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## Response Surface Methodology as an Approach to Optimize the Preparation of Maize Straw Panels Applying Soy-based Adhesives

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# Response Surface Methodology as an Approach to Optimize the Preparation of Maize Straw Panels Applying Soy-based Adhesives

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**Abstract.** In this study, maize straw was used to prepare formaldehyde-free panels with soy-based adhesives. Influences of adhesive amount, hot-pressing time, temperature and pressure on the internal bonding strength were optimized by applying response surface methodology (RSM). The results showed that based on experimental mathematical model, the optimum hot-pressing conditions were obtained. Adhesive amount was 14%, hot-pressing pressure was 3.0 MPa, hot-pressing temperature was 126 °C, hot-pressing time was 28 s/mm. The actual internal bonding strength was 0.52 MPa while the predicted value was 0.51 MPa, indicating that the experimental mathematical model was accurate and reliable.

## 1. Introduction

Straw is an important kind of renewable resources in agricultural production. It is estimated that the production amount of straw is nearly two billion ton per year around the world [1, 2]. China, as a vast country of agriculture, one of the countries with the most abundant resources of crop straw, produces over 0.64 billion ton per year. Crop straw is valuable renewable resource, which plays a key role in stabilizing the balance of agriculture ecosystem and promoting sustainable development to accelerate the comprehensive utilization. Moreover, the utilization of straw resources is involving thousands of households and the sustainable development problems in agricultural production, such as soil fertility, soil and water conservation, environmental security of the rural and so on, as well as how to reasonably make full use of straw resources with higher value and higher efficiency [3]. However, the outer surface of maize straw cells including silicon cells and plug cells has dense epidermis, which are composed of SiO<sub>2</sub> and wax [4, 5]. Because of mineralization and suberization, these layers forming around the epidermis make the adhesives difficult to penetrate the outer layer of straw cells. In addition, internal macromolecular reaction ability is decreased greatly, which is difficult to form effective internal bonding during the curing process and directly influence the internal bonding strength. In addition, there is rising demand of formaldehyde-free panels with the concerning about the detriment of formaldehyde emission. Thus, the prospect and application of formaldehyde-free soy-based adhesives as the materials of indoor panels preparation is promising.



In this study, formaldehyde-free panels were prepared by applying maize straw and soy-based adhesives as the main materials. By using response surface method (RSM) to optimize processing conditions, mathematics model between the internal bonding strength and hot-pressing process was established. The optimal parameters for maize straw panels were obtained, which provided theoretical basis and the reference for further utilization of both high value and high efficiency.

## 2. Materials and Method

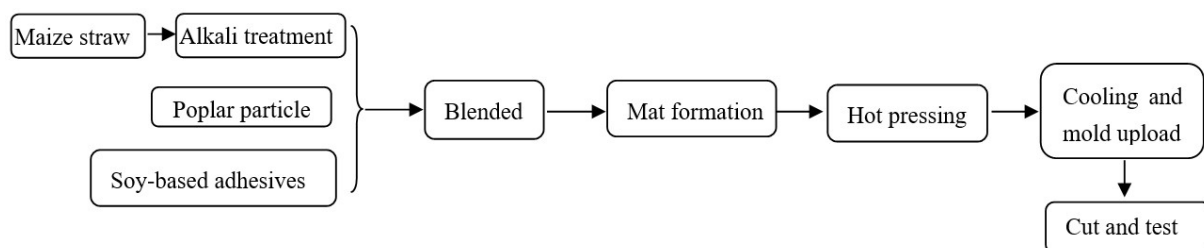
### 2.1. Materials

1) Maize straw. The material of maize straw was collected from local peasant household with moisture content around 10%. It was crushed to the size of 0.5 cm~4 cm by a small grinder. The material was treated in the NaOH solution with 8% concentration at 60 °C for 50 min. After treatment, the moisture content of the material was oven-dried to 3%~5%. The moisture content of poplar panels was 8%~12% with particle size of 0.5 cm~4 cm.

2) Preparation of soy-based adhesives. The defatted soybean flour was hydrolyzed and degraded by alkaline method. Then the adhesive was prepared by mixing 75% of soy-based adhesive, 20% of waterborne polyurethane, 3% of waterproof agents and 2% of preservative. And the mixture was stirred for 30 min at room temperature.

### 2.2. Method

2.2.1. *Panels preparation procedure.* The dimension of panels was 400 mm×400 mm×10 mm. The preparation procedure of maize straw-based panels was shown in Figure 1.



**Figure.1** Production scheme of maize straw panels

2.2.2. *Parameters of hot-pressing.* The main factors on effecting the bonding strength of panels are adhesive types, the proportion of adhesives, hot-pressing pressure, temperature and time. Compared with traditional formaldehyde adhesives, the solid content of soy-based adhesive is lower whilst adhesive amount is higher. In addition, the longer the hit-pressing time, the more beneficial the curing process. What's more, it is averse to curing process with temperature too high that can induce over denature of soy-protein. Hot pressing and hot-pressing time also influence the curing process of adhesives. Therefore, this study selected four factors, including the adhesive amount, hot-pressing pressure, hot-pressing temperature and hot-pressing time. Based on the early research<sup>[6]</sup>, the conditions of appropriate adhesive amount, hot pressing pressure, hot pressing temperature and time were 10%~18%, 2.0 MPa~4.0 MPa, 20 s/mm~40 s/mm, 110 °C~130 °C respectively, while the internal bonding strength (IB) was used as response value. The bonding strength test of samples were carried out according to the method stipulated by the national standard GB/T 17657-2013.

2.2.3. *Experimental design scheme of hot-pressing process.* This experiment selected the adhesive amount, hot-pressing pressure, hot-pressing temperature and hot-pressing time as the factors, with +1, 0, -1 on behalf of high, middle and low level, respectively. And the encode of the experiment were shown in Table 1. Using Box-Benhknken experimental design principle, the internal bonding strength

(IB) was assigned as the response value with the Design-Expert 10.0.2 software design of four factors and three levels of response surface analysis experiment. There were 29 experiments. In this experiment, two-polynomial mathematical model was established and the maximum internal bonding strength was obtained by analyzing the experimental results.

**Table 1.** The response surface of level of factors

Factors	Level		
	-1	0	+1
X <sub>1</sub> Adhesive amount(%)	10	14	18
X <sub>2</sub> Pressure(MPa)	2.0	3.0	4.0
X <sub>3</sub> Temperature(°C)	110	120	130
X <sub>4</sub> Time(s/mm)	20	30	40

### 2.3. Verification of hot pressing process

To validate the optimal conditions and the corresponding predicted value by RSM, three repeated experiments under the optimal conditions were carried out and its average value was used as the results to validate the reliability of the optimization model.

## 3. Results and Discussion

### 3.1. Variance analysis of response surface experiment

The response surface design and experimental results are shown in Table 2. An analysis of the experimental results with Design-Expert 10.0.2 software was implemented. And the results of variance analysis were shown in Table 3. As could be seen in Table 3 of the variance analysis, the influence of X<sub>1</sub>, X<sub>3</sub>, X<sub>4</sub>, X<sub>1</sub>X<sub>3</sub>, X<sub>3</sub><sup>2</sup> and X<sub>4</sub><sup>2</sup> on the internal bonding strength (IB) was significant, indicating that the adhesive amount, hot pressing temperature and time were important factors controlling the internal bonding strength (IB). In addition, the interaction effect between adhesive amount and hot-pressing temperature was significant, while it was not significant among other factors.

**Table 2.** The experiment results of response surface

NO.	Glue amount (X <sub>1</sub> , %)	Pressure (X <sub>2</sub> , MPa)	Temperature (X <sub>3</sub> , °C)	Time (X <sub>4</sub> , s/mm)	IB (Y, MPa)
1	0	1	1	0	0.49
2	0	0	0	0	0.55
3	0	-1	0	1	0.45
4	0	1	-1	0	0.38
5	0	1	0	-1	0.31
6	0	0	-1	-1	0.27
7	0	0	-1	1	0.46
8	-1	0	-1	0	0.28
9	1	-1	0	0	0.29
10	0	-1	1	0	0.51
11	-1	0	1	0	0.24
12	-1	-1	0	0	0.19
13	0	0	0	0	0.51
14	0	0	1	-1	0.42
15	0	0	0	0	0.5
16	1	0	0	1	0.35
17	0	0	0	0	0.51
18	-1	1	0	0	0.24
19	0	0	1	1	0.48
20	0	-1	-1	0	0.38
21	-1	0	0	1	0.31
22	0	-1	0	-1	0.36
23	0	1	0	1	0.38
24	-1	0	0	-1	0.17
25	1	0	1	0	0.44
26	1	0	0	-1	0.23
27	0	0	0	0	0.54
28	1	1	0	0	0.41
29	1	0	-1	0	0.21

As shown in Table 3, the effect of hot pressing process upon the internal bonding strength (IB) according to priority was hot-pressing time, hot-pressing temperature, adhesive amount and hot-pressing pressure. The meaning of lack of fit is that the experimental data is not in accordance with the model. And the value of the lack of fit in this study was 0.0989 without great significance, illustrating that there was no abnormal point in the data which was accurate and reliable.

**Table 3.** The variance analysis of response surface

Sources	Sum of squares	df	Mean square	F	<i>p</i>	significance
Model	0.35	14	0.025	17.12	< 0.0001	**
X <sub>1</sub>	0.021	1	0.021	14.27	0.0020	**
X <sub>2</sub>	7.4×10 <sup>-5</sup>	1	7.5×10 <sup>-5</sup>	0.051	0.8240	
X <sub>3</sub>	0.04	1	0.03	20.55	0.0005	**
X <sub>4</sub>	0.027	1	0.037	25.62	0.0002	**
X <sub>1</sub> X <sub>2</sub>	1.225×10 <sup>-3</sup>	1	1.226×10 <sup>-3</sup>	0.84	0.3752	
X <sub>1</sub> X <sub>3</sub>	0.018	1	0.018	12.48	0.0033	**
X <sub>1</sub> X <sub>4</sub>	1.0×10 <sup>-4</sup>	1	1.0×10 <sup>-4</sup>	0.068	0.7973	
X <sub>2</sub> X <sub>3</sub>	1.0×10 <sup>-4</sup>	1	1.0×10 <sup>-4</sup>	0.068	0.7973	
X <sub>2</sub> X <sub>4</sub>	1.0×10 <sup>-4</sup>	1	1.0×10 <sup>-4</sup>	0.068	0.7973	
X <sub>1</sub> <sup>2</sup>	1.0×10 <sup>-4</sup>	1	1.0×10 <sup>-4</sup>	0.068	0.7973	
X <sub>2</sub> <sup>2</sup>	4.225×10 <sup>-3</sup>	1	4.225×10 <sup>-3</sup>	2.89	0.1110	
X <sub>3</sub> <sup>2</sup>	0.22	1	0.22	151.66	< 0.0001	**
X <sub>4</sub> <sup>2</sup>	0.02	1	0.02	13.93	0.0022	**
Residue	0.02	14	1.46×10 <sup>-3</sup>			
Lack of fit	0.019	10	1.856×10 <sup>-3</sup>	3.95	0.0989	
Pure error	1.88×10 <sup>-3</sup>	4	4.7×10 <sup>-4</sup>			
Total	0.37	28				
R <sup>2</sup>	0.9448					

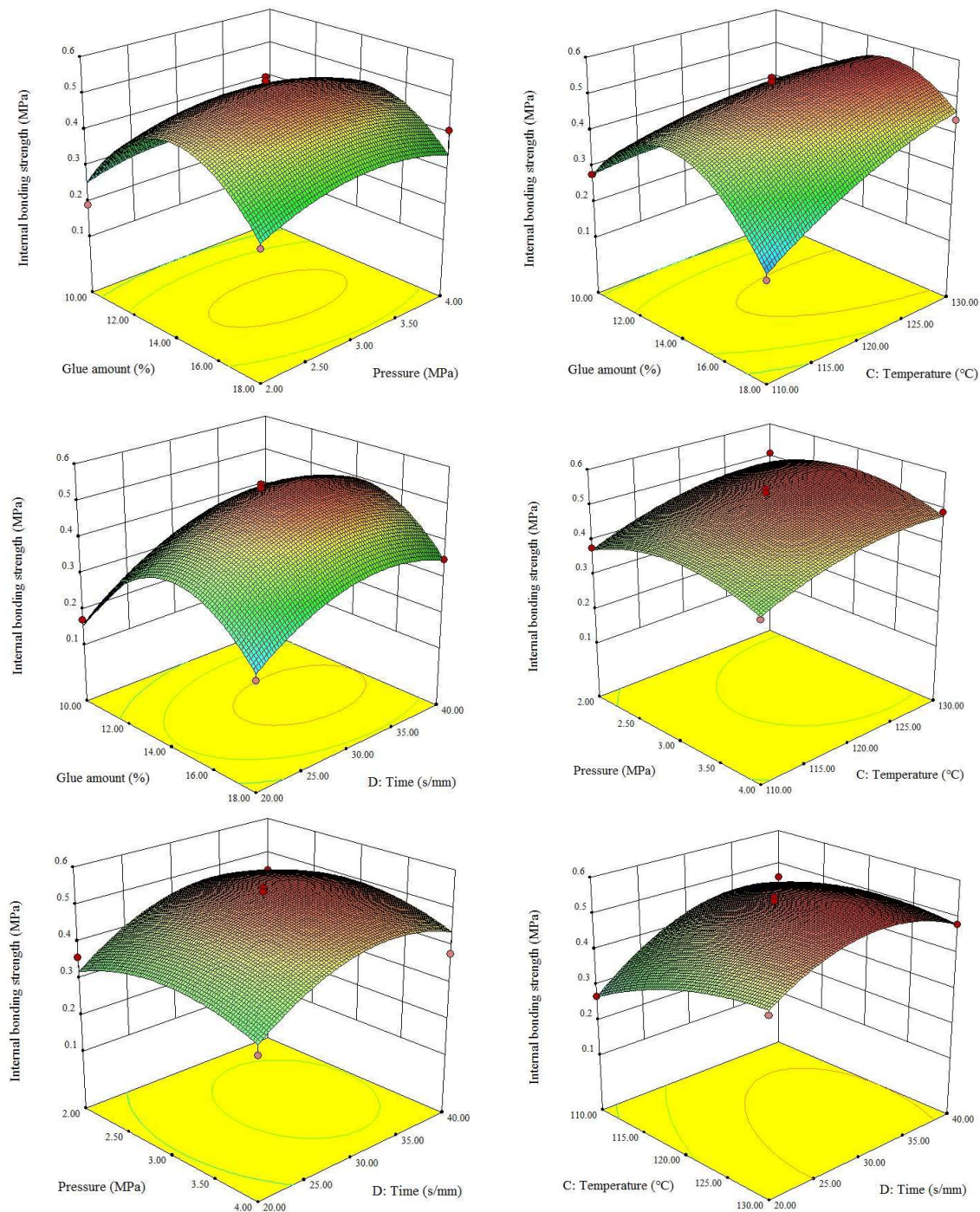
A two-regression analysis of the experimental data was carried out by using Design-Expert 10.0.2 software, and the regression equation of the internal bonding strength (IB) of Y was obtained by polynomial fitting of four factors:

$$Y = 0.62 + 0.042X_1 + 2.5 \times 10^{-3}X_2 + 0.050X_3 + 0.056X_4 + 0.017X_1X_2 + 0.067X_1X_3 - 5 \times 10^{-3}X_1X_4 - 5 \times 10^{-3}X_2X_3 - 5 \times 10^{-3}X_2X_4 - 0.033X_3X_4 - 0.18X_1^2 - 0.056X_2^2 - 0.035X_3^2 - 0.081X_4^2$$

The R<sup>2</sup> value of the model was 0.9448, indicating that the model equation could explain the variation of the internal bonding strength (IB) in 94.48% of the panels, which demonstrated that the equation was well fitted [7-9].

### 3.2. Analysis of main factors influencing on internal bonding strength

The effects of cross interaction of different factors were illustrated in Figure 2. It could be seen that each graph presented one analysis of the interaction between two different factors, while the other two factors were fixed at zero levels. Figure 2 clearly showed the influence of various factors on the internal bonding strength (IB) in response value. The steeper or flatter the trend of the response surface, the more obvious or smaller the influence of the factors. The interaction curve trend of adhesive amount and hot-pressing temperature was the steepest shown in Figure 2, demonstrating that the influence of interaction among adhesive amount and hot-pressing temperature was the most significant, which was corresponding with the *p* value of X<sub>1</sub>X<sub>3</sub> shown in Table 3. What's more, the trend curve of hot-pressing time, hot-pressing pressure and hot-pressing temperature was flat relatively, indicating that the interactions were not significant among hot-pressing time, hot-pressing pressure and hot-pressing temperature.



**Figure 2.** Response surface plots for the effects of cross interactions among the factors

### 3.3. Determination and verification of optimal hot-pressing process

By solving the equation of the internal bonding strength (IB) (Y value), the prediction value of the internal bonding strength (IB) was 0.51 MPa with the corresponding conditions as follows: 14.42%, 3.32 MPa, 125.76 °C, 27.94 s/mm represented adhesive amount, hot-pressing pressure, hot-pressing temperature and hot-pressing time respectively. According to the optimal predictive parameters, the optimal conditions can be chosen as 14%, 3.0 MPa, 126 °C, and 28 s/mm. Under the condition, the experiment was repeated for three times to test the internal bonding strength (IB) of the panels to validate

the feasibility of RSM model. The result showed that the average of the internal bonding strength (IB) value of the three repeated experiments was 0.52 MPa, which was close to the predictive value, indicating that the preparation process with response surface optimization was feasible.

#### 4. Conclusion

This study was based on soy-based adhesives applied to maize straw particleboard preparation, using RSM for hot-pressing process optimization. Through the establishment of internal bonding strength with the two multivariate regression model with the factors including adhesive amount, hot-pressing pressure, hot-pressing temperature and hot-pressing time, the optimum conditions of hot pressing process were 14%, 3.0 MPa, 126 °C, 28 s/mm respectively. Moreover, optimal prediction value was 0.51 MPa. Through experimental verification, three parallel experiments implemented under the predictive condition, the average value was 0.52 MPa, which was close to the actual predictive value and showed that the model was reasonable and reliable.

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