Sains Malaysiana 43(5)(2014): 775–782

# Response Surface Methodology Models of Processing Parameters for High Performance Phenolic *Compreg* Wood (Model Kaedah Permukaan Respons bagi Parameter Pemprosesan untuk

Kayu Fenolik Termampat Berprestasi Tinggi)

# A. ZAIDON\*, G.H. KIM, E.S. BAKAR & H. RASMINA

#### ABSTRACT

*The aim of the study was to develop response surface methodology (RSM) models for polymer loading, density, dimensional stability, strength and stiffness of compressed wood of sesenduk (*Endospermum diadenum*) treated with phenol formaldehyde (PF). Central composite design (CCD) using RSM with three processing parameters was studied in their specific ranges: PF concentration (PC) from 24-40%, pre-curing time (PCT), 3-9 h and compression ratio (CR), 70-90%. The experimental design was analysed and interpreted using the Design Expert Software (Stat Ease version 8) and the responses of 3d plots were built using the same software. Quadratic models in terms of PC, PCT and CR were developed for polymer loading, density, reduction in water absorption and modulus of rupture in static bending. Multiple linear equations were developed for anti-swelling efficiency and modulus of elasticity. The experimental values were in good agreement with predicted ones and the models were highly significant with correlation coefficients between 0.626 and 0.926. PC and CR had significant effects on the responses. The range of PCT used did not significantly affect the responses. It was also found that the improvement of properties ranged from moderately to highly correlated with the polymer loading in the compreg wood.* 

*Keywords: Central composite design; compreg; phenol formaldehyde*

### ABSTRAK

*Tujuan kajian ini adalah untuk membangunkan model kaedah permukaan respons (RSM) untuk ketahanan polimer, ketumpatan, kestabilan dimensi, kekuatan dan keanjalan kayu sesenduk (*Endospermum diadenum*) termampat (Compreg) yang dirawat dengan fenol formaldehid (PF). Reka bentuk komposit tengah (CCD) menggunakan RSM dengan tiga parameter pemprosesan dikaji dalam julat kepekatan PF (PC) 24-40%, masa pra-pematangan (PCT), 3-9 jam dan nisbah mampatan (CR), 70-90%. Reka bentuk ujian dianalisis dan ditafsirkan menggunakan perisian Design Expert (Stat Ease version 8) dan plot respons 3d dihasilkan dengan menggunakan perisian yang sama. Model kuadratik dibangunkan untuk ketahanan polimer, ketumpatan, pengurangan penyerapan air dan modulus kepecahan dalam lenturan statik berdasarkan PC, PCT dan CR. Persamaan linear berganda dibangunkan untuk keberkesanan anti-pembekaan dan modulus kekenyalan. Nilai yang diperoleh daripada ujian mempunyai persamaan dengan nilai yang diramalkan dan model yang dihasilkan mempunyai kesan yang ketara dengan pekali korelasi antara 0.626 dan 0.926. PC dan CR mempunyai kesan yang besar ke atas respons. Julat PCT yang digunakan tidak memberi kesan ketara kepada respons. Kajian ini juga mendapati peningkatan sifat kayu berkait rapat dengan ketahanan polimer dalam kayu compreg dalam skala sederhana ke tahap tinggi.*

*Kata kunci: Compreg; fenol formaldehid; reka bentuk komposit tengah*

### **INTRODUCTION**

A series of work has been conducted to enhance the properties of low density tropical hardwood through impregnating the wood strips with low molecular weight (approximately 600) phenol formaldehyde and subsequently followed by laminating the product under hot press (Zaidon 2009). The treated product, which is also known as *compreg* laminate, was found to be suitable for parquet flooring, panelling and furniture components. A cost-effective way to produce high performance *compreg* laminates is through optimizing the operational conditions. This can be done by employing the response surface

methodological (RSM) approach. It was also been found that the dominant factors in improving the properties of this product are the level of polymer loading and the density of the produced *compreg.* The dimensional stabilisation of compressed wood is dependent on polymer loading and polymer distribution in the treated wood (Rowell & Konkol 1987), while density and mechanical properties of the compressed wood increased with increasing polymer loading coupled with the densification process (Yano et al. 2001)

The RSM is a mathematical and statistical technique used for analyzing the effects of several independent variables (Myers & Montgomery 2002). It is a robust mathematical and statistical technique for models that alleviate tribulation where multiple parameters may influence responses. In many cases, the relationship between the response and independent variables is not known. This approach is normally designed using central composite design (CCD) in which the design is to explore the effect of variables on the response in the region of investigation. The CCD with RSM has previously been used to evaluate the effects of multiple parameters on properties of flakeboards (Wang & Lam 1999) and medium density fibreboards (Li et al. 2009). This approach has also been successfully used to optimize processing variables on polymer loading in compressed wood (Zaidon et al. 2012). It was also found that the treating solution concentration, pre-curing time and compression ratio were the most effective independent variables. In the first step of RSM, it is crucial to approximate the response in terms of analyzing the independent variables. In this case, a loworder polynomial equation in a pre-determined region of the independent variables is employed, which will be analysed later to locate the optimum values of independent variables for the optimum response (Ceylan et al. 2008). In this study, the RSM models for polymer loading, density, dimensional stability, strength and stiffness of compressed wood of *sesenduk* (*Endospermum diadenum)* treated with phenol formaldehyde (PF) were developed. The PF concentration (PC), pre-curing time (PCT) and compression ratio (CR) were taken as the independent variables. The CR is taken as the percentage of the thickness ratio after and before compressing the wood. This paper also reported the correlation of polymer loading with density, dimensional stability and mechanical properties of the compressed wood.

#### MATERIALS AND METHODS

### RESPONSE SURFACE METHODOLOGY AND CENTRAL COMPOSITE DESIGN

In the present study, CCD using RSM was used to determine the optimal treatment variables of impregnating the *sesenduk* wood (*Endospermum diadenum)* with low molecular weight (approximately 600) phenol formaldehyde (PF) resin, subsequently followed by compressing in a hot press. Three independent variables, phenol formaldehyde concentration (PC), pre-curing time (PCT) and compression ratio (CR) were selected and the response variables included polymer loading (PL), density, reduction in water absorption (RWA), anti-swelling efficiency (ASE), modulus of rupture(MOR) and modulus of elasticity (MOE) in static bending. The CCD was applied using Design Expert Software (State Ease, Design Expert 8). In the regression equation, the test variables were coded according to (1):

$$
xi = \frac{\left(x_i - x_{i0}\right)}{\Delta x_i},\tag{1}
$$

where *xi* is the independent variable coded value,  $x_i$  is the independent variable real value,  $x_{i0}$  is the independent real value on the centre point (Roriz et al. 2009) and  $\Delta x$  is the interval,  $i = 1, 2, 3$ .

The range and the levels of the variables under investigation are given in Table 1. Each of the independent variables was studied at five different levels containing two star points ( $\alpha = 1.68$ ) and seven replications of the central point with a total of 20 experiments. The effect on the responses corresponding to the combined effects of the three variables was studied in their specific ranges: PC from 24-40%, PCT, 3-9 h and CR (percent change of thickness after compressing), 70-90%. The plan of CCD in coded and actual levels of the three independent variables is shown in Table 2.

#### *COMPREG* TREATMENT

Pre-weighed, air dry (15% MC) wood strips of the *sesenduk* measuring 25 mm wide  $\times$  150 mm long  $\times$  5 mm thick were treated with low molecular weight (approximately 600) PF resin (PC, 24.05-40%) using a vacuum-pressure process. The process involved 30 min vacuum followed by filling the set-up with a solution and left soaked under a pressure of 690 kPa for 30 min. After the treatment, they were precured (PCT, 3-9 h) in an oven at a temperature of 65ºC prior to compressing (CR, 70-90%) in a hot press at  $150\pm2\degree C$  for 20 min (Rowell & Konkol 1987). The *compreg* was then conditioned in a conditioning room at  $25 \pm 2^{\circ}C$ ,  $65 \pm 2\%$ RH until a constant weight was achieved. The weight and volume were recorded. The polymer loading (PL) and density were determined as follows:

PL 
$$
\% = 100 [(W_f - W_i)] / W_i.
$$
 (2)

Density kgm<sup>-3</sup> = W<sub>f</sub> / V<sub>f</sub>, 
$$
(3)
$$

Factor	Variable	Unit	Range and level of actual and coded values					
			$-\alpha$	-			$\alpha$	
$\lambda$ ,	РC	%	20	24.05	30	35.95	40	
$\Lambda_{\alpha}$	PCT			4.21		7.79		
v $\Lambda$	CR	$\%$	70	74.05	80	85.95	90	

TABLE 1. The range and levels of the variables

PC = phenol formaldehyde concentration, PCT = pre-curing time, CR = compression ratio

Run		Coded factor		Actual factor				
	$x_{I}$	$x_{\scriptscriptstyle 2}^{}$	$x_{3}$	PC(%)	PCT(h)	$CR(\%)$		
1	1	$-1$	$-1$	24.05	4.21	74.05		
2		$-1$	$-1$	35.95	4.21	74.05		
3			$-1$	24.05	7.79	74.05		
$\overline{4}$			$-1$	35.95	7.79	74.05		
5		$-1$	1	24.05	4.21	85.95		
6		$-1$	1	35.95	4.21	85.95		
7	1	1	1	24.05	7.79	85.95		
8			1	35.95	7.79	85.95		
9	$-1.68$	$\theta$	$\mathbf{0}$	19.99	6.00	80.00		
10	1.68	$\theta$	$\mathbf{0}$	40.01	6.00	80.00		
11	$\mathbf{0}$	$-1.68$	$\mathbf{0}$	30.00	2.99	80.00		
12	$\mathbf{0}$	1.68	$\overline{0}$	30.00	9.01	80.00		
13	$\overline{0}$	$\theta$	$-1.68$	30.00	6.00	69.99		
14	$\mathbf{0}$	$\Omega$	1.68	30.00	6.00	90.01		
15	$\mathbf{0}$	0	$\mathbf{0}$	30.00	6.00	80.00		
16	$\mathbf{0}$	$\theta$	$\mathbf{0}$	30.00	6.00	80.00		
17	$\mathbf{0}$	$\Omega$	$\mathbf{0}$	30.00	6.00	80.00		
18	$\mathbf{0}$	$\theta$	$\mathbf{0}$	30.00	6.00	80.00		
19	$\overline{0}$	$\theta$	$\boldsymbol{0}$	30.00	6.00	80.00		
20	0	0	$\overline{0}$	30.00	6.00	80.00		

TABLE 2. Experimental conditions of central composite design (CCD)

 $PC =$  phenol formaldehyde concentration,  $PCT =$  pre-curing time,  $CR =$  compression ratio

where  $W_f$  is the constant weight,  $V_f$  is the volume in a conditioning room after treatment and  $W<sub>i</sub>$  is the constant weight in a conditioning room before treatment.

### DIMENSIONAL STABILITY EVALUATION

Dimensional stabilization was quantified by comparing the samples of the treated and untreated specimens. The dimensional stability of the treated samples was determined in terms of RWA and ASE and this was done by vacuumsoaking the treated and untreated strips in distilled water based on the method of Ashaari et al. (1990). The weights and volumes before and after soaking in water were measured and these data were used to calculate RWA and ASE using the following equations:

$$
ASE\% = 100 [(S_c - S_t)/S_c].
$$
 (4)

$$
RWA \, \% = 100 \, [(W_c - W_t) / W_c)], \tag{5}
$$

where  $S$  is the untreated volumetric swelling coefficient,  $mm<sup>3</sup>, S<sub>t</sub>$  is the treated volumetric swelling coefficient, mm<sup>3</sup>, W is the weight gain in the untreated wood due to water pick-up after 24 h,  $\%$ , and W is the weight gain in the treated wood under the same condition, %.

## STATIC BENDING EVALUATION

The strength and stiffness in static bending of the treated wood were evaluated based on modulus of rupture (MOR) and modulus of elasticity (MOE), respectively. The tests were performed according to the procedure specified in the British Standard BS 373:1957 for small clear test specimen (BSI 1957) with a modification of the specimen size. Smaller test blocks were used in this study due to the limitation in the sample size. The tests were made on the specimens with the dimension of  $10 \times 20 \times 150$ mm. All tests were conducted under static load carried out on 50 kN load. The centre loading was applied at a span length of 120 mm. MOE and MOR were calculated as follows:

$$
MOE, Nmm^{-2} = P1L3/4Dbh3.
$$
 (6)

$$
MOR, Nmm^{-2} = 3P_m L / 2bh^2,
$$
 (7)

where  $P_1$  is the load at proportional limit, N,  $P_m$  is the maximum breaking load, N, L is the span of the test specimens, mm, D is the deflection at mid-span resulting from  $P_1$ , mm, b is the width of the test specimens, mm, h is the height of the test specimens, mm,  $P_m$  is the load at proportional limit under compression, N, and A is the area of cross-section normal to the direction of load, mm2 .

### EXPERIMENTAL DETAILS

The results of the experimental design were analysed and interpreted using the Design Expert Software (Stat Ease version 8) and the responses of the 3D plots were built using the same software. Pearson's correlation analysis was carried also out to determine the effect of polymer loading and density on the dimensional stability, strength and stiffness of the *compreg* wood.

## RESULTS AND DISCUSSION

The actual and predicted percentages of the responses obtained through the RSM analysis are presented in Table 3. The actual values corresponded to the measured response data for a particular run and the predicted values were evaluated from the model. We used both quadratic and multiple linear models to explain the mathematical relationship between the independent variables and the dependent responses. Stepwise analysis was used to obtain precise RSM models. Any coefficients that were statistically insignificant were deleted from the equation and added to the lack of fit. As a result, new regression models were established for the actual factors. Four quadratic equations (8-10 and 12) and multiple linear equations (11 and 13) were obtained in this study.

$$
WPG = 656.2 + 2.322PC - 16.21CR + 0.098CR2.
$$
\n(8)

Density = 
$$
6635 - 51.48PC - 43.38PCT
$$
  
- 120.49CR + 1.620PCxPCT  
+ 0.0.607PCxCR + 0.602CR<sup>2</sup>. (9)

$$
RWA = 463.17 + 0.895PC - 10.04CR + 0.061CR2.
$$
\n(10)

$$
ASE = -91.58 + 1.115 PC + 1.260CR.
$$
 (11)

$$
MOR = 1199 - 12.423PC - 23.55CR
$$

$$
+ 0.173PC \times CR + 0.12214CR2 \tag{12}
$$

$$
MOE = 68043 - 2424PC - 685.7CR + 32.77PC \times CR.
$$

(13)

### ADEQUACY OF THE MODEL

The fitted models need to be assessed to ensure that they give sufficient approximation of the results obtained in the experimental conditions. The adequacy of the models is given in Table 4. The models of the equation and each term were significant at less than 1% level, while the lack of fit values were insignificant reflecting that the model was statistically appropriate for further analysis. The coefficient of multiple regressions,  $r^2$ , is a global statistic parameter to assess the fit of a model (Myers & Montgomery 2002) and the adjusted  $r^2$  was used for confirming the model adequacy. In these models, the  $r^2$ values ranged between 0.626 and 0.926, which indicated that the fitness of the models varied from moderate to very high. For further validation of the model, the adjusted  $r<sup>2</sup>$  was used for confirming the model adequacy. The adjusted  $r^2$  values were calculated to be between  $0.540$ and 0.912, which indicated fair to good models for use in the field conditions. A residual analysis was also carried out for validating the model accuracy (Myers & Montgomery 2002). Identification of the outliers was performed by examining the internally studentised residuals. The residual should be approximately normal with mean zero and unit variance. The results in Table 4 showed that none of the studentised residuals had a value higher than 3. In order to validate the models further and check the outliers, the R-studentised residuals were calculated (Table 3). All the values ranged within -2.5

TABLE 3. The actual and predicted responses

Run		PL, $\%$		Density, $kg.m^{-3}$		RWA, %	ASE, $%$			$MOR, N/mm^{-2}$	$MOE$ , $N/mm^{-2}$	
	Act.	Pred.	Act.	Pred.	Act.	Pred.	Act.	Pred.	Act.	Pred.	Act.	Pred.
1	52.33	51.52	840	838	75.31	77.42	22.19	28.33	131.7	134.7	17633	17326
2	83.44	79.16	838	842	88.05	88.08	46.15	41.48	140.1	139.3	19558	17356
3	54.18	51.52	827	823	80.50	77.42	25.90	28.33	135.3	134.7	18355	17326
4	84.55	79.16	868	895	91.66	88.08	54.38	41.48	134.4	139.3	17569	17356
5	45.44	46.04	729	725	74.15	74.65	45.53	43.33	152.3	136.5	19081	18545
6	75.37	73.67	787	814	89.36	85.30	63.55	56.48	172.6	165.6	24083	23217
7	45.70	46.04	719	709	70.88	74.65	45.56	43.33	135.5	136.5	20604	18545
8	80.57	73.67	872	867	91.42	85.30	57.93	56.48	171.7	165.6	26022	23217
9	40.32	35.87	722	725	75.14	70.23	33.60	31.34	122.8	125.5	16839	17134
10	77.44	82.35	903	861	82.93	88.15	37.75	53.46	153.1	153.9	19512	21088
11	56.20	59.11	814	777	74.50	79.19	33.26	42.40	133.9	139.7	17552	19111
12	59.61	59.11	835	809	83.48	79.19	39.87	42.40	139.3	139.7	18426	19111
13	68.28	73.58	924	913	86.24	87.67	31.61	29.79	146.7	140.2	16598	16134
14	62.30	64.35	794	793	80.73	82.99	54.48	55.01	149.6	163.7	21056	22088
15	57.32	59.11	794	793	83.14	79.19	52.15	42.40	137.4	139.7	18178	19111
16	54.44	59.11	754	793	77.02	79.19	44.49	42.40	155.3	139.7	20038	19111
17	59.91	59.11	789	793	80.17	79.19	42.72	42.40	142.1	139.7	17722	19111
18	56.21	59.11	751	793	77.75	79.19	34.76	42.40	123.6	139.7	17752	19111
19	56.70	59.11	790	793	77.53	79.19	43.30	42.40	144.1	139.7	18162	19111
20	59.53	59.11	798	793	73.53	79.19	38.88	42.40	131.5	139.7	17479	19111

PL = polymer loading, RWA reduction in water absorption, ASE= anti-swelling efficiency, MOR = modulus of rupture, and MOE = modulus of elasticity

	Model F-value (P-value)	Lack of Fit F-value (P-value)	r-squared	Adjusted r-squared	R-studentised residual
PL	$66.66 \, (\leq 0.0001)$	4.43(0.174)	0.926	0.912	0 <sub>±</sub> 1.0
Density	$13.02 \, \text{(} < 0.0001\text{)}$	1.89 (0.297)	0.857	0.791	$0+2.0$
<b>RWA</b>	10.21(0.0005)	1.74 (0.280)	0.657	0.593	0 <sub>±</sub> 1.70
<b>ASE</b>	14.25 (0.0002)	1.56 (0.326)	0.626	0.580	$0+2.60$
<b>MOR</b>	6.61(0.0028)	0.55(0.8003)	0.638	0.540	$0+2.80$
<b>MOE</b>	11.03 (0.0002)	3.22 (0.1033)	0.674	0.613	$0+2.30$

TABLE 4. Adequacy of the models

PL = polymer loading, RWA reduction in water absorption, ASE= anti-swelling efficiency, MOR = modulus of rupture, and MOE = modulus of elasticity

and  $+2.5$  (values between  $-3$  and  $+3$  were the acceptable limits), thereby validating the models.

### EFFECTS OF INDEPENDENT VARIABLES ON THE RESPONSES

The effects of variables on the responses are shown in Figures 1-6. The graphs were constructed by plotting the central values of the variables that affected the responses, i.e. 30% PC, 6 h PCT and 80% CR, respectively, representing (a), (b) and (c) plots. As shown in Figure 1(a), the polymer loading increased with increasing resin concentration

regardless of pre-curing time and reached its highest value (74%) at 36% of resin concentration. Increasing the PC and decreasing the CR increased the polymer loading in the wood (Figure 1(b)). A maximum loading of  $80\%$  was attained at 36% PC and 74% CR. Figure 1(c) shows that the PL increased with decreasing CR at any PCT. PCT gave no significant effect on PL in the wood within the range of the tested period. The maximum loading was 65%. Figure 2 exhibits the density as a function of the variables. The plot  $2(a)$  showed that at a fixed value of  $80\%$  CR, the density increased with increasing PC regardless of PCT.



PL = polymer loading, PC = phenol formaldehyde concentration, PCT = pre-curing time, CR = compression ratio and RWA reduction in water absorption

FIGURE 1. 3D-surface plots of PL as a function of PC and PCT at a fixed value of 80% CR (a), PC and CR at a fixed value of 6 h PCT (b) and PCT and CR at a fixed value of 30% PC (c)



PL = polymer loading, PC = phenol formaldehyde concentration, PCT = pre-curing time, CR = compression ratio and RWA reduction in water absorption

FIGURE 2. 3D-surface plots of density as a function of PC and PCT at a fixed value of 80% CR (a), PC and CR at a fixed value of 6 h PCT (b) and PCT and CR at a fixed value of 30% PC (c)

The maximum density was 870 kgm<sup>-3</sup>. At a fixed value of PCT 2(b), the density increased and reached its highest value (860 kgm<sup>-3</sup>) at 36% PC and 74% CR. As for PL, the density of the *compreg* was not affected by the PCT within the range of the tested period  $2(c)$ .

The trend of reduction in water absorption (RWA) was very much similar to PL. The PCT was found to have no significant effect on RWA at any fixed value of PCT and CR (Figure 3(a) and 3(b)). The maximum RWA achieved was 87.5% at 36% PC and 74% CR (Figure 3(b)) and 82.5% at 74% CR at a fixed value of 30% PC (Figure 3(c)). Figure 4 shows that the anti-swelling efficiency (ASE) was linearly affected by PC and CR, while PCT gave no significant effect on the response. At a fixed value of CR (80%), regardless of PCT, the ASE reached its highest value (49%) at 36% PC (Figure 4(a)) and at any PCT, the maximum ASE attained was 55% when the wood was impregnated with 36% PC and compressed at 86% CR (Figure 4(b)).

As regard to the strength of the *compreg* in terms of modulus of rupture (MOR) in static bending, at a fixed value of  $CR$ , only PC affected the response (Figure 5(a)). The MOR increased with the increasing PC with a maximum value of 148 Nmm<sup>-2</sup> at 36% PC. At any PCT, the highest value for MOR (165 Nmm<sup>-2</sup>) was attained at PC 36% and CR 86% (Figure 5(b)). At a fixed value of PC, the response increased to a maximum value of 150 Nmm-2 as the CR increased up to 86% CR, while PCT had no effect on the response (Figure 5(c)). For MOE (Figure 6(a)), at  $80\%$  CR, the response increased with increasing PC to 36% with the highest value of 20,200 Nmm<sup>-2</sup>. At any PCT, MOE reached its highest value  $(23,000 \text{ Nmm}^{-2})$  at 36% PC and 86%  $CR$  (Figure 6(b)). Figure 6(c) shows the maximum MOE (20,500 Nmm-2) at 30% PC and 86% CR.

As a whole, it was found that only PC and CR had a significant effect on the responses. The range of PCT used did not significantly affect the responses. The purpose of pre-curing is to plasticize the cell wall before initiating collapse at lower pressure (Shams & Yano 2004). The PF resin is effective since it polymerised before curing and fixed the deformation condition permanently after curing (Shams & Yano 2011). It is believed that the PF resin had been polymerised earlier than the range of PCT used in this study. Any attempt to prolong the PCT would not make any changes on the degree of the polymerisation.

It is also interesting to note that the dominant factors in improving the dimensional stability and properties of the treated wood are the level of polymer loading (Zaidon et al. 2012) and polymer distribution (Inoue et al. 1991).



 $PL =$  polymer loading,  $PC =$  phenol formaldehyde concentration,  $PCT =$  pre-curing time,  $CR =$  compression ratio and RWA reduction in water absorption

FIGURE 3. 3D-surface plots of RWA as a function of PC and PCT at a fixed value of 80% CR (a), PC and CR at a fixed value of 6 h PCT (b) and PCT and CR at a fixed value of 30% PC (c)



PC = phenol formaldehyde concentration, PCT = pre-curing time, CR = compression ratio, ASE= anti-swelling efficiency, MOR = modulus of rupture, and  $MOE =$  modulus of elasticity

FIGURE 4. 3D-surface plots of ASE as a function of PC and PCT at a fixed value of 80% CR (a), PC and CR at a fixed value of 6 h PCT (b) and PCT and CR at a fixed value of 30% PC (c)

PC = phenol formaldehyde concentration, PCT = pre-curing time, CR = compression ratio, ASE= anti-swelling efficiency, MOR = modulus of rupture, and  $MOE =$  modulus of elasticity

FIGURE 5. 3D-surface plots of MOR as a function of PC and PCT at a fixed value of 80% CR (a), PC and CR at a fixed value of 6 h PCT (b) and PCT and CR at a fixed value of 30% PC (c)



 $PC =$ phenol formaldehyde concentration,  $PCT =$ pre-curing time,  $CR =$ compression ratio,  $ASE =$ anti-swelling efficiency, MOR = modulus of rupture, and  $MOE =$  modulus of elasticity

FIGURE 6. 3D-surface plots of MOE as a function of PC and PCT at a fixed value of 80% CR (a), PC and CR at a fixed value of 6 h PCT (b) and PCT and CR at a fixed value of 30% PC (c)

Density and mechanical properties of the compressed wood increased with increasing polymer loading coupled with the densification process (Yano et al. 2001). It is therefore, worth to analyse the correlation between PL and other dependent variables.

 $\overline{1}$ 

#### EFFECTS OF POLYMER LOADING ON DIMENSIONAL STABILITY AND MECHANICAL PROPERTIES OF THE *COMPREG*

The Pearson's correlation between PL and the independent variables of the *compreg* wood is shown in Table 5. Positive and significant correlations were found between PL and the independent variables at *p*<0.05. High correlations were found for density  $(r=0.719)$  and R  $(r=0.884)$ . The results showed that PL was not the only factor that affected density and it might also be postulated due to the compression process. Rowell (2005) found that PL together with hotpressing densification determined the final density of the *compreg*. It has also been proven that the density of the *compreg* increased with the degree of compression in a hot press (Rabia'tol Adawiah et al. 2012). Higher correlation (*r*=0.884) obtained for reduction in water absorption could be due to the higher amount of polymer retained in the wood structure, thus acting as a barrier that limits water penetrating into the wood.

The low correlation between PL and ASE (*r*=0.499) indicated that the resin had successfully bulked the cell wall to some extent. In addition, during hot pressing of the PF resin, the methyl groups in the phenolic rings were converted to methylene bridges resulting in the formation of a very highly cross-linked thermoset polymer (Collins 1996). The PL also gave significant effects to the mechanical strength (MOR) and stiffness (MOE) of the *compreg* wood, but the effects were low (*r*=0.520 for MOR and *r*=0.482 for MOE).

### **CONCLUSION**

The RSM was successfully applied to determine the optimal operational conditions for high performance *compreg E. diadenum* wood. The quadratic models in terms of PC, PCT and CR were developed for polymer loading, density, reduction in water absorption and modulus of rupture in static bending. Multiple linear equations were developed for anti-swelling efficiency and modulus of elasticity. The experimental values were in good agreement with the predicted values and the models were highly significant with correlation coefficients between 0.626 and 0.926. It was found that only PC and CR had significant effects on the responses. On the other hand, the range of PCT used did not significantly affect the responses. It was also found that the improvement of properties varied from moderately to highly correlate with the PL in the *compreg* wood.

TABLE 5. Pearson's correlations of PL with other dependent variables

		$PL\%$	Density, $k_{\rm g}$ mm <sup>-3</sup>	$RWA$ , $%$	ASE, $%$	$MOR Nmm^{-2}$	$MOE Nmm-2$
PL, $\%$	Mean	62.04	808	80.67	42.70	142.6	19,100
	Pearson's		0.719	0.884	0.499	0.520	0.482
	Sig.		0.0004	< 0.0001	0.0249	0.0186	0.0311
	N	20	20	20	20	20	20

PL = polymer loading, RWA reduction in water absorption, ASE= anti-swelling efficiency, MOR = modulus of rupture, and MOE = modulus of elasticity

### ACKNOWLEDGEMENTS

The authors wish to thank Universiti Putra Malaysia for the Research University Grant Scheme (RUGS) to finance this research.

#### **REFERENCES**

- Ashaari, Z., Barnes, H.M., Vasisth, R.C., Nicholas, D.D. & Lyon D.E. 1990. Effect of aqueous polymer treatments on properties. Part I: Mechanical properties. *IRG/WP/3610*.
- BSI (British Standard Institute). 1957. BS 373*. Methods of Testing Small Clear Specimens of Timber.* London: British Standard Institution.
- Ceylan, H., Kubilay, S., Aktas, N. & Sahiner, N. 2008. An approach for prediction of optimum reaction conditions for laccase-catalysed bio-transformation of 1-nalphtol by response surface methodology (RSM). *Bioresource Technology* 99: 2025-2031.
- Collins, P.J. 1996. Current research activities on wood adhesives at CSIRO. *25th Forest Research Conference*, 18–21 November 1996, Victoria, Australia.
- Inoue, M., Norimoto, M., Otsuka, Y. & Yamada, T. 1991. Surface compression of coniferous lumber III. Permanent set of the surface compression layer by water solution low molecular weight phenolic resin. *Mokuzai Gakkaishi* 37(30): 234-240.
- Li, X., Li, Y., Zhong, Z, Wang, D., Ratto, J.A., Sheng, K. & Sun, X.S. 2009. Mechanical and water soaking properties of medium density fiberboard with wood fiber and soybean protein adhesive. *Bioresource Technol*. 100: 3556-3562.
- Myers, R.H. & Montgomery, D.C. 2002. *Response Surface Methodology*. New York: John Willey & Sons.
- Rabia'tol Adawiah, M.A., Zaidon, A., Nurizreen, F.A., Bakar, E.S., Mohd Hamami, S. & Paridah, M.T. 2012. Addition of urea as formaldehyde scavenger for low molecular weight phenol formaldehyde treated *compreg* wood. *Journal of Tropical Forest Science* 24(3): 265-274.
- Roriz, M.S., Osma, J.F., Teixeira, J.A. & Couto, S.R. 2009. Application of response surface methodological approach to optimize reactive black 5 decolouration by crude laccase from *Trametes pubescens*. *Journal of Hazardous Materials* 169: 691-696.
- Rowell, R.M. & Konkol, P. 1987. Treatments that enhance physical properties of wood. *Gen. Tech. Rep. FPL-GTR-55.* U.S. Department of Agriculture, Forest Service, Forest Product Laboratory. Madison.
- Rowell, R.M. 2005. Chemical modification of wood. In *Handbook of Wood Chemistry and Wood Composites*, edited by Rowell, R.M. Florida: CRC Press.
- Shams, M.I. & Yano, H. 2004. Compressive deformation of wood impregnated with low molecular weight phenol formaldehyde (PF) resin II; effects of processing parameters. *J. Wood Sci.*  50: 343-350.
- Shams, M.I. & Yano, H. 2011. Compressive deformation of phenol formaldehyde (PF) resin-impregnated wood related to the molecular weight of resin. *Wood Sci. Tech.* 45: 73-81.
- Wang, K. & Lam, F. 1999. Quadratic RSM models of processing parameters for three-layer oriented flakeboards. *Wood Fiber Sci*. 31: 173-186.
- Yano, H., Hirose, A., Collings, P.J. & Yazaki, Y. 2001. Effects of matrix substances as a pretreatment in the production of high strength resin-impregnated wood based materials. *Journal of Material Science Letters* 20: 1125-1126.
- Zaidon, A. 2009. Improvement of raw materials from underutilised timber species through chemical and densification treatments for value added laminated products. *End of Reports submitted to the Ministry of Science and Technology, Malaysia.* Report No. 06-01-04-SF0656 (unpublished).
- Zaidon, A., Kim, G.H., Paridah, M.T., Bakar, E.S. & Rushdan, I. 2012. Optimisation of the processing variables in gaining high polymer loading in compressed wood using response surface methodology (RSM). *Journal of Tropical Forest Science* 24(2): 241-248.

A. Zaidon\* & H. Rasmina Faculty of Forestry Universiti Putra Malaysia 43400 Serdang, Selangor D.E. Malaysia

G.H. Kim Division of Environmental Science and Ecological Engineering Korea University Seoul 136-713 Korea

E.S. Bakar Institute of Tropical Forestry and Forest Products Universiti Putra Malaysia 43400 Serdang, Selangor D.E. Malaysia

\*Corresponding author; email: zaidon@upm.edu.my

Received: 30 May 2013 Accepted: 3 September 2013