well into the quadratic and linear polynomial models, respectively.

Verification of regression model

In order to ensure that the fitted models can provide an adequate approximation to the real system, the normality plot was evaluated. The normal probability plots of the residuals are shown in Figure 2 for examination of the normality plot for mechanical models. The plots indicated that the residuals generally fell on a straight line showing that the errors were distributed normally.

The Pareto charts (Figures 3) display the ranking of the factors in the models. The following equation was used for calculating the percentage effect of each factor:

$$p_i = \left(\frac{\beta_i^2}{\sum \beta_i^2} \right) \times 100 \quad (i \neq 0) \qquad (5)$$

where, β_i = the amount of each factor (X_1 = buffer solution, X_2 = temperature, X_3 = time) and their interactions based on the coefficients of the quadratic and linear polynomial equations (Equation 3 and 4), p_i = the percentage effect of each factor on the treated wood properties.

As demonstrated in Figure 3 (MOR), the terms of temperature (X_2) was significant in the MOR model, while the second importance was related to X_2^2. However, the synergistic (+) and antagonistic (-) signs determined the direction of the conditions' effect on the response. As the model illustrated, the quadratic parameters had synergistic and antagonistic effect on MOR response. As shown in figure 4 (MOE), the temperature factor (X_2) was remarkable in the MOE model, while the second importance belonged to buffer solution (X_1). The synergistic (+) and antagonistic (-) signs determined the

Table 4 Analysis of variance (ANOVA) and regression coefficients for response surface quadratic and linear models of MOR and MOE of treated OPW

Responses	ANOVA and regression coefficients of responses													
	Model		Residual											
			LOF		PE									
	F- value	p-value	F-value	p-value	SS	MS	R^2	Adjusted R^2	Predicted R^2	Adequate precision	SD	CV%	PRESS	Mean
MOR[q]	375.38	< 0.0001*	0.27	0.908**	2.03	0.41	0.997	0.994	0.992	67.25	0.51	1.40	7.22	36.42
MOE[l]	53800	< 0.0001*	0.92	0.579**	96.68	19.34	0.998	0.997	0.996	756.68	4.28	0.08	470.70	5351

* = significant; ** = not significant, q = quadratic model, l = linear model, LOF = lack of fitness; PE = pure error, SS = sum of square, MS = mean square, R = regression, SD = standard deviation, CV = coefficient of variation, PRESS = prediction residual error sum of squares

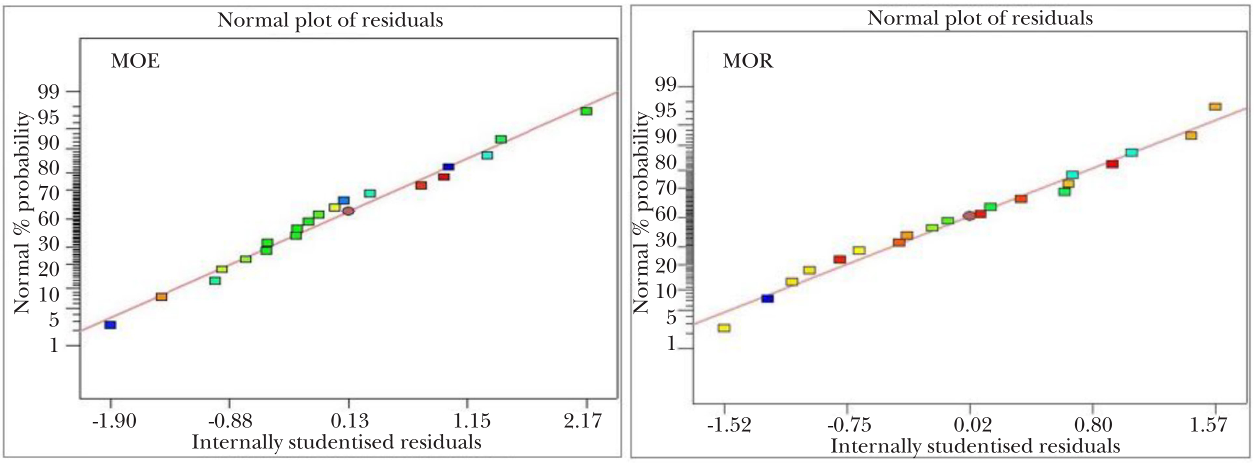
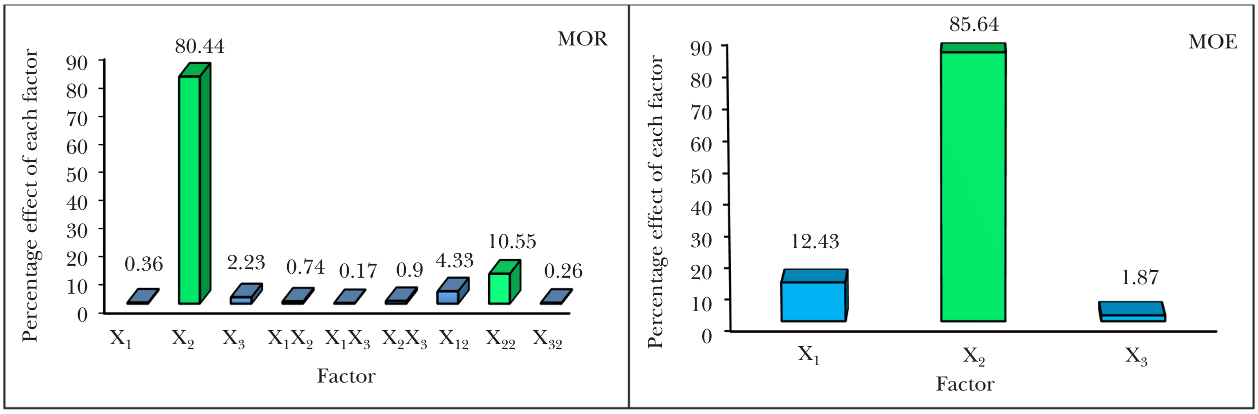


Figure 2 Normal probability of internally studentised residuals for MOR and MOE



X_1 = buffer solutions with different pHs, X_2 = temperature, X_3 = time; other terms such as X_1X_2, X_1X_3 and X_2X_3 represent the interaction between the relevant parameters; square terms of parameters such as X_1^2, X_2^2, and X_3^2 are the same parameters in the square form

Figure 3 Pareto chart represents the effect of independent variables and their interactions on response of MOR and MOE

direction of the term's effect on the response. Therefore, the linear parameters had synergistic and antagonistic effect on the MOE response.

Response surface analysis

The effects of independent variables on the response of MOR and MOE of the hydrothermally treated OPW are displayed in Figures $4a_{1,\,2}$ and $4b_{1,\,2}$. Figure $4a_1$ shows the effect of temperature and the buffer solution pH on MOR and MOE, while time is fixed at center point level. The plot $4a_1$ shows that the buffer solution pH in higher and lower temperature does not significantly affect MOR of treated OPW. The higher the temperature and time, the greater the MOR. On the other hand, maximum MOR was observed at minimum temperature and neutral buffer. Minimum MOR was also observed in the alkaline and acidic buffer at temperature 140 °C. As shown in figure $4a_2$, the MOE increased with decreasing temperature from 140 °C to 80 °C and the acidification of medium pH. However, the effect of temperature was higher than the buffer solutions on MOE.

Figure $4b_1$ illustrates the effect of treatment time and buffer solution pH on MOR when temperature was fixed at center point level. As in Figure $4b_1$, the best conditions of MOR was in less treatment time and neutral medium. MOE in plot $4b_2$, for interaction between treatment time and buffer solution pH, was carried out with temperature fixed at center point level. The MOE decreased with increasing treatment time and

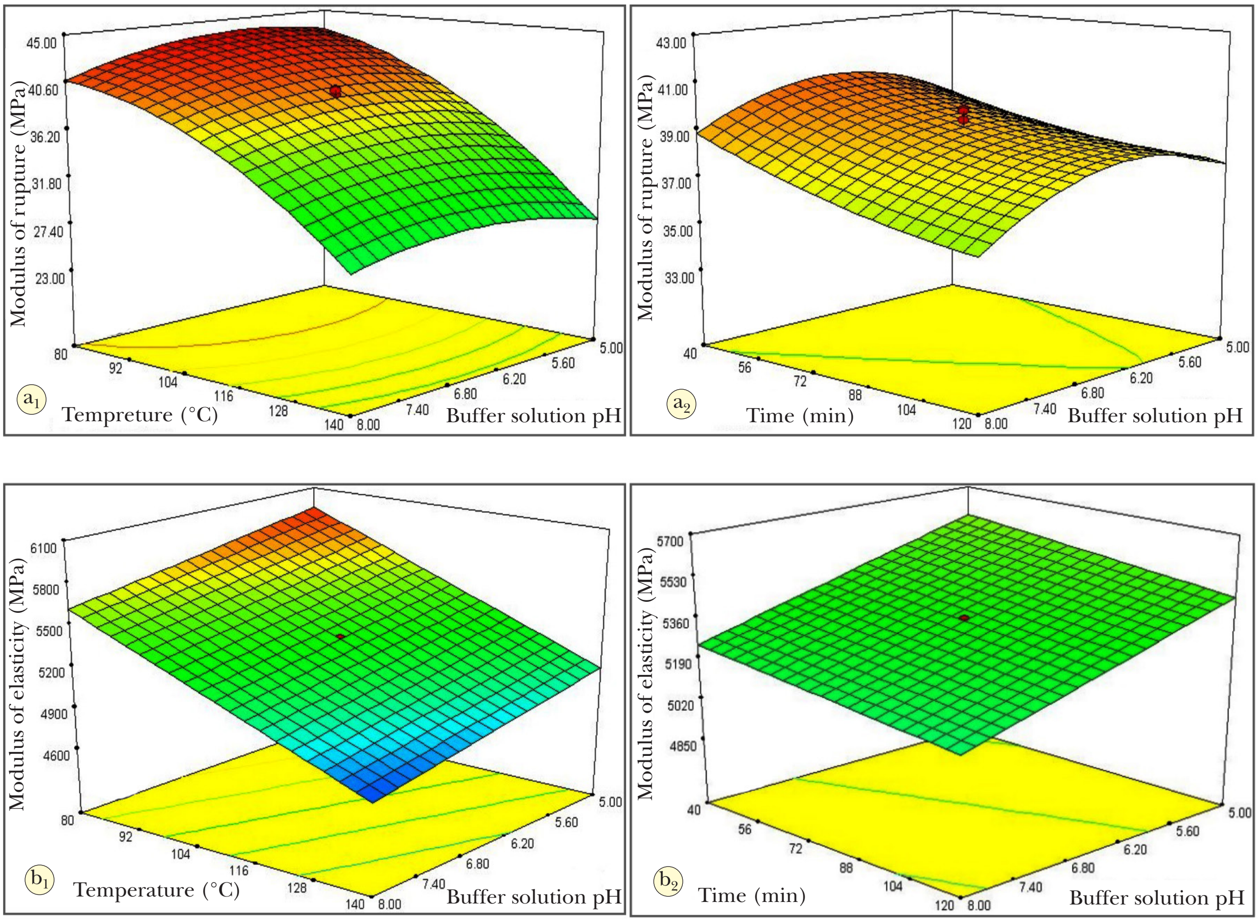


Figure 4 Response surface plot of temperature vs. buffer solution (X_2X_1) ($a_{1,2}$) and time vs. buffer solution (X_3X_1) ($b_{1,2}$) on MOR and MOE

alkalinisation of treatment medium. Therefore, it could be concluded that treatment temperature is a more influential factor than treatment time.

On the other hand, the pH of the heating media showed some changes when treatment temperature and time remained constant. At the same level of temperature and time, samples treated in neutral media (pH ~ 6.5–7) displayed better MOE compared to samples treated in acidic and alkaline media. In addition, treated OPW in acidic media (pH ~ 5) represented higher MOR compared to samples treated in neutral and alkaline media.

The results were comparable to the findings of other researchers on OPW with different methods (Ebadi et al. 2015, Poncsak et al. 2006). The effect of temperature on MOR and MOE is dependent on the species. The MOR and MOE are important factors for measuring mechanical properties of wood (Santos 2000, Sandberg & Navi 200). Viitaniemi and Jämsä (1996) stated that decrease of strength depends on the treatment process. The created micro cracks in cell walls of treated wood during heat treatment is one of the main reasons that decrease mechanical properties of thermally treated wood (Oltean et al. 2007).

Kim et al. (1998) reported that a close relationship exists between the reduction of bending strength (MOR and MOE) and treatment conditions (temperature and time). The highest reduction of MOR was observed in treated samples in alkaline and acidic media, and the lowest reduction was observed in neutral buffer, due to less degradation of carbohydrates. The reduction of MOR increasingly accelerated with increasing treatment temperature. The temperature rise and polysaccharides degradation are accompanied by formation of acetic acid, formic acid and furfural (Boonstra et al. 1998, Sandberg & Navi 2007). The degradation of hemicellulose is a major factor for decreasing mechanical strength, especially on bending and tensile strength (Garrote et al. 1999, Bezerra et al. 2008). In addition, Winandy and Lebow (2007) noted that a close relationship exists between the amount of hemicellulose and bending strength. There is a direct relationship between the increase of weight loss and decline of mechanical strength of wood (Kollmann & Cote 1968, Gündüz et al. 2009). Buffering ability of the solutions decreased after relatively severe thermal treatment (> 140 °C) due to acidification of medium and release of larger values of organic acids (Ebadi et al. 2015).

Yildiz et al. (2003 & 2002) noted that MOR and MOE of treated specimens reduced with increasing temperature and treatment time. The released acids during hydrothermal process led to pH reduction, deacetylation of hemicellulose, mass loss and consequent reduction of mechanical strength (Garrote et al. 1999, Sundqvist et al. 2006). Poncsak et al. (2006) stated that MOR and MOE reduced with increasing treatment temperature. Increasing acidity of treatment medium during thermal treatment under wet conditions is due to the formation of weak organic acids such as acetic and formic acids, caused by the decomposition of acetyl functional groups of hemicellulose during acidic hydrolysis process (Kubojima & Ohta 2000, Sulaiman et al. 2012). Phuong et al. (2007) reported that the main factor affecting brittleness in treated wood is the degradation of amorphous polysaccharides during treatment. Higher MOE can lead to lower elasticity and higher brittleness as well. In addition, treatment in acidic medium degrades starch, hemicellulose and other extractives, which causes brittleness (Kim et al. 1998).

The highest degree of de-acetylation occurs in acidic medium, probably due to acidification of treatment medium during the process. Therefore, mechanical properties decrease with the release of organic acids during thermal treatment (Sandberg & Navi 2007, Talaei 2010, Widiarti et al. 2015). The role of buffer solution pH is to prevent the reduction of mechanical strength. The decrease in mechanical strength is justified according to increasing weight loss of treated specimens in acidic buffer compared to buffer of weak alkaline and neutral (Talaei et al. 2014). Additionally, hydrothermal treatment in buffered solutions is an effective method in controlling destructive effects of acids formed through carbohydrate destruction during treatment (Talaei & Karimi 2012b). The decrease of mechanical properties in treated wood is lessened by neutralisation of released acids by buffered solutions. In addition, buffering of treatment medium at weak alkaline and neutral levels via neutralisation of medium's acidic pH can effectively control the negative influences on the strength of treated wood (Talaei 2010, Talaei & Karimi 2012b). It appears that the buffer solution, in range of neutral pH and relatively low temperature, could control the released acids resulting from destruction of hemicellulose during hydrothermal treatment, and probably could avoid slight degradation of lignin (Merakeb et al. 2009).

Optimisation by response surface methodology and model validation

The samples were tested and analysed to validate predicted optimum conditions. Validated models can be predicted based on optimal factors such as buffered solutions, treatment temperature and time of hydrothermal treatment. For this purpose, RSM by a CCD was adopted to identify optimal conditions. The samples were treated on suggested conditions and the results of responses (MOR and MOE) were compared with the predicted values (Table 5). According to the results, the model validity was confirmed and the experimental (actual) values were then determined, close to the predicted values, with residual standard error (RSE) of less than 5%. In addition, the experiential model obtained from experimental design of RSM can be used to describe the sufficient relationship between the independent variables and responses.

Therefore, the buffer solution with pH 7.12, rather low temperature (110 °C) and time 120 mins would be the optimum condition for the treatment of OPW.

CONCLUSION

It can be concluded that the bending strength of OPW were partly enhanced by hydrothermal treatment in buffered media. A summary of the most important findings are as follows:

- The neutral buffer solution in relatively low temperature can control the destructive effects of the released acids resulting from acidic hydrolysis during hydrothermal treatment by the neutralisation of medium acidity.
- Treatment temperature is a more important factor in the bending strength improvement of OPW compared to treatment time.

Table 5 Predicted and observed response values for optimal conditions

Model	Independent variable			MOR		RSE (%)	MOE		RSE (%)
	X_1	X_2	X_3	Actual	Predicted		Actual	Predicted	
	7.12	110	120	39.09	38.98	0.0028	5613	5647	0.006

X_1 = buffer solution pH, X_2 = temperature (°C), X_3 = time (min), RSE = residual standard error

- R^2 correlation coefficients for the quadratic and linear models in MOR and MOE were satisfactory as 0.997 and 0.998, respectively.
- The experimental values agreed with the predicted results indicating suitability of the models, showing that RSM experimental model can be adequately used to describe the relationship between variables and response in buffer solutions.
- In particular, the derived model could be used to optimise the hydrothermal treatment conditions and improve mechanical properties.

ACKNOWLEDGMENT

This research was supported by Universiti Putra Malaysia. The authors would like to thank the Institute of Tropical Forestry and Forest Products (INTROP) for providing the research grant.

REFERENCES

ABDULLAH CK, JAWAID M, ABDUL KHALIL HPS, ZAIDON A & HADIYANE A. 2012. Oil palm trunk polymer composite: morphology, water absorption and thickness swelling behaviours. *Composite of Palm and Polymer Bioresources* 7: 2948–2948.

AMARULLAH M, BAKAR ES, ZAIDON A, HAMAMI SM & FEBRIANTO F. 2010. Reduction of formaldehyde emission from phenol formaldehyde treated oil palm wood through improvement of resin curing state. *Journal of Tropical Wood Science and Technology* 8: 9–14.

BAKAR ES, TAHIR PM, SAHRI MH, MOHD NOOR MS & ZULKIFLI FF. 2013. Properties of resin impregnated oil palm wood (Elaeis Guineensis Jacq). *Pertanika Pertanika Journal of Tropical Agricultural Science* 36: 93–100.

BEZERRA MA, SANTELLI RE, OLIVEIRA EP, VILLAR LS & ESCALEIRA LA. 2008. Response surface methodology (RSM) as a tool for optimisation in analytical chemistry. *Talanta* 76: 965–77.

BOONSTRA MJ, VAN ACKER J, TJEERDSMA BF & KEGEL EV. 2007a. Strength properties of thermally modified softwoods and its relation to polymeric structural wood constituents. *Annals of Forest Science* 64: 679–690.

BOONSTRA MJ, TJEERDSMA BF & GROENEVELD HAC. 1998. *Thermal Modification* of *Non-Durable Wood Species. 1. The Plato Technology: Thermal Modification of Wood.* Document No. IRG/WP 98. International Research Group on Wood Preservation, Stockholm.

BS 373: 1957 (1986) *Standard Methods of Testing Small Clear Specimens of Timber.* British Standards Institution, London.

DAVID EK & MADISON W. 2010. *Handbook Wood as an Engineering Material.* Chapter 5. Forest Products Laboratory, Forest Service, U.S. Department of Agriculture, Madison.

EBADI SE, ASHAARI Z, NAJI HR, JAWAID M, SOLTANI M & SAN HP. 2016. Mechanical behavior of hydrothermally treated oil palm wood in different buffered media. *Wood and Fiber Science* 48: 193–201.

EBADI SE, KARIMI A, CHOO AC, ASHAARI Z, NAJI HR, SOLTANI M & RIDZUAN SMA. 2015. Physical behavior of hydrothermally treated oil palm wood in different buffered pH media. *BioResources* 10: 5317–5329.

ENDO K, OBATAYA E, ZENIYA N & MATSUO M. 2016. Effects of heating humidity on the physical properties of hydrothermally treated spruce wood. *Wood Science and Technology* 50: 1161–1179.

ERWINSYAH V. 2008. Improvement of oil palm trunk properties using bioresin. PhD thesis. Faculty of Environmental Sciences, Technische Universitat Dresden, Dresden.

GARROTE G, DOMINGUEZ H & PARAJO JC. 1999. Hydrothermal processing of lignocellulosic materials. *European Journal of Wood and Wood Products, HolzalsRoh-und Werkstof* 57: 191–202.

GÜNDÜZ G, AYDEMIR D & KARAKAS G. 2009. The effects of thermal treatment on the mechanical properties of wild pear (pyrus elaeagnifolia pall.) wood and changes in physical properties. *Materials and Design* 30: 4391–4395.

HARTLEY CWS. 1977. *The Oil Palm (Elaeis Guineensis Jacq).* Third Edition. Longmans, Green and Co. Ltd., London.

HILL CAS. 2007. *Wood Modification: Chemical, Thermal and Other Processes.* Volume 5. John Wiley & Sons, Chichester.

KHURI AI & MUKHOPADHYAY S. 2010. Response surface methodology. *Wiley Interdisciplinary Reviews: Computational Statistics* 2: 128–149.

KIM G, YUN K & KIM J. 1998. Effect of heat treatment on the decay resistance and the bending properties of radiata pine sapwood. *Material und Organism* 32: 101–108.

KOLLMANN F & COTE W. 1968. *Principales of Wood Science and Technology.* Springer Verlag, New York.

KUBOJIMA Y & OHTA M. 2000. Bending strength and toughness of heat treated wood. *Journal of Wood and Fiber Science* 46: 8–15.

LOH YF, PARIDAH MT, HOONG YB & CHOO ACY. 2011. Effects of treatment with low molecular phenol formaldehyde resin on the surface characteristics of oil palm (*Elaeis quineensis*) stem veneer. *Materials and Design* 32: 2277–2283.

Merakeb S, Dubois F & Petit C. 2009. Modeling sorption hysteresis in hygroscopic materials. *Comptes Rendus Mecanique* 337: 34–39.

Myers RH & Montgomery DC. 2002. *Response Surface Methodology: Process and Product Optimisation Using Designed Experiments*. 2nd Edition. Wiley, New York.

Phuong L, Shida S & Saito Y. 2007. Effects of heat treatment on brittleness of *Styrax tonkinensis* wood. *Journal of Wood Science* 53: 181–186.

Poncsak S, Kocaefe D, Bouazara M & Pichette A. 2006. Effect of high temperature treatment on the mechanical properties of birch (*Betula papyrifera*). *Wood Science Technology* 40: 647–663.

Oltean L, Teischinger A & Hansmann C. 2007. Influence of temperature on cracking and mechanical properties of wood during wood drying—a review. *BioResources* 2: 789–811.

Sandberg D & Navi P. 2007. I*ntroduction to Thermo-Hydro-Mechanical (THM) Wood Processing*. Report No. 30. School of Technology and Design, Sweden.

Saliman MAR, Ashaari Z, Bakar ES, Hua LS, Tahir PM, Halip JA & Leemon NF. 2017. Hydrothermal treatment of oil palm wood: effect of treatment variables on dimensional stability using response surface methodology. *Journal of Oil Palm Research* 29: 130–135.

Santos JA. 2000. Mechanical behaviour of Eucalyptus wood modified by heat. *Wood Science and Technology* 34: 39–43.

Shams M & Yano H. 2004. Compressive deformation of wood impregnated with low molecular weight phenol formaldehyde (PF) resin II: effects of processing parameters. *Journal of Wood Science* 50: 343–350.

Sulaiman O, Fahmi AW M, Hashim R & Mondal MIH. 2012. The effect of relative humidity on the physical and mechanical properties of oil palm trunk and rubberwood. *Cellulose Chemistry and Technology* 46: 401–407.

Sundqvist B, Karlsson O & Westermark U. 2006. Determination of formic-acid and acetic acid concentrations formed during hydrother maltreatment of birch wood and its relation to colour, strength and hardness. *Wood Science and Technology* 40: 549–561.

Talaei A & Karimi A. 2015. Compression strength, hardness and shear strength of heat treated beech (*Fagus orientalis*) wood in buffered media. *International Journal of Mechanical And Production Engineering* 3: 2320–2092.

Talaei A, Karimi A & Paridah MT. 2014. *Heat Treatment of Wood: Methods and Properties*. Universiti Putra Malaysia, Serdang.

Talaei A, Karimi A & Thevenon MF. 2013. Influence of heat treatment medium on fungal resistance of beech wood. Paper presented at the International Research Group on Wood Preservation, IRG44 Conference, Stockholm.

Talaei A & Karimi A. 2012a. Chemical analysis of hydrothermally treated beech wood in buffered media. Paper presented at the IRG43 Conference, Kuala Lumpur.

Talaei A & Karimi A. 2012b. Mechanical properties of hydrothermally treated beech wood in buffered media. Paper presented at the IRG43 Conference, Kuala Lumpur.

Talaei A. 2010. Effects of hydrothermal treatment in buffered medium on natural durability and mechanical properties of beech wood. PhD thesis, University of Tehran, Iran.

Tjeerdsma BF & Militz H. 2005. Chemical changes in hydrothermal treated wood: FTIR analysis of combined hydrothermal and dry heat-treated wood. *Holz als Roh- und Werkstoff* 63: 102–111.

Viitaniemi P & Jämsä S. 1996. Modification of wood with heat treatment Espoo, Finland. *VTT Building Technology* 1: 1–7.

Wang JY & Cooper PA. 2005. Effect of oil type, temperature and time on moisture properties of hot oil-treated wood. *Holz als Roh-und Werkstoff* 63: 417–422.

Widiarti L, Gea S & Wirjosentono B. 2015. Mechanical properties of oil palm trunk by reactive compregnation methode with dammar resin. *International Journal of PharmTech Research* 8: 74–79.

Winandy JE & Lebow PK. 2007. Modeling strength loss in wood by chemical composition. Part I. An individual component model for southern pine. *Wood Fiber Science* 33: 239–254.

Yildiz U, Gercek Z, Yildize S et al. 2003. The effects of heat treatment on the specific gravity of beech and spruce wood. Paper presented at the IRG Annual Meeting, Australia.

Yildiz S, Çolako lu G, Yildiz ÜC, Gezer DE & Temiz A. 2002. *Effects of Heat Treatment on Modulus of Elasticity of Beech Wood*. The International Research Group on Wood Preservation, Stockholm.

Zaidon A, Lee SH, Mustamel MN & Salleh NFM. 2015. Strength improvement of jelutong (*Dyera costulata*) wood via phenolic resin treatments. *Journal of the Indian Academy of Wood Science* 12: 132–136.