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RESPONSE SURFACE METHODOLOGY MODEL OF HYDROTHERMAL TREATMENT PARAMETERS ON DECAY RESISTANCE OF OIL PALM WOOD

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Effect of hydrothermal treatment on oil palm wood (*Elaeis guineensis*) in term of decay resistance against white rot fungus (*Trametes versicolor*) and equilibrium moisture content (EMC) was investigated in the present study. Response surface methodology (RSM) models for treatment temperature, treatment duration and buffered medium of hydrothermally treated oil palm wood were developed. Oil palm wood were immersed in the buffered media ranging from pH 4–9 and heated, 60–160 °C for 13–147 min, respectively. After treatment, weight loss and EMC of the treated samples caused by white rot fungus were evaluated. Equations with high adjusted r^2 value were obtained using the RSM model. The results revealed that the weight loss of the treated oil palm wood caused by white rot fungus reduced as the treatment temperature and time increased. Buffered medium showed insignificant effect on improvement of decay resistance of the oil palm wood. However, slightly better decay resistance was found in the samples treated in neutral and alkaline medium. On the other hand, EMC of the treated samples reduced with increasing temperature, and the reduction was prominently higher in acidic medium.

Keywords: Buffered medium, central composite design, *Elaeis guineensis*, *Trametes versicolor*, white rot fungus

INTRODUCTION

Wood from matured oil palm (*Elaeis guineensis*) trunks (OPT) or oil palm wood (OPW) is an ideal alternative material to substitute timber that is now experiencing depletion. As the world second largest producer of palm oil, there are 5.64 million ha of oil palm plantations in Malaysia, which accounts for 17% of the total land area of Malaysia (MPOB 2015). Oil palm trees are being replanted at the end of their economic lifespan, which is 25–30 years, generating an estimation of 7 million metric tons of felled OPT annually (Bakar et al. 2008). As a biomass waste, full utilisation of these felled OPT could be beneficial from environmental and socio-economical aspects. Nevertheless, OPW is very susceptible to biological deterioration agents such as termites and fungi, despite having better radial and tangential stability than rubberwood. The biological durability of OPW is very low, classified as class 5 or perishable (Bakar et al. 2013). Therefore, treatment is needed to improve its biological durability. Heat treatment is highly recommended as it is eco-friendly and can serve as an alternative to chemical treatment (Choowang & Hiziroglu 2015, Poonia & Tripathi 2016).

Better fungal decay resistance was reported in heat-treated wood (Esteves et al. 2014). Umar et al. (2016) heated rubberwood in hot palm oil ranging from 172 to 228 °C and observed an improvement in decay resistance against white rot fungus. Oil is used as a heating medium because of its good heat transfer capacity that can readily and evenly transfer heat into wood samples. Hydrothermal treatment using water as heating medium also serves the same purpose. Hydrothermal treatment is well known as a cost effective and efficient method of wood modification. Chen et al. (2013) treated Moso bamboo in a two-step heat treatment, i.e. hydrothermal treatment in different aqueous solutions followed by heating in a kiln, and found that the resistance of the treated sample improved against mould. Endo et al. (2016)

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hydrothermally treated Sitka spruce wood in an autoclave at 120 °C, and observed that the hygroscopicity and equilibrium moisture content (EMC) of the treated wood reduced. The reduction of hygroscopicity reduces water retention in treated wood and subsequently enhances its fungal resistance, compared to the untreated wood (Kamdem et al. 1999).

However, the formation of formic and acetic acid during thermal treatment under moist conditions increase wood acidity (Tjeerdsma et al. 1998). Increase in wood acidity invariably lead to reduction in strength properties of the wood (Kamdem et al. 2002). Therefore, to nullify the effect of the acetic acid, Talaei and Karimi (2012a) conducted hydrothermal treatment of wood in alkaline and neutral buffered media to ameliorate the thermal induced degradation in wood. Beech wood was treated in three different buffered media (pH 5, 7 and 8), and revealed that the hydrothermal treatment in neutral and alkaline medium did not significantly change the mechanical properties of the wood. Information on hydrothermal treatment of oil palm wood in different buffered media for decay resistance properties is scarce. Moreover, the effectiveness of hydrothermal treatment is highly dependent on treatment temperature and time. Therefore, in this study, a response surface methodology (RSM) was used to analyse the effects of several independent variables on the decay resistance and EMC of the OPW. To optimise the processing variables, RSM model for independent variables, namely treatment temperature (°C), time (min) and buffered medium (pH), of hydrothermally treated OPW was developed.

MATERIALS AND METHODS

Response surface methodology (RSM) and central composite design (CCD)

Central composite design using RSM was used in the present study to investigate the effects of treatment variables on decay resistance and EMC of oil palm samples. Three independent variables, namely, buffer media (pH), treatment temperature (°C) and treatment time (min) were selected, and the response variables were decay resistance against *Trametes versicolor* and EMC. 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 at five different levels (Table 1), where the pH, treatment temperature and treatment times ranged from 4 to 9 and 60 to 160 °C, and 13 to 147 min, respectively. Experimental conditions of coded and actual values, developed using RSM with central composite design, are shown in Table 2.

Material preparation and hydrothermal treatment schedule

Oil palm trees with 25 years of age were harvested at University Agriculture Park, Universiti Putra Malaysia (UPM). The harvested oil palm trees were sent to Forest Research Institute Malaysia (FRIM), Kepong, to be cut into samples of 60 cm × 5 cm × 5 cm (length × width × thickness). Cut samples with a density range of 650–750 kg m^{-3} were selected for experiment implementation. The selected samples were stored in a cold room at 4 °C to retain moisture and prevent fungi attack. The samples were assigned to 20 groups of 20 runs, for treatment at various buffered medium, temperatures and durations (Table 2). Hydrothermal treatment was carried out by boiling the samples in different buffered media ranging from pH 4 to 9 above atmospheric pressure by using twin digesters (GIST Co. Ltd.). After loading the samples into the twin digesters, buffer solution was added and twin digesters were tightly closed to prevent the influence of external pressure. Buffer solution was subsequently heated to the required temperature ranging

Table 1 Central composite design (CCD) using respose surface methodology (RSM), range and levels of variables

Factor	Variable	Units	Range and level of actual and coded values				
			$-\alpha$	-1	0	1	α
X1	pH	-	4	5	6.5	8	9
X2	Temperature	°C	60	80	110	140	160
X3	Time	minutes	13	40	80	120	147

Table 2 Experimental design using central composite design

Run	Coded factor			Actual factor		
	X_1	X_2	X_3	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

from 60–160 °C, as suggested by RSM model. During the boiling process, pressure inside the digester is maintained to keep the buffer solution in a liquid state (Wongprot et al. 2013). At the end of the required treatment duration (13–147 min), steam pressure inside the digester was released slowly until it reached atmospheric pressure, before the digester cover was opened, to prevent explosion due to high pressure. The treated samples were removed from the digester and cut into a required size, prior to fungal decay test.

Fungal decay test

The hydrothermally treated oil palm wood was tested in the laboratory for decay resistance against white rot fungus, *T. versicolor*, following the standard method, Wood Preservatives: Test Method for Determining Protective Effectiveness Against Wood Destroying *Basidiomycetes*—Determination of the Toxic Values (BS EN 113: 1997). Treated oil palm wood samples were autoclaved at 121 °C at 15 psi for 15 min prior to the test. The autoclaved samples were then placed in an oven at 103 ± 2 °C for 18 h. The samples were cooled to room temperature in a desiccator and weighed to determine the initial dry mass (W_i). Malt agar medium were prepared using 40 g malt extract powder, 20 g agar and water to make up to 1000 ml. The mixture was dissolved by warming in a boiling water bath. The mixture was then poured in a kolle flask and sterilised in an autoclave at 121 °C for 20 min. After cooling, *T. versicolor* was inoculated into the kolle flask and the oil palm samples were placed into the kolle flask, once the mycelium completely covered the surface of the culture medium. For each run, five replicates of treated oil palm samples with dimensions 50 mm × 25 mm × 15 mm were placed into the malt agar medium-contained kolle flask for fungi exposure. After introducing the test samples, kolle flask was stored in a humidity chamber for 16 weeks at temperature of 22 ± 2 °C and relative humidity 70 ± 5%. At the end of the testing period, the samples were collected and the adhering mycelium removed. The cleaned samples were dried in an oven at 103 ± 2 °C until constant mass, and weighed to determine its final dry mass (W_o). Weight of the test samples, before and after the test, was recorded in order to calculate the percentage of weight loss by decay (WL) based on equation (1):

$$WL\ (\%) = [(W_i - W_o)/W_i] \times 100 \qquad (1)$$

where, WL = weight loss (%), W_i = initial dry mass before exposes to fungus (g) and W_o = final dry mass after exposes to fungus (g). The EMC of the treated oil palm wood was determined using equation (2) after reconditioning at 20 ± 2 °C and 65 ± 5% relative humidity in a conditioning room:

$$EMC\ (\%) = [(M_2 - M_1)/M_1] \times 100 \qquad (2)$$

where, M_1 = oven dried weight (g) and M_2 = constant weight after reconditioning (g).

Data analysis

The results of the experimental design were analysed and interpreted using Design Expert Software (Stat Ease version 6), and the responses of the 3D plots were built.

RESULTS AND DISCUSSION

Regression and adequacy of the model

The RSM model for weight loss by *T. versicolor* was developed and a linear equation was obtained. The final equation in terms of actual factors are shown as follow:

$$WL = 58.09 - 0.60\ pH - 0.26\ tempreture - 0.05\ time \qquad (3)$$

where WL = weight loss. For EMC, a quadratic equation in terms of actual factors was obtained, as shown in equation (4):

$$EMC = 1.74 + 0.52\ pH + 0.14\ tempreture - 0.001 tempreture^2 \qquad (4)$$

where EMC = equilibrium moisture content. Assessment was conducted on the fitted models to confirm sufficient approximation of the results obtained in the experimental conditions. Adequacy of the models were confirmed by the insignificant lack-of-fit value of 0.1203 and 0.50 for weight loss and EMC, respectively, indicating that the models were statistically appropriate for further investigation. The fitness of the models was evaluated using coefficient of multiple regression (r^2), and adjusted r^2 was used for confirmation of the model adequacy. Based on the analysis, a r^2 value of 0.9383 was obtained for the linear model, indicating high fitness. The high, adjusted r^2 value of 0.9628 further proved the adequacy of the model. For the quadratic model of EMC, high r^2 value of 0.8489 and adjusted r^2 value of 0.7129 showed fitness and adequacy of the model. Therefore, these developed models are highly accurate in determining the relationship between response and variables.

Effects of the hydrothermal treatment on decay resistance and EMC of samples

One of the most prominent effect of thermal treatment is the darkening of the treated samples, as shown in Figure 1. The effects of variables on the response of weight loss are shown in Figure 2. The weight loss of OPW reduced significantly with increased treatment temperature and time. The weight loss of OPW also reduced when pH of buffered medium increased from 4 to 9. Figure 3 shows the effects of variables on EMC of the treated samples, which was significantly affected by treatment temperature and pH, while treatment time exerted insignificant effect. The weight loss of the hydrothermally treated OPW, caused by white rot fungus, ranged from 9.00% to 37.75%. The lowest weight loss was observed when OPW was treated at highest temperature, 160 °C for 80 min in buffer 6.5. Meanwhile, the highest weight loss was recorded in wood

Figure 1 Colour change in oil palm wood before (a) and after (b) hydrothermal treatment

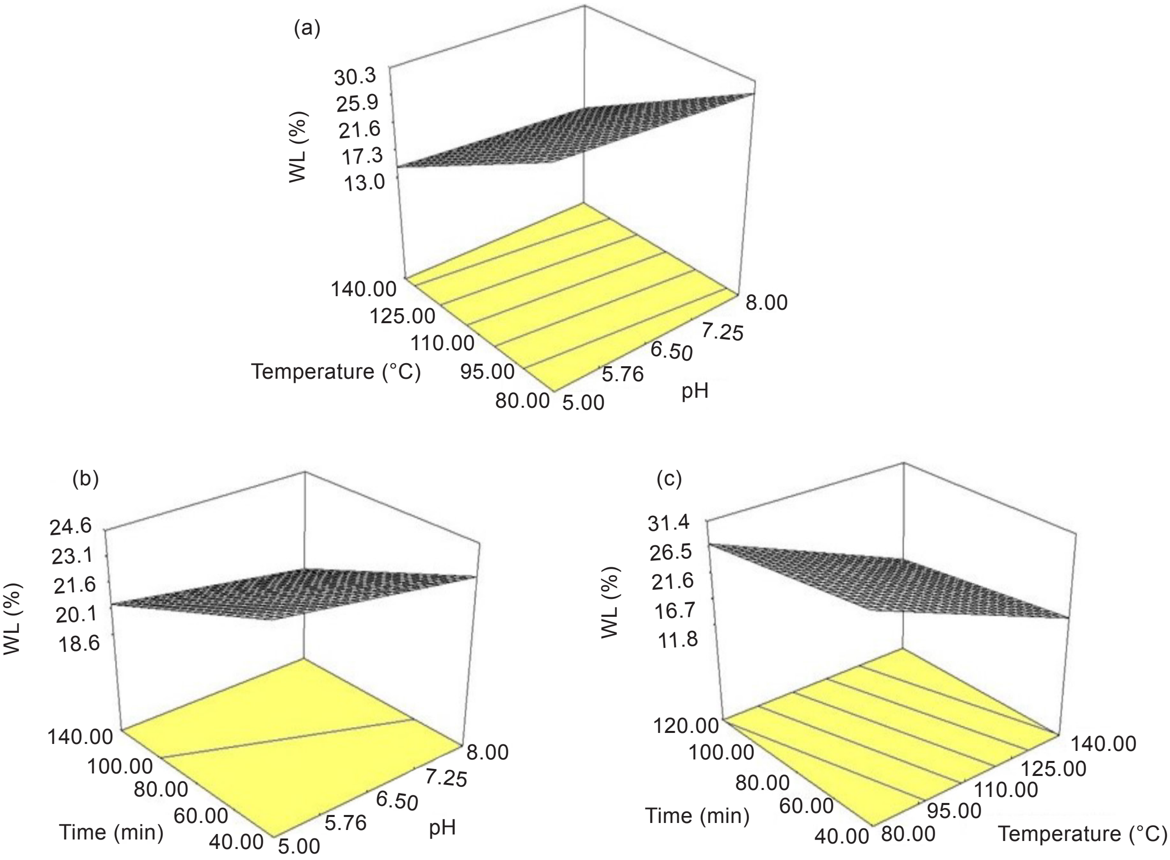

Figure 2 3D-surface plot of weight loss (WL) in percentage as a function of temperature and buffered pH, time and buffered pH and temperature and time

treated with the lowest temperature, 60 °C for 80 min in buffer 6.5. The results implied that treatment temperature is the most influential factor in enhancing decay resistance in OPW. Apart from that, treatment time also exerted significant influence on the decay resistance of OPW. When the treatment temperature and pH value remained constant, a decrement in weight loss was observed with the increase of treatment time, from 40 to 120 min. However, the influence of treatment time on decay resistance enhancement was less than treatment temperature. For example, sample treated at 110 °C for 147 min showed higher weight loss compared to sample treated at 160 °C for 80 min (18.24% versus 9.00%).

The improvement in decay resistance could be attributed to the reduction of EMC in treated samples. The reduction of EMC is directly proportional to the increase of temperature and time (Lee et al. 2015). As shown in Figure 3, lower EMC was observed when the samples were treated at higher temperature. When the pH value of the treating medium remained constant, samples treated at 140 °C for 40 min exhibited lower EMC in comparison to that treated at 80 °C for 120 min. Decrement of hydroxyl groups, when subjected to elevated temperature, led to lower moisture absorption. Lower moisture content of treated OPW results in insufficient moisture for fungi survival, which prevents the attack of white rot fungus (Nabil et al. 2016).

It is interesting to note that, samples treated in acidic medium showed lower EMC. Lower EMC was observed in OPW treated in acidic medium (buffer 4 and 5), followed by neutral (buffer 6.5) and alkaline (buffer 8 and 9) medium. Decomposition of hemicellulose and cellulose are highly dependent on the formation of carbonic acid during hydrothermal treatment (Tjeerdsma and Militz 2005). Therefore, treatment in acidic medium accelerated the decomposition of holocellulose, especially hemicellulose, and subsequently reduce the accessibility of the hydroxyl groups in hemicellulose to water, to a greater extent. Treatment in alkaline

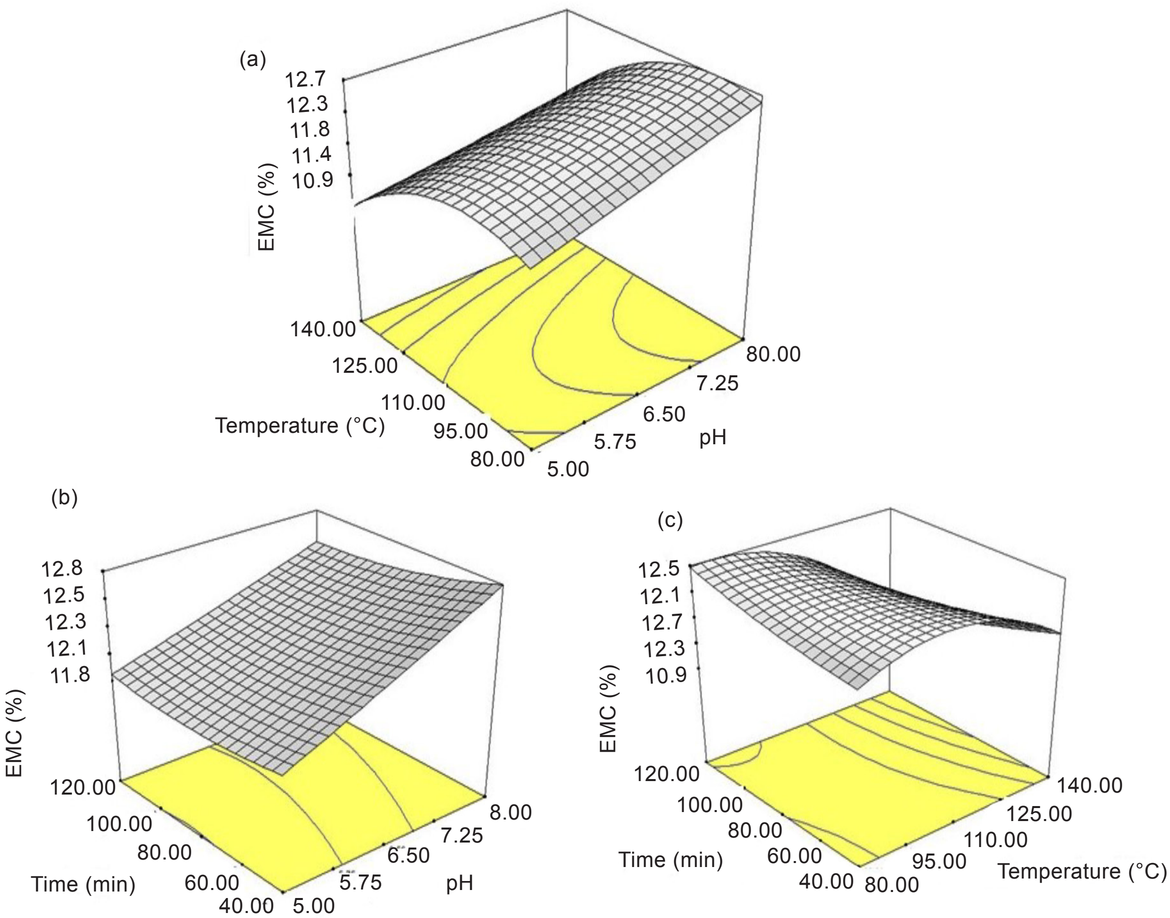

Figure 3 3D-surface plot of equilibrium moisture content (EMC) in percentage as a function of temperature and buffered pH, time and buffered pH and temperature and time

medium ameliorated this effect, as hydrothermal treatment in alkaline medium reduced the destructive effects of the released acids which led to lower hemicellulose decomposition rate (Ebadi et al. 2016).

On the contrary, buffer medium did not significantly affect the decay resistance of OPW. However, although statistically insignificant, better decay resistance was observed in OPW treated in neutral (buffer 6.5) and alkaline (buffer 8 and 9) medium, compared to acidic medium (buffer 4 and 5). Kamdem et al. (2002) suggested that the degradation of chemical contents take place in the sequence of hemicellulose, cellulose and lignin, with lignin being the most thermally stable constituent in wood. Therefore, higher lignin content was observed as a result of degradation of hemicellulose and cellulose caused by higher rate of hydrolysis and carbohydrate degradation in acidic buffered media (Talaei & Karimi 2012b). As lignin is a constituent that is preferred by white rot fungus, higher mass loss was observed in OPW treated in acidic buffered medium.

CONCLUSIONS

A linear and a quadratic equation was successfully developed using RSM model and the adequacy of the models was proved by high adjusted r^2 values. Improvement in decay resistance against *T. versicolor* was observed in OPW after hydrothermal treatment. Among the treatment variables, treatment temperature and time exerted significant effects on the weight loss of OPW, caused by white rot fungus. Despite showing significant effect on EMC reduction, buffered medium exhibited insignificant effect on decay resistance enhancement of OPW. Therefore, it was concluded that the treatment temperature and time are important factors, with treatment temperature as the most crucial factor, determining the extent of decay resistance of OPW.

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