

OPTIMIZATION OF MATERIAL COMPOSITION FOR THE SYNTHESIS OF UREA FORMALDEHYDE (UF) RESIN MODIFIED BY POLYVINYL ALCOHOL (PVA)

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SUMMARY

Polyvinyl alcohol (PVA) was added to the synthesis of urea formaldehyde (UF) resin to reduce free formaldehyde (F) content, improve resin quality. The PVA-modified UF resin was synthesized in the laboratory. This modified resin was used as an adhesive for the production of plywood. Response surface methodology (RMS) was applied to optimize the amount of PVA addition and the molar ratio in the first stage of reaction process (F/U1) based on the quality criteria of the resin, including: solid content, curing time, water solubility, free formaldehyde (F) content and tensile shear strength, modulus of rupture, modulus of elasticity of plywood using modified resin to producing. The experiment was set with central composite design (CCD) for investigating the effects of the amount of PVA addition (1 – 3%) and molar ratio of F/U1 (1.8 – 2.0) on selected criteria. The results showed that the modified resin was optimal when using the amount of PVA = 1.6% of the total amount of urea and molar ratio of F/U1 = 1.91. The quality criteria achieved: solid content, curing time, water solubility, free formaldehyde content of the resin were 54.1%; 84 seconds; 3.2 times; 0.62%, respectively and tensile shear strength, modulus of rupture (MOR), modulus of elasticity (MOE) of the plywood were 2.09 MPa; 55.7 MPa; 9.1 GPa, respectively. **Keywords:** Modified resin, PVA, response surface methodology, UF.

1. INTRODUCTION

Urea formaldehyde (UF) resin is used widely in the manufacture plywood, medium density fiberboard (MDF), particleboard, and other non-structural wood products, with many advantages such as simple materials, fast curing, water solubility, low price and high bond strength. However, in composition of UF resin, there is always the content of free formaldehyde (F) - a compound that can cause cancer to humans and it can be emitted from pressed-wood products even under normal conditions (IARC 2004). To control the content of free F, many solutions have been applied successfully, such as lowering the molar ratio of F to urea (U), controlling reaction conditions, using additives to capture free F, introducing modifiers to react with F... (Thuan, 2021). Polyvinyl alcohol (PVA) is viscous, which improves the initial viscosity and bond strength of the resin (Pan et al., 2014). It has numerous hydroxyl groups, resulting in the reaction with F to form polyvinyl formal under acidic conditions; this reduces the content of

hydrophilic hydroxymethyl groups of UF resin and enhances the water resistance and the initial viscosity of the adhesive (Gungor and Kiskan 2018). In the published research results, PVA was added to the synthesis process to reduce free F content, creating modified UF resin with better performance than neat resin. The study by Liu et al., (2018) showed that a large amount of hydroxyl groups on the PVA chains reacted with F and acetalization occurred. The double networks of UF and PVA were formed simultaneously, interpenetrated with each other and thus the toughness of the blends was enhanced. Zhang et al., (2014) found that when the mass ratio of PVA/U was increased, the free F content of the UF resin was decreased, the storage stability was improved; the structure formed by the reaction of PVA with F increases the water solubility of the adhesive and captures residual F in the adhesive.

Besides, the crystallite regions related to the colloidal particle of the UF resin became more uniform by incorporation of the proper amount of PVA (with mass ratio of PVA/U = 0.005). Ya-san and Li-dan (2012) indicated that when

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the synthesis of UF resin was carried out under condition F/U = 1.87; pH = 4.5; T = 40°C and the amount of PVA = 0.2 g, free F content reached 0.64%, water resistance was 14 hours, good stability. The research using PVA to toughen urea-formaldehyde foam for thermal insulation applications (Shen et al., 2016) has reported that the best performance was obtained for PVA modified UF foam with 1.5% added PVA, low thermal conductivity similar to a polyurethane foam and no melt drops formed during burning.

The main materials to synthesize PVA-modified UF resin include U, F and PVA. The quality of modified resin depends mainly on the F/U molar ratio and the amount of PVA addition (Zhang Y and Snover DA, 2016). The influence of PVA on properties of the UF resin has been studied (Thuan et al., 2020a, 2020b). These results are important to supplement the theoretical basis in the studies of UF resin production technology in general and in particular with PVA modified UF resin. However, for the research to have more practical significance, it is necessary to propose the optimal technological parameters in the allowable conditions.

This work will inherit the research results of the previous works (Thuan et al., 2020a, 2020b) to determine the optimal parameters including: allocation proportion of PVA and F/U1 molar ratio (U1 - the number of molar of urea in the first stage) to synthesize PVA modified UF resin by applying response surface methodology (RMS) with central composite design (CCD).

2. RESEARCH METHODOLOGY

2.1. Materials

Veneer of *acacia hybrid* 1.5 mm thickness; urea (98%); formaldehyde solution (37%); polyvinyl alcohol (PVA), polymerization degree 1700, hydrolysis degree of 99%.

2.2. Experimental equipment

Automatic control resin synthesis

equipment; analytical balance TX-4202L; pH meter; drying cabinet; hot press machine BYD114 (College of Wood Industry and Interior Design, Vietnam National University of Forestry)

2.3. Preparation of test samples

- Resin preparation

F solution was poured into a three-necked flask and heated to 40°C. The pH of the reaction system was adjusted to 8.5 – 9.0 with NaOH 20% and the first part of urea (U1) was added (U1 changes according to experimental design). The temperature was kept at 40°C and stirred well for 10 minutes. PVA was added, heated up to 60°C (1 °C/min), stirred well for 15 minutes. The temperature was gradually increased to 90 – 92°C (2 °C/min), kept for 60 minutes. The second part of urea (U2) was added and kept for 15 minutes (U2 = 30% of the total amount of urea). The pH was adjusted to 5.0 – 5.5 with formic acid and condensation of the reaction system was continued until the turbidity point (white turbidity appears when the solution was dropped into water 45-55°C). When the turbidity point was reached, the pH was adjusted to 7.0 – 8.0. After that, the third part of urea (U3) was added and kept for 20 minutes (U3 changes according to U1). The pH was adjusted to 6.0 – 6.5 then the resin was taken out to measure the viscosity, when the viscosity reached 190 mPa.s, stopped heating. Finally, the pH value of the system was adjusted to 8.0 again, reduced the heat and the resin was taken out.

- Plywood preparation

Veneers of *acacia hybrid* had dimension of length x width x thickness = 300 mm x 300 mm x 1.5 mm; moisture reached 13 – 15%; veneers were peeled at the same wood peeler, with fixed peeling technological parameters, dried naturally. A calculated amount of the glue mix spread (150 g/m²) per face was spread onto five veneers to make 5-ply plywood. Pressing parameters were temperature (120°C); time (1.5

minutes/mm); pressure (1.2 MPa).

2.4. Design of experiments

Step 1: The factors and survey ranges were selected for optimization. The experiment was carried out with 2 factors: amount of PVA addition, X1 (1 – 3%); molar ratio of the first stage F/U1, X2 (1.8 – 2.0).

Step 2: The levels and design model were selected. RSM approach had been successfully

applied for identification of significant factors, modeling and optimization of experimental factors. Design Expert (DX) software was mostly used for graphical and regression analyses and analyses of the variance (ANOVA) of the obtained data. Each factor was surveyed with 5 levels (- α , -1, 0, +1, + α), calculated from running the software and the results were shown in Table 1

Table 1. Coded variables and survey levels

Independent variables	Code	Actual levels of coded variables				
		- α	-1	0	+1	+ α
The amount of PVA addition (%)	X ₁	0.6	1.0	2.0	3.0	3.4
Molar ratio of F/U1	X ₂	1.76	1.80	1.90	2.00	2.04

With 5 times of repetitions at the center and 2 studied factors, DX software designed

experiments randomly to get out 13 treatments as shown in Table 2

Table 2. Experimental design layout and experimental results of the responses (experiments were carried out according to the run order)

Run	PVA addition, %	Molar ratio of F/U1	Solid content, %	Curing time, sec.	Water solubility, time	Free F content, %	Tensile shear strength, MPa	Modulus of rupture, MPa	Modulus of elasticity, GPa
13	2.0 (0)	1.90 (0)	53.8	85.3	3.4	0.61	2.20	56.80	9.7
10	2.0 (0)	1.90 (0)	53.9	85.5	3.6	0.59	2.40	55.00	9.9
6	3.4 (+ α)	1.90 (0)	53.6	88.5	3.5	0.55	2.16	55.00	8.5
8	2.0 (0)	2.04 (+ α)	53.7	95.3	3.1	0.48	2.11	54.00	9.3
1	1.0 (-1)	1.80 (-1)	54.6	82.4	2.5	0.75	1.34	54.65	7.15
4	3.0 (+1)	2.00 (+1)	53.4	85.2	3.8	0.49	2.35	56.00	10.5
9	2.0 (0)	1.90 (0)	53.9	84.6	3.4	0.59	2.20	56.00	9.7
11	2.0 (0)	1.90 (0)	54.2	86.1	3.3	0.61	2.10	57.00	9.6
3	1.0 (-1)	2.00 (+1)	54.1	88.5	2.4	0.51	1.56	53.50	7.5
12	2.0 (0)	1.90 (0)	53.8	85.3	3.7	0.62	2.50	56.90	10
7	2.0 (0)	1.76 (- α)	53.8	101.2	1.8	0.68	0.94	55.94	7.2
5	0.6 (- α)	1.90 (0)	55.1	82.1	1.4	0.70	0.92	52.00	4.5
2	3.0 (+1)	1.80 (-1)	53.3	92.1	3.5	0.65	2.11	57.00	8.8

2.5. Data analysis

The experiments were carried out according to the design shown in Table 2. The desirability functions: solid content, curing time, water solubility, free F content, tensile shear strength, MOR, MOE were also collected according to designed experimental samples. DX software was used for analysis of variance (ANOVA)

with least significant difference (LSD) at significance 95%.

The fit of the predicted models for the desirability functions was evaluated through the coefficient R², the P-value of lack of fit and the adequate precision (AP). The predicted model for desirability functions was quadratic (equation 1).

$$Y = b_0 + b_1X_1 + b_2X_2 + b_{12}X_1X_2 + b_{11}X_1^2 + b_{22}X_2^2 \quad (1)$$

Where: Y are the desirability functions; b_0 - the constant coefficient; b_1, b_2 - the linear coefficients; b_{11}, b_{22} - quadratic coefficients; b_{12} - the interaction coefficient; X_1, X_2 are variables.

2.6. Test methods

For the resin: solid content, curing time and water solubility are determined according to the method specified in the Chinese standard GB/T 14732-2017. Free formaldehyde content was determined according to the method specified in the Vietnamese standard TCVN 11569:2016.

For the plywood: tensile shear strength (sampling and cutting of test pieces is carried out by TCVN 8328-1:2010, and testing the

tensile shear strength after the samples are conditioned, for the purpose of determining the tendency to change when the resin synthesis conditions change, in order to determine the technological parameters to synthesize PVA-modified UF resin); MOR and MOE applied the Vietnamese standard TCVN 7756-6 : 2007.

3. RESULTS AND DISCUSSION

3.1. Effect of material composition on some properties of modified UF resin and plywood

The ANOVA test was applied for the data in Table 2. The graph, contour, regression equation of desirability functions were shown (see Fig. 1, Table 3 and Fig. 2).

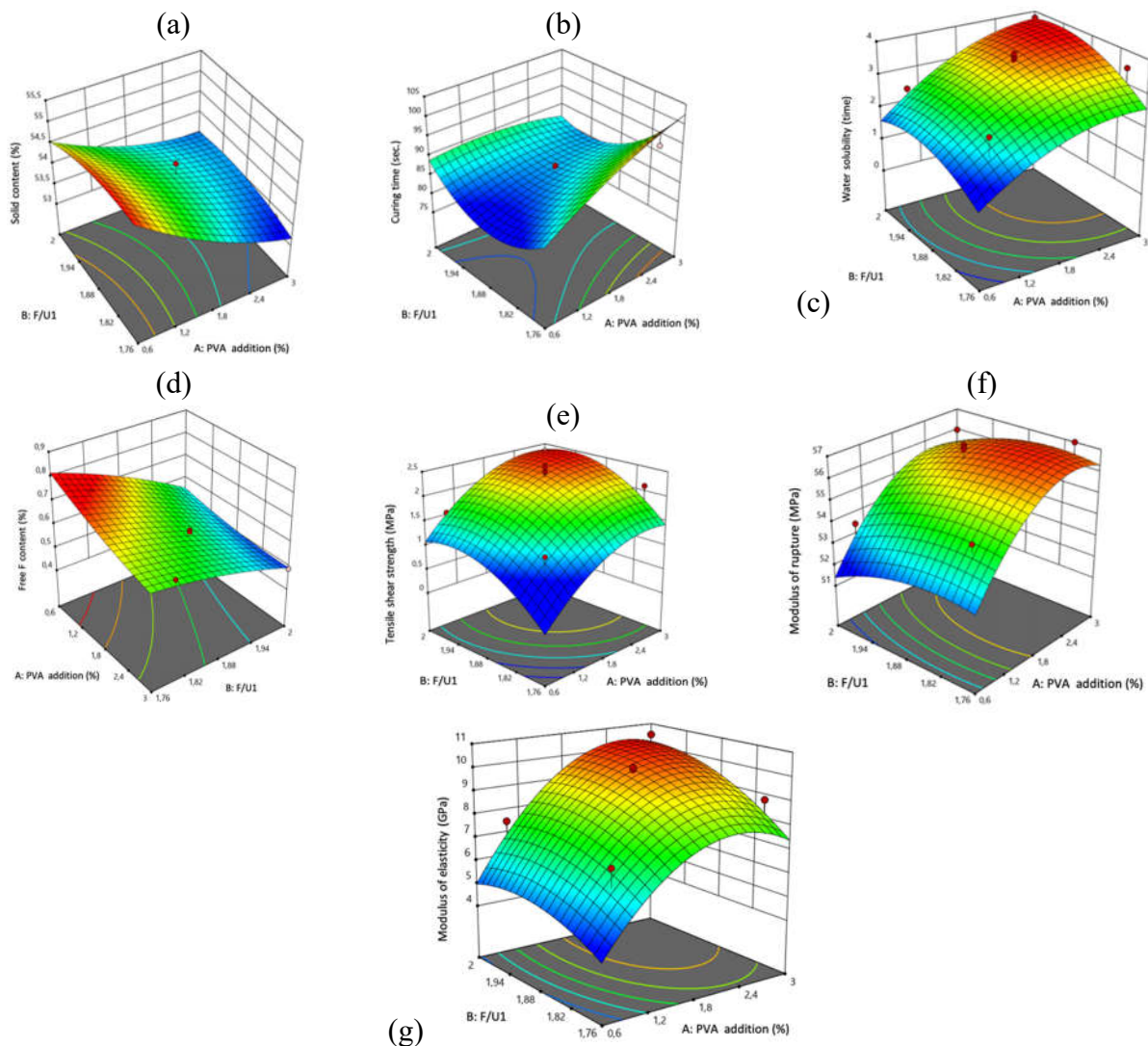


Fig. 1. Response surface and contour plot for the effect of the interaction between amount of PVA addition and molar ratio of F/U1 on the modified UF resin and plywood

Response surfaces, contour plots in Fig. 1 (a, b, c, d, e, f, g) show that amount of PVA addition and molar ratio of F/U1 both influence according to the quadratic model on the quality criteria of the resin and plywood.

The solid content tends to increase when the molar ratio of F/U1 increases from 1.76 to 1.85 and reaches the optimal value at F/U1 = 1.80 then decreases as F/U1 continues to increase to 2.0. The solid content decreases with increasing amounts of PVA addition. Solid content reaches the optimal value of 55.1%, corresponding to the amount of PVA addition = 0.6% and molar ratio of F/U1 = 1.80 (Fig. 1a).

In contrast, curing time tends to decrease when F/U1 increases from 1.76 to 1.9 and reaches the optimal value of 79.7 at F/U1 = 1.87 then increases when F/U1 continues to increase to 2.0. Curing time increases with increasing amounts of PVA addition. Curing time reaches the optimal value of 79.7 seconds corresponding to the amount of PVA addition = 0.6% and molar ratio of F/U1 = 1.87 (Fig. 1b).

The amount of PVA addition and molar ratio of F/U1 have a clear effect on water solubility according to the quadratic model. Water solubility tends to increase as F/U1 increases from 1.76 to 1.95 and reaches the optimal value at F/U1 = 1.94 then decreases as the molar ratio continues to increase to 2.0. The amount of PVA addition increases, the water solubility tends to increase. This criterion of the resin reaches the optimal value of 3.8 times, corresponding to the amount of PVA addition = 2.5% and F/U1 = 1.94 (Fig. 1c).

Meanwhile, the response surface and contour plots in Fig. 1d shows that the free F content tends to decrease with increasing F/U1 and the amount of PVA addition. Free F content reaches the optimal value of 0.49%, corresponding to the amount of PVA addition = 2.5% and the molar ratio of F/U1 = 2.0 (Fig. 1d).

The amount of PVA addition and molar ratio of F/U1 influence according to the quadratic

model on the quality criteria of plywood. Fig. 1e indicates that the tensile shear strength tends to increase when F/U1 increases from 1.76 to 1.95 and reaches the optimal value at F/U1 = 1.94 then decreases as the molar ratio continues to increase to 2.0. Tensile shear strength also tends to increase as the amount of PVA addition increases, and reaches the optimal value of 2.47 MPa corresponding to PVA addition = 2.5% and F/U1 = 1.94. Similarly, MOR tends to increase as F/U1 increases from 1.76 to 1.85 and reaches the optimal value at 1.83 then decreases as the molar ratio continues to increase to 2.0. MOR also tends to increase with increasing the amount of PVA addition from 0.6% to 2.5%, then decreases with continued increase of the amount of PVA addition to 3%. MOR reaches the optimal value at 56.83 MPa corresponding to PVA addition = 2.5% and F/U1 = 1.83 (Fig. 1f). In Fig. 1g, MOE tends to increase when F/U1 increases from 1.76 to 2.0 and reaches the optimal value at 1.98. MOE also tends to increase with increasing amounts of PVA addition to 2.5% then decreases as PVA addition continues to increase by 3%. MOE reaches the optimal value of 10.4 GPa, corresponding to the amount of PVA addition = 2.5% and molar ratio of F/U1 = 1.98.

The above results show that, when the resin synthesis using PVA addition exceeds 2.5% and the molar ratio F/U1 exceeds 2, most of the quality criteria of the resin are reduced because the PVA increases, the activity of functional groups is decreased, so the degree of reaction of compounds is reduced.

According to Liu et al. (2018), by introducing PVA, in addition to F reacting U to form UF networks, F also reacts with PVA to form UF/PVA interpenetrating networks. The PVA macromolecules tightly embedded in the UF network, enhancing the mutual entanglement between the two networks, and the degree of crosslinking within the blends was thus improved.

However, the amount of PVA addition is too excessive (> 2.5%), the PVA reacts more with F causing the lack of F to react with U. The hydroxyl groups of excessive PVA do not participate in reaction and they absorb water, reducing the amount of osmotic adhesives into layers of veneers.

Similarly, when the molar ratio of F/U1 is larger 2, the remaining F of the condensation

stage is less (due to the total amount of F being constant), decreased reaction degree, formation of loose network structure, and a small amount of product in the condensation reaction.

The ANOVA results show that the correlation model built by the linear, interaction and quadratic coefficients of the two studied factors affected the quality criteria of the resin and plywood (Table 3).

Table 3. ANOVA results and linear, interaction, quadratic coefficients of the regression equations to predict the values of the criteria

Response variables	Source								
	Intercept	A	B	AB	A ²	B ²	LOF	R ²	AP
Solid content	53.92	-0.51516	-0.06767	0.15	0.165	-0.135			
p-values		< 0.0001	0.2927	0.1178	0.0361	0.0721	0.4421	0.9295	14.2242
Curing time	85.36	1.93137	-1.14298	-3.25	-1.21125	5.26375			
p-values		0.0894	0.2817	0.0515	0.2870	0.0016	0.0009	0.8483	9.1847
Water solubility	3.46	0.671231	0.25481	0.1	-0.355	-0.355			
p-values		0.0029	0.1342	0.6527	0.0637	0.0637	0.0146	0.8282	7.0103
Free F content	0.604	-0.04151	-0.08535	0.02	0.00987	-0.012625			
p-values		0.0012	< 0.0001	0.1172	0.2828	0.1806	0.0733	0.9557	19.0703
Tensile shear strength	2.28	0.41420	0.26432	0.005	-0.29312	-0.30062			
p-values		0.0029	0.0245	0.9706	0.0214	0.0192	0.0874	0.8627	7.6267
Modulus of rupture	56.34	1.13658	-0.61169	0.0375	-1.15687	-0.42187			
p-values		0.0073	0.0842	0.9330	0.0094	0.2368	0.4556	0.8173	6.9370
Modulus of elasticity	9.78	1.28836	0.62748	0.3375	-1.36188	-0.48687			
p-values		0.0007	0.0266	0.3225	0.0008	0.0826	0.0027	0.9154	11.4889

Note: A – amount of PVA addition; B – molar ratio of F/U1

According to Zabeti et al., (2009), a good correlation model needs to have the fit between the actual value and the predicted value, where lack of fit (LOF) p-value > 0.05 is not significant and adequate precision (AP) > 4. In addition, the correlation coefficient R² > 0.8 (Guan X and Huiyuan Y (2008)). Accordingly, the LOF tests with low p-value (curing time, water solubility and MOE, p-value of less than 0.05) indicate that there is a lack of fit. In other

words, the LOF errors are significantly larger than the random or pure error. However, the LOF test is based on the residuals. Moreover, the coefficient of determination R² and AP of all models met the necessary conditions well (R² ≥ 0.8; AP ≥ 6.9). Thus, the models have enough reliability to predict the correlation of factors with product quality criteria, and the actual values are sufficiently close to the predicted values (Fig. 2).

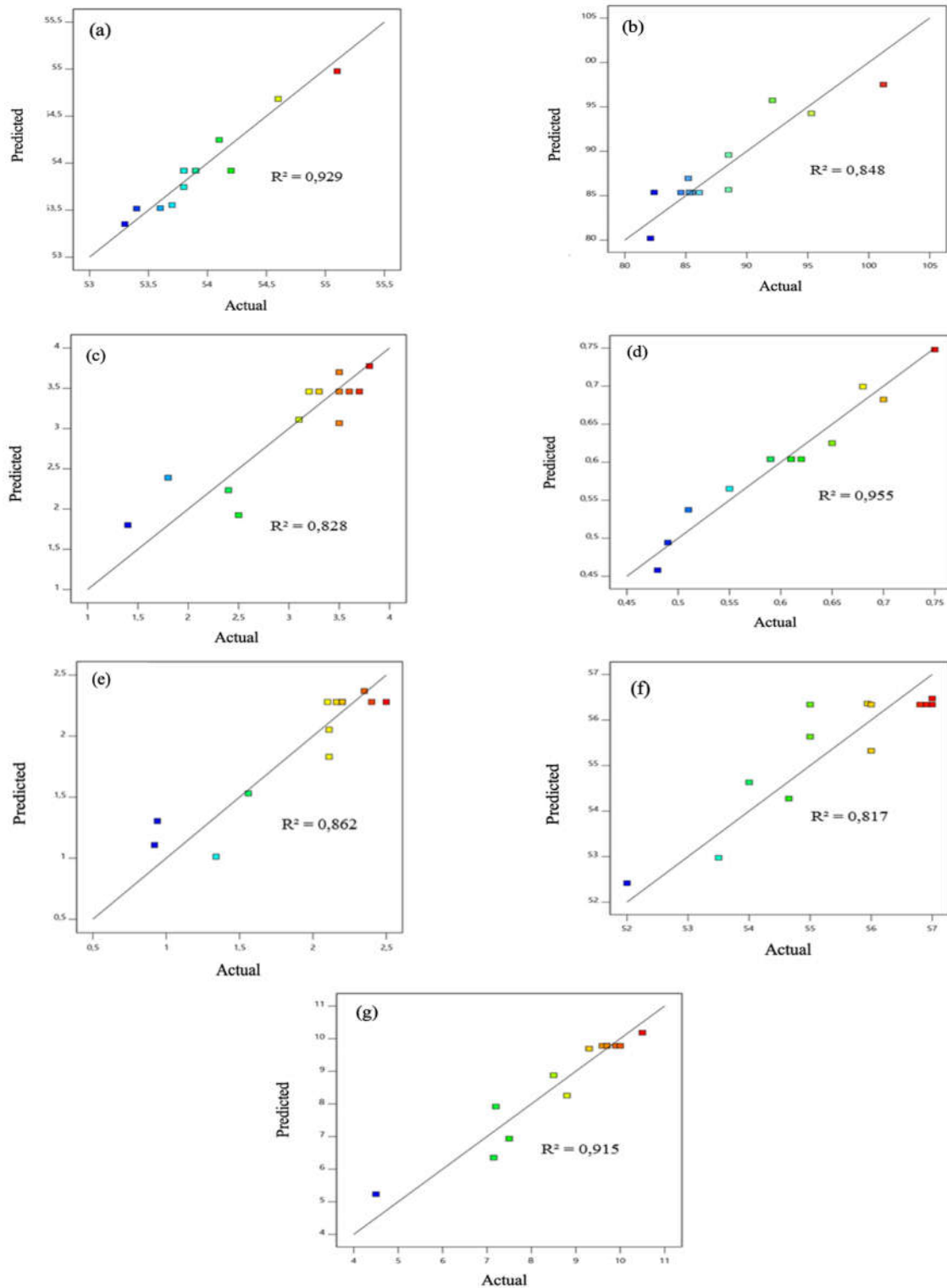


Fig. 2. The predicted values versus the experimental values for (a) solid content, (b) curing time, (c) water solubility, (d) free F content, (e) tensile shear strength, (f) MOR, (g) MOE

3.2. Optimization of synthesis conditions

Optimization of multiple responses can be a key point for industrial applications, especially resin performance is significantly increased when the material composition is optimized. This was done based on the desirability function

methodology. This method incorporates desired values and priorities for each variable. After optimizing the amount of PVA addition and F/U1 molar ratio for each response, the results are presented in Table 4.

Table 4. Optimal processing parameter on each response

No.	Desirability functions	Optimal values	PVA addition, %	Molar ratio of F/U1
1	Solid content, %	55.1	0.6	1.80
2	Curing time, sec.	79.7	0.6	1.87
3	Water solubility, time	3.8	2.5	1.94
4	Free F content, %	0.49	2.5	2.00
5	Tensile shear strength, MPa	2.47	2.5	1.94
6	Modulus of rupture, MPa	56.83	2.5	1.83
7	Modulus of elasticity, GPa	10.4	2.5	1.98

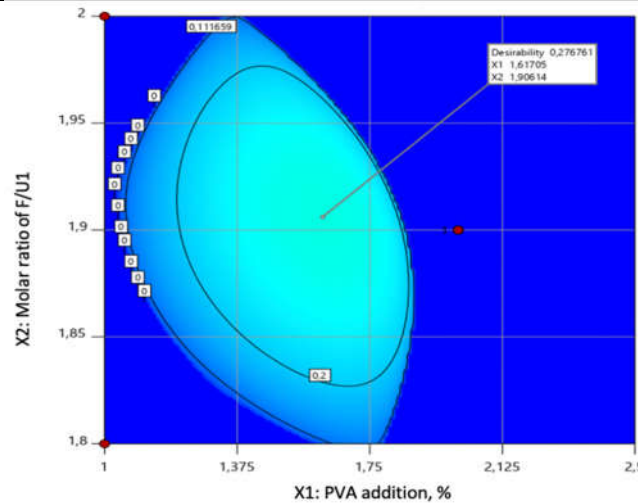


Fig. 3. Contour plot for simultaneous optimization of multiple responses

All response variables investigated were found to be highly dependent on the independent variables, with strong interactions observed between the independent variables. It was found that optimum overall desirability of

material composition could be obtained at the amount of PVA addition 1.6% and F/U1 molar ratio 1.91 (see fig. 3). The value of desirability functions are presented in Table 5.

Table 5. Optimal processing parameter on all responses

No.	Desirability functions	Optimal values	PVA addition, %	F/U1 molar ratio
1	Free F content, %	0.62		
2	Tensile shear strength, MPa	2.09		
3	Solid content, %	54.10		
4	Curing time, sec.	84.00	1.60	1.91
5	Modulus of elasticity, GPa	9.10		
6	Water solubility, l/ân	3.20		
7	Modulus of rupture, MPa	55.70		

4. CONCLUSION

The study found the optimal parameter of material composition for desirability functions (solid content, curing time, water solubility, free F content, tensile shear strength, MOR, MOE), including the amount of PVA = 1.6% compare to the total amount of urea and molar ratio in the first stage of reaction process F/U1 = 1.91. With this material composition, the modified resin

has the most suitable solid content, curing time and water solubility, and the smallest free F content. When using this type of resin to produce plywood, the product obtains the highest tensile shear strength, MOR, MOE. This optimized PVA-modified UF resin has good quality for a class E1 resin of 0.11 mg/m³ (the value measured by the chamber method, class E1 requirement ≤ 0.124 mg/m³ air).

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TỐI ƯU HÓA TỶ LỆ THÀNH PHẦN NGUYÊN LIỆU TỔNG HỢP KEO UREA FORMALDEHYDE BIẾN TÍNH BẰNG POLYVINYL ALCOHOL (PVA)

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TÓM TẮT

Polyvinyl alcohol (PVA) được thêm vào quá trình tổng hợp keo urea formaldehyde (UF) để giảm hàm lượng formaldehyde (F) tự do, cải thiện chất lượng keo. Keo UF biến tính bằng PVA được tổng hợp trong phòng thí nghiệm. Keo biến tính này được sử dụng làm chất kết dính để sản xuất ván dán. Sử dụng phương pháp bề mặt đáp ứng (RSM) để tối ưu hóa lượng dùng PVA và tỷ lệ mol trong giai đoạn phản ứng cộng F/U1 của quá trình tổng hợp keo UF dựa vào các chỉ tiêu chất lượng của dung dịch keo gồm: hàm lượng khô, thời gian đóng rắn, độ tan trong nước, hàm lượng formaldehyde (F) dư trong keo và độ bền kéo trượt, độ bền uốn tĩnh, mô đun đàn hồi uốn tĩnh của gỗ dán sử dụng keo biến tính để sản xuất. Thí nghiệm được thiết kế theo phương pháp phối hợp có tâm (CCD) nhằm khảo sát ảnh hưởng của lượng dùng PVA (1 - 3%) và tỷ lệ mol F:U1 (1,8 – 2,0) đến các chỉ tiêu được chọn. Kết quả nghiên cứu cho thấy, keo biến tính tổng hợp đạt tối ưu khi dùng lượng PVA = 1,6% so với tổng lượng urea và tỷ lệ mol F:U1 = 1,91. Hàm lượng khô, thời gian đóng rắn, độ tan trong nước, hàm lượng F dư của keo lần lượt là 54,1%; 84 giây; 3,2 lần; 0,62% và độ bền kéo trượt màng keo, độ bền uốn tĩnh, mô đun đàn hồi uốn tĩnh của ván dán lần lượt là 2,09 MPa; 55,7 MPa; 9,1 GPa.

Từ khóa: Keo biến tính, phương pháp bề mặt đáp ứng, PVA, UF.

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