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Optimization of preparation conditions for melamine urea formaldehyde based adhesive for plywood application using response surface methodology

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Filler is added to adhesive formulations to reduce resin utilization leading to cost savings. Melamine urea formaldehyde (MUF) resin has been synthesized in the laboratory. The palm kernel meal (PKM) and palm shell (PS) are used as fillers to formulate the melamine urea formaldehyde (MUF) resin based adhesive for interior plywood manufacturing. These formulations are compared with commercialized industry wheat flour (IF) filler. Response surface methodology (RSM) is used for identification of the optimum temperature and pressing time for wood adhesive performance. The experiments have been conducted in the temperature range from 100 to 150°C and pressing time from 50 s to 250 s. The result indicates that the effect of the filler type on plywood shear strength and formaldehyde emission is significant. The optimum shear strength and formaldehyde emission performance of PKM, IF and PS are 1.41 MPa, 1.30 MPa, 1.21 MPa and 0.9988 mg/L, 0.5345 mg/L, 1.2735 mg/L respectively. In addition, the optimum hot press temperature and time of PKM, IF and PS are 124.9°C, 130.9°C, 127.9 °C and 156 s, 153 s, 149 s respectively. This work concludes that, PKM based MUF adhesive resins exhibit potential applications involved in plywood production.

Keywords: Palm kernel meal, Melamine urea formaldehyde resin, Filler, Wood adhesive.

The continuous increasing global demand for energy, the impending depletion of fossil fuels, and concern over global climate change has led to the resurgence in the development of alternative energy sources. Oil palm has acquired significant economic importance for its seed oil which can be converted to biodiesel, is emerging as an alternative to petro-diesel. Besides this, the by-product of palm oil mill such as palm kernel meal (PKM) is also increased directly. In Malaysia, total exports of wood based products were RM 14.89 billion in 2011[1]. However, the production cost of the commercial adhesive for wood composite is increasing day by day because most of them are petrochemical based. At present, thermosetting formaldehyde based adhesives such as phenol formaldehyde (PF), urea formaldehyde (UF) and melamine urea formaldehyde (MUF) resins were used predominantly as adhesives[2]. The emission of formaldehyde, especially from the breakdown of formaldehyde based resins in wood product poses a great hazard to human health because formaldehyde is a human carcinogen[3]. In order to resolve this scenario, a few bio based adhesives such as soybean adhesive, phenol-containing lignins and tannins adhesives, animal glue, casein-based adhesive and blood based adhesive are currently used as wood adhesives[2]. Bio based adhesives are sub-standard to petroleum based adhesives in terms of water resistance and cost. However, in recent years, limited petroleum resources and pollution caused by formaldehyde-based adhesives have spurred many efforts to develop bio based adhesives with good adhesion properties that can compete with synthetic petroleum based adhesives[4]. Resin and filler are the main material for an adhesive for plywood production. Adhesive often compounded with filler to increase viscosity, control rheology and reduce raw material cost. Wood surface consists of small holes, and the filler in the adhesive is used to fill up all the small pores at the surface of wood for strengthening the boding interactions among the components. Apart from this, filler also used to reduce the penetration of resin into the small pores of the wood[5]. There are several types of filler which are used by the industries, such as, wheat flour, plastic, corn starch flour, tapioca flour and others. Experimental design technique is a very useful tool to provide the statistical models which are helpful in understanding the interaction among the parameters to be optimized. The optimization of experimental conditions using RSM is existed widely applied in various processes. However, its application in plywood preparation is very exceptional. Some of the previous studies found in applying RSM in preparation of plywood[6-8]. The focus of this work was to carry out a statistical optimization to determine the optimum preparation condition for plywood, which gives high shear strength and low formaldehyde emission. The effect of hot pressing time and temperature were evaluated in the production of plywood. The materials like PKM, IF and PS were used in this study using three level factorial design method. Empirical models correlating with the shear strength and formaldehyde emission were developed.

Experimental Section

PKM, PS and IF preparation

Palm kernel (*Elaeisguineensis var. tenera*) cake was provided by Felda Palm Industries Sdn. Bhd, Kuantan, Pahang, Malaysia. It was dried at 60°C in the oven for 16 h, later the remaining oil was removed by an extraction process to obtain oil free PKM. It was pulverized in order to obtain a desired size of 50 µm fine powder. The IF and PS were provided by Shin Yang Chemical Sdn. Bhd, Malaysia. The raw IF and PS were sieved by using 50 µm sieve to obtain the uniform fine powder. The materials were stored in an air tight polyethylene bags.

MUF resin preparation

The MUF resin preparation was adopted from Bono *et al*. (2003)[9]. The main materials were used to produce MUF resin are formaldehyde, melamine, and urea respectively. In brief, 100 mL of 37% formalin was poured in a 500 mL three-necked flask and required amounts of melamine, urea were added under vigorous stirring. The temperature of the mixture was maintained at 80°C for 2 h to reach the end point. After that, the *p*H was adjusted again to 8.5-9.0 to stop the polymerization. The mixture was cooled down to 60°C, and excess amount of urea was added to the resin to further reduce the free formaldehyde. The stirring was continued until the temperature reached the room temperature and finally transferred to a plastic container for further testing.

Wood adhesive preparation

For the adhesive (glue) preparation, MUF resin was weighted, and transfer into a beaker, urea was added into the MUF resin and mixed well with a mixer (KHIND Model SM 210). The fillers (IF, PS, PKM) were added in different percentage to the above mixture and mixed well for 5 min. The hardener (ammonium chloride) was added into the mixture (the amount of the hardener is subjected to change).

Type II Plywood preparation

The production of type II plywood was conducted by using Red-Meranti 300 mm × 300 mm × 3.3 mm veneer. In order to get consistence result, veneer was maintained at 10% moisture content and, an equal amount of wood adhesive was used for every plywood produced. The adhesive was applied onto two sides of a core veneer using a glue spreader. The unfinished plywood was left at room temperature for 5 min before it was cold pressed 9 kg/ cm^2 for 20 min. The unfinished plywood was removed after 20 min from the cold press device and left it free for 5 min before it was transferred to hot press device. During hot pressed the pressure 9 kg/cm^2 and temperatures of 100-150°C were applied for 50-250 s. After the process the plywood panel was released from the device and stored at room environment for further testing.

Design of Experiments

The RSM is a statistical method uses quantitative data from appropriate experiments to determine regression model equations and operating conditions[10]. This is for modeling and analysis of problems in which a response of interest is influenced by several variables[11]. A standard RSM design called three level factorial designs; it was applied in this work to study the variables for preparing plywood sample. This method is suitable for fitting a quadratic polynomial model and regression coefficient[12]. The non-linear computer generated quadratic model used in response was as follows, Eq. (1):

$$Y = b_0 + \sum_{i=1}^{n} b_i x_i + \left(\sum_{i=1}^{n} b_{ii} x_i \right)^2 + \sum_{i=1}^{n} \sum_{j=i+1}^{n} b_{ij} x_i x_j \quad ...(1)$$

where Y is the predicted response, b_o the constant coefficient, b_i the linear coefficient, b_{ij} the interaction coefficient, b_{ii} is the quadratic coefficient and x_i, x_j are the coded values of plywood sample preparation variables.

The experimental data were analyzed using statistical software Design Expert software version 6.0.10 (STAT-EASE Inc., Minneapolis, USA) for regression analysis and fit into the model. The model equations developed and evaluated for the statistical significance.

Shear Strength test of plywood

The shear strength of the type II plywood produced, and it was determined by using bonding test according to the Japanese Agriculture Standard (JAS)[13]. Nine plywood test panels (25 mm × 80 mm) were tested for every plywood panel produced. Prior the test, the test pieces were soaked in the hot water bath at 100°C for 4 h. After that, they were dried at 60±3°C for 20 h. Later they were soaked in cold water bath at room temperature. When the test pieces reached cold state, they were used for shear strength testing by using universal testing machine.

Formaldehyde Emission test of plywood

The formaldehyde emission test for the type II plywood was determined according to the JAS[13]. Tenrectangular test panels with the dimensions of 150 mm × 50 mm were prepared. A crystallizing dish with a diameter of 120 mm and a height of 60 mm was placed at the center of (inner volume 9-11 liters) desiccator. 300 mL of distilled water was filled in the crystallizing dish. The test pieces were fixed apart from each other and holds by a metallic holder. After that, metallic holder was placed on the crystallizing dish and keep for 24 h at 20°C. After 24 h, distilled water was used as sample solution for measuring formaldehyde concentration because it absorbed formaldehyde emissions release from test pieces. The measurement of formaldehyde concentration was conducted by using the method of ethyl-acetone luminous intensity absorbance. A sample of 25 mL solution was put into a conical flask with a co-ground stopper. After that, 25 mL of acetyl-acetone ammonium acetate solution is added into a conical flask. The conical flask with a co-ground stopper was warm at 65°C for 10 min. The solution was transferred into an absorbance cell and measured at a wavelength at 412 nm using an UV/V spectrophotometer.

Results and Discussion

Optimization on processing parameter

The optimized values obtained for hot press temperature and time of PKM, IF and PS fillers were used in this study and the values are given in Table 1. The experiments were carried out to evaluate, the maximum shear strength and minimum formaldehyde emission of MUF resin in the range of hot pressing time and temperature.

Development of regression model equation

A polynomial regression equation was developed by using three level factorial design to analyze the factor of interactions by identifying the significant factors contributing to the regression model. The complete design matrix together with response values obtained from the experimental works are given in Table 1. The shear strength and formaldehyde emission of filler were found to be 0.90 MPa to 1.49 MPa and 0.3383 mg/L to 2.1502 mg/L respectively.

According to the sequential model sum of squares, the models were selected based on the highest order polynomials, where the additional terms were significant, and the models were not aliased. For shear strength and formaldehyde emission of plywood, the quadratic models were selected due to higher order polynomial. Besides that, normal probability of studentized from the quadratic model for shear strength and formaldehyde emission performance were evaluated. The plots were satisfactory. So it was concluded that, the quadratic model was adequate to

Table 1 — Average shear strength and formaldehyde emission of various types of natural fillers.

No.run	Temperature, x_1 (°C)	Pressing Time, x_2 (sec)	Average Shear Strength, Y_1 (MPa)			Formaldehyde Emission, Y_2 (mg/L)		
			PKM, x_3	IF, x_3	PS, x_3	PKM, x_3	IF, x_3	PS, x_3
1	100.00	50.00	1.08	0.90	0.91	1.5773	1.2150	2.1502
2	125.00	250.00	1.22	1.11	1.02	0.8524	0.4555	0.7431
3	150.00	50.00	1.12	1.08	0.97	0.9973	0.3785	1.4422
4	100.00	150.00	1.24	0.96	0.94	1.3997	0.9516	1.5774
5	125.00	50.00	1.13	1.03	0.93	1.1891	0.7374	1.4985
6	100.00	250.00	1.13	0.93	0.87	1.1066	0.6759	1.0002
7	125.00	150.00	1.37	1.28	1.19	0.9809	0.5275	1.2392
8	150.00	250.00	1.11	1.06	0.92	0.5367	0.3383	0.9212
9	150.00	150.00	1.22	1.10	1.05	0.7735	0.3563	1.1513

describe the shear strength and formaldehyde emission response surface of the processing parameter optimization[6]. The final empirical models are in terms of factors for shear strength performance (Y_1) and formaldehyde emission performance (Y_2) shown in Eq.(2) to (7) respectively.

$$Y_1[PKM] = -3.0413 + 0.0640x_1 + 5.8235\times10^{-3}x_2 - 2.5370\times10^{-4}x_1^2 - 1.7023\times10^{-5}x_2^2 - 4\times10^{-6}x_1x_2 \quad \ldots(2)$$

$$Y_1[IF] = -3.5286 + 0.067x_1 + 5.7569\times10^{-3}x_2 - 2.5370\times10^{-4}x_1^2 - 1.7023\times10^{-5}x_2^2 - 4\times10^{-6}x_1x_2 \quad \ldots(3)$$

$$Y_1[PS] = -3.3952 + 0.0655x_1 + 5.6069\times10^{-3}x_2 - 2.5370\times10^{-4}x_1^2 - 1.7023\times10^{-5}x_2^2 - 4\times10^{-6}x_1x_2 \quad \ldots(4)$$

$$Y_2[PKM] = 5.5659 - 0.0516x_1 - 5.741\times10^{-3}x_2 + 1.3627\times10^{-4}x_1^2 - 3.7133\times10^{-6}x_2^2 + 3.7933\times10^{-5}x_1x_2 \quad \ldots(5)$$

$$Y_0[IF] = 5.0504 - 0.0515x_1 - 5.063\times10^{-3}x_2 + 1.3627\times10^{-4}x_1^2 - 3.7133\times10^{-6}x_2^2 + 3.7933\times10^{-5}x_1x_2 \quad \ldots(6)$$

$$Y_0[PS] = 5.669 - 0.0478x_1 - 7.6716\times10^{-3}x_2 + 1.3627\times10^{-4}x_1^2 - 3.7133\times10^{-6}x_2^2 + 3.7933\times10^{-5}x_1x_2 \quad \ldots(7)$$

Positive sign in front of the terms indicates synergistic effect, whereas a negative sign indicates that antagonistic effect. The quality of the model developed was evaluated mainly based on the correlation coefficient value, R^2 and R^2_{adj}. The R^2 value in the rang of 0.9032 - 0.9625 for the Eqs. 2 to 7. This indicated that 90.32% and 96.25% of the total variation in the shear strength and formaldehyde emission performance of the plywood respectively. The R^2 value approaches to 1.0, the model is able to provide predicted values are closer to the actual values. The R^2 value of Eq.(2) to (7) were considered relatively good, indicating that there was good agreement with the experimental, predicted shear strength and formaldehyde emission performance of plywood. R^2_{adj} (adjusted determination coefficient) is the correlation measure for testing the goodness of fit of the regression equation. If the R^2_{adj} value is high, degree of correlation between the observed and predicted values will be higher[14]. The R^2_{adj} was in the range of 0.8638 to 0.9472 for the Eqs 2 to 7 respectively. It shows that the model was highly significant and indicated that a high degree of correlation between the observed and predicted data. Furthermore, coefficient of variation (CV) indicates the degree of precision with which the experiments are compared. A relatively low value of (CV) obtained for shear strength and formaldehyde emission performance of the plywood was 5.58 and 9.36 respectively, which shows the better precision and reliability of the experiments carried out.

Statistical analysis

The result of the surface quadratic model in the form of analysis of variance (ANOVA) is given in Table 2 for the shear strength of plywood. ANOVA is required to justify the significance and adequacy of the models. The mean squares were obtained by dividing the sum of the squares of each of the variation sources, the model and the error variance, by the respective degrees of freedom. If the value of Prob>F less than 0.05, the model terms are considered as significant[15,16]. From the Table 2, the model F - value is 22.90 and Prob>F is < 0.0001 it implied that this model was significant. The significant of each coefficient can determined using Prob>F value in Tables 2 and 3. The Prob>F value can be used as a tool to check the significance of each coefficient and the interaction strength between each independent variable. The corresponding variables would be more significant at greater F value and smaller Prob>F value[17]. In this case, x_1, x_3, x_1^2 and x_2^2 factors were significant model term where as x_2, x_1x_2, x_1x_3 and x_2x_3 were insignificant to the response. The lack of fit measures and the failure of the model is represented the data in the experimental domain at a point which are not included in the regression[12]. As shown in Table 2, F - value and Prob>F of the lack of fit were 2.48 and 0.06 respectively. It also implied that, it was not significant relatively to the pure error and indicated that model equation was adequate for predicting the shear strength value of the plywood under any combination of values of the variable.

The ANOVA for the quadratic model for formaldehyde emission performance is listed in Table 3. From the ANOVA for response surface and quadratic model for formaldehyde emission, the model F – value is 62.95 and Prob>F is < 0.0001. It showed that the model was significant. In this case, x_1, x_2, x_3, x_1^2, x_1x_2 and x_2x_3 were significant model terms whereas x_2^2 and x_1x_3 were insignificant to the response. As showed in Table 3, F - value and Prob>F of the lack of fit were 0.85 and 0.6264 respectively, which implied that it was not significant relative to the pure error and indicated that the model equation was adequate for predicting the formaldehyde emission value of the plywood under any combination of values of the variable.

From the statistical results shows that the above models were adequate to predict the shear strength and formaldehyde emission performance within the range of variables studied. Fig. 1 (a) and (b) were shown the predicted values versus the experimental values for shear strength and formaldehyde emission performance respectively. The obtained predicted values are close to the experimental values, indicating that the models developed were satisfactory in

Table 2 — Analysis of variance (ANOVA) for response surface quadratic model for Shear Strength of plywood.

Source	Sum of Squares	DF	Mean Square	F Value	Prob>F	Comment
Model	1.04	11	0.095	22.90	< 0.0001	Significant
x_1	0.025	1	0.025	6.02	0.0208	
x_2	2.689E-03	1	2.689E-03	0.65	0.4273	
x_3	0.27	2	0.014	32.83	< 0.0001	
x_1^2	0.21	1	0.21	50.32	< 0.0001	
x_2^2	0.24	1	0.24	58.00	< 0.0001	
x_1x_2	1.200E-03	1	1.200E-03	0.29	0.5947	
x_1x_3	0.017	2	8.439E-03	2.04	0.1498	
x_2x_3	1.478E-03	2	7.389E-04	0.18	0.8375	
Residual	0.11	27	4.140E-03			
Lack of Fit	0.084	15	5.633E-03	2.48	0.0600	Not significant
Pure Error	0.027	12	2.273E-03			
Cor total	1.15	38				
C.V.	5.58					
R^2	0.9032					
R^2_{adj}	0.8638					

Table 3 — Analysis of variance (ANOVA) for response surface quadratic model for formaldehyde emission of plywood

Source	Sum of Squares	DF	Mean Square	F Value	Prob>F	Comment
Model	5.97	11	0.54	62.95	< 0.0001	Significant
x_1	1.26	1	1.26	145.89	< 0.0001	
x_2	1.15	1	1.15	133.71	< 0.0001	
x_3	3.14	2	1.57	181.89	< 0.0001	
x_1^2	0.060	1	0.060	6.97	0.0136	
x_2^2	0.011	1	0.011	1.32	0.2598	
x_1x_2	0.11	1	0.11	12.52	0.0015	
x_1x_3	0.035	2	0.017	2.02	0.1525	
x_2x_3	0.22	2	0.11	12.75	0.0001	
Residual	0.23	27	8.623E-03			
Lack of Fit	0.12	15	7.975E-03	0.85	0.6264	Not significant
Pure Error	0.11	12	9.434E-03			
Cor total	6.20	38				
C.V.	9.36					
R^2	0.9625					
R^2_{adj}	0.9472					

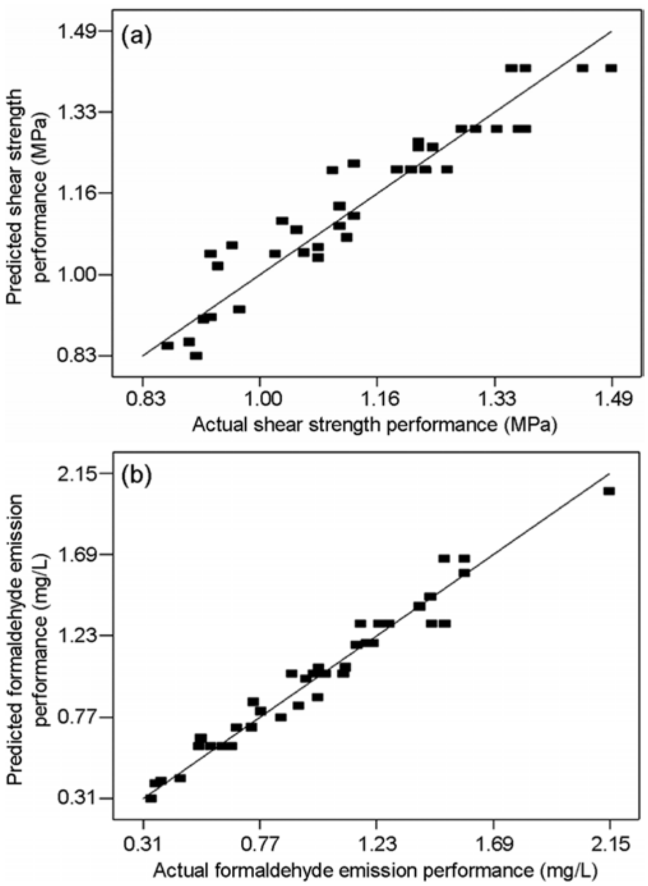

Fig. 1 — Predicted vs. experimental (a) shear strength, (b) formaldehyde emission performance.

capturing the correlation between operating parameter to the response[18].

Shear strength and formaldehyde emission performance

Referring to Table 2, different types of fillers showed the largest *F* - value 32.83 among the factors, indicating that this variable imposed the significant effect on the shear strength performance of plywood. The effect of hot press temperature was significant. However, the effect of hot press time on the response was relatively insignificant. The interaction effects between the variables were insignificant. Figure 2 (a) to (c) shows the three dimensional response surface shows the effects of the hot press temperature, time and different types of fillers on the plywood shear strength performance. The effects of hot press temperature and different types of fillers were studied and, they have significant effects on the response. Figure 2 (a) to (c) shows the shear strength performance increases with an increase in a hot press time and temperature. However, shear strength performance decrease with further increase in a hot press time and temperature after the response met the

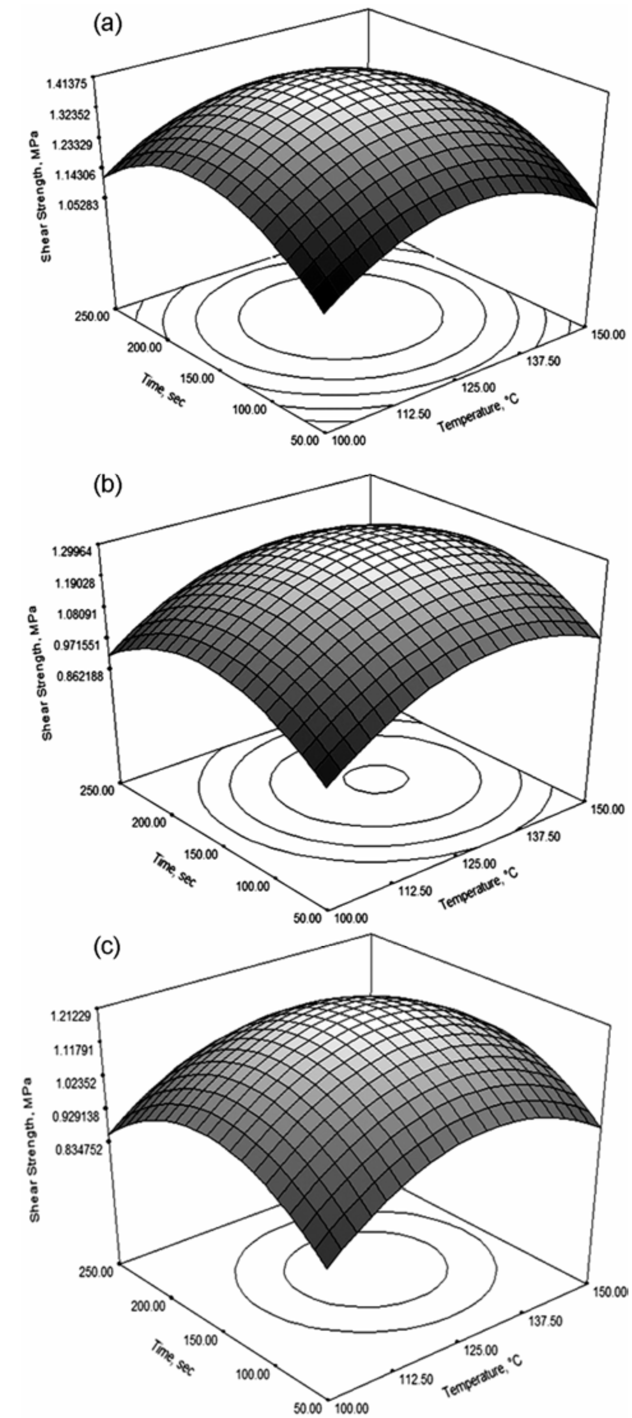

Fig. 2 — Three-dimensional response surface plot of shear strength for (a) PKM, (b) IF, (c) PS as filler.

highest value. The response surface in Fig. 2 (a) to (c) shows elliptical contours. These are obtained when there is a perfect interaction between the independent variables[19]. In this research, all variables studied were found to have synergistic on the shear strength performance in plywood application. The highest

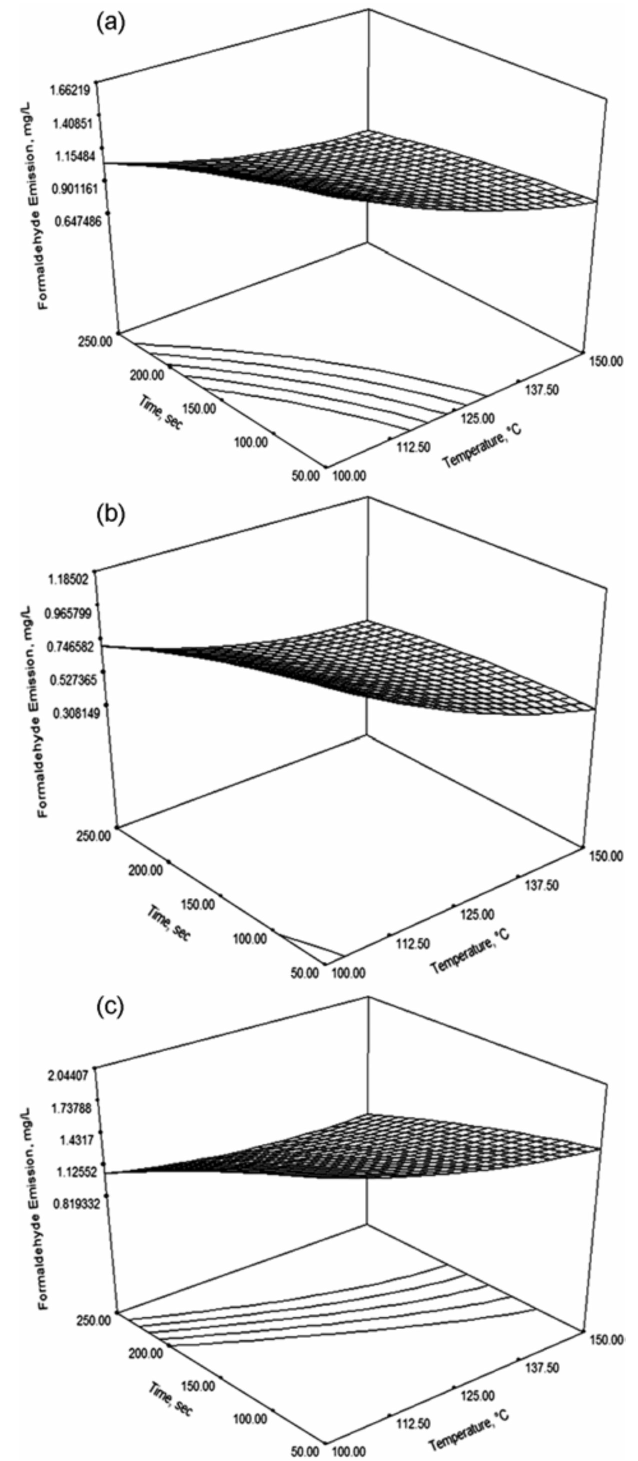

Fig. 3 — Three-dimensional response surface plot of formaldehyde emission for (a) PKM, (b) IF, (c) PS as filler.

shear strength performance value was obtained when both the variables (pressing time and temperature) were at the middle within the range studied. As shown by the analysis, PKM was shown greater result than IF, it's due to protein content of PKM was higher than IF. The shear strength value of IF was higher than PS. It indicated that filler with high protein content can enhance shear strength of the wood adhesive. This is due to the amino group $-NH_2$ inside the fillers enhanced the bonding formation between the wood adhesive and surface of veneer[2,7,8]. Therefore, plywood bonded with high protein content had greater shear strength value than plywood bonded with low protein content. Bono *et al*. (2011)[7] also found that the considerable amount of protein inside PKM can enhance the hydrogen bonding between wood adhesive and wood surface. The protein content of PKM and IF is 20 and 8% respectively[20,21].

In the formaldehyde emission performance (Y_2) of the fillers were found to have the greatest effect on overall process. The fillers effect, hot press temperature and time were studied. The three-dimensional parameter results shows in Fig. 3 (a) to (c). It was found that to decrease with increasing hot press temperature and time. The lowest response was obtained when hot press temperature and time were at the maximum point within the range of study. Besides that, IF has given lowest formaldehyde emission performance value among the fillers. The results obtained were in good agreement with works by Martins *et al*. (2007)[22]. He found that hot press temperature and time played an important role in the formaldehyde emission performance. The increase in a hot press temperature at fixed hot press time vice versa will significantly decrease the formaldehyde emission of the wood based panels. The filler with higher protein content did not show lower formaldehyde emission than others, while IF with less protein content compared to PKM has shown lower formaldehyde emission. This may be due to the nature of PKM, because the functional group did not effectively react with free formaldehyde in MUF resin.

Process optimization

The three level factorial designs have been used to optimize the parameters affecting the shear strength and formaldehyde emission test response. In the optimization analysis, the target criteria's were set as maximum and minimum values for shear strength and formaldehyde emission respectively. The optimum processing parameter provided by the model was shown in Table 4. The predicted, and experimental results of shear strength and formaldehyde emission obtained at optimum conditions were shown in Table 5. The optimum shear strength and formaldehyde emission performance of PKM, IF and

Table 4 — Optimum processing parameter on responses.

Temperature (°C)	Time (sec)	Fillers	Shear Strength, (MPa)	Formaldehyde Emission (mg/L)	Desirability
124.9	156	PKM	1.41	0.9988	0.877
130.9	153	IF	1.30	0.5345	0.694
127.9	149	PS	1.21	1.2735	0.552

Table 5 — Model Validation

Fillers	Shear Strength Performance (MPa)			Formaldehdye emission Performance (mg/L)		
	Predicted	Experimental	Error (%)	Predicted	Experimental	Error (%)
PKM	1.41	1.37	2.83	0.9988	0.9809	1.79
IF	1.30	1.28	1.54	0.5345	0.5275	1.31
PS	1.21	1.19	1.65	1.2735	1.2392	2.69

PS were 1.41 MPa, 1.30 MPa, 1.21 MPa and 0.9988 mg/L, 0.5345 mg/L, 1.2735 mg/L respectively. In addition, the optimum hot press temperature and time of PKM, IF and PS were 124.9°C, 130.9°C, 127.9°C and 156 s, 153 s, 149 s respectively. It was observed that the experimental values obtained were in good agreement with the value calculated from the models, with relatively small errors.

Conclusion

The study on the effect of plywood preparation variables on the shear strength and formaldehyde emission of plywood have been conducted by using three level factorial design. Through analysis of the response surface methodology, pressing temperature and different types of filler imposed the greater effect on the shear strength and formaldehyde emission performance. The optimum hot press temperature and time of PKM, IF and PS were 124.9°C, 130.9°C, 127.9°C and 156s, 153s, 149s respectively. It was observed that experimental values obtained were in good agreement with the value calculated from the models.

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