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Research Article

Preparation of Reed Straw-Based Panels Bonded by Soy-Based Adhesives: Optimization via Response Surface Methodology

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The optimization of manufacturing conditions for reed straw-based particleboard by soy-based adhesive was performed through response surface methodology. The interactions of various conditions, including adhesive amount, hot-pressing temperature, and hot-pressing time on wet internal bonding strength were investigated. A 3-level-3-factor Box–Behnken design was used to test the optimal preparation conditions of reed straw particleboard. The polynomial regression model for manufacturing conditions had a very significant level ($p < 0.01$). In addition, the determination coefficient (R^2) and the adjust determination coefficient ($\sqrt{R^2}$) of this model were found to be 0.969 and 0.9292, respectively. The conditions optimized by the model were 25% of adhesive amount, 138°C of hot-pressing temperature, and 27 min of hot-pressing time. Under the optimal conditions, validation tests were performed, and the average value of parallel experiments was 0.17 ± 0.02 MPa. Moreover, the thickness swelling of water absorption after soaking and mechanical properties (MOE and MOR) of samples prepared under optimized conditions were further measured, which all met the requirement of Type P6 particleboard. It could provide an efficient method for massive production of reed straw particleboard.

1. Introduction

The overall consumption of wood-based panels, including plywood, hardboard, oriented strand board, and medium-density fiberboard has grown rapidly throughout the world from the last decade. Particleboard is considered as one of the world's favorite wood-based panels owing to low expense and excellent processing performance [1]. The consumption of particleboard is more than the half of the whole wood-based panel consumption, and the market demand for particleboard is continuously increasing from 2 to 5% annually [2, 3]. The applications of particleboard are mainly housing construction, interior decoration, and furniture manufacturing. In addition, due to severe deforestation and huge demand for wood-based boards, the wood resources around the world are continuously decreasing. The issue how to sustainably supply wood for this demand has been raised. Consequently, it is crucial to find an alternative fiber in particleboard manufacturing because of these concerns.

A series of nonwood plants and agricultural and forestry wastes have been developed as the raw materials of particleboard, such as wheat straw [4, 5], corn pith [6], cotton stalks [7], rice husk [8, 9] and rice straw [10], waste grass clippings [11], branch wood and bark [12], reed straw [13], and bagasse [14], which can all be employed as alternative materials. All these wastes can be used as the raw materials for adhesives, bioethanol, chemicals, and particleboard [15, 16]. However, among all these usages, preparation of particleboard is the most economic and environment-friendly process because of zero release of carbon and other wastes. Reed (*Phragmites communis* (Cav.) Trin. ex Steud.) straw as a nonwood material is one of the most prospective alternatives for particleboard manufacturing. Reed is a species of grass that is widely distributed in the world currently used for papermaking [17]. However, there is large amount of wastewater discharged during paper production, which cannot be carbon-neutral and environment-friendly. Reed straw can be an interesting alternative for particleboard manufacturing owing to its similar composition with wood but different in proportions [13]. It mainly contains 35–45% cellulose, 25–30% hemicellulose, 15–20% lignin, and 15–17% amorphous silica and waxes [18, 19], which is suitable for particleboard production. At present, the consumption of urea formaldehyde resins for the manufacturing of particleboards has accounted for approximately 90% [20]. Though these resins are economical with excellent adhesion properties, they are petroleum-based and nonrenewable. In addition, it can release formaldehyde during the production process and product applications, which pose health hazards to workers in the particleboard manufacturing and end use consumers. Due to these facts, many countries are tending to gradually eliminate the use of formaldehyde-based resins or, at least, reduce the formaldehyde emission from these resins. Undoubtably, this could encourage the development of environment-friendly adhesives.

Wood adhesives derived from biomaterials as the substitutes, including starch [21], plant proteins, sucrose [22, 23], tannin [24], and lignin [25], have been reported extensively in the past decades. These adhesives can be used as the synthetic adhesives without formaldehyde in their formulations. Soy protein as one of these renewable materials is more suitable for the preparation of wood adhesive due to its low price and easy availability. Moreover, the preparation process of soy-based adhesive is also proved to be more convenient than that of other bio-based adhesives, which can be blend together with modifiers to prepare soy-based adhesives. Soy flour, as a nonfood plant by-product derived from soy oil production, has high protein content (~52%) [26]. Moreover, it is also abundant, affordable, and readily available. However, the water resistance of soy-based adhesive is poor, resulting from inherent chemical structures of soy proteins. These proteins are globular with few active functional groups in the surface and have low reactivity during the curing process, resulting in failure to form interpenetrating network structure. Therefore, many attempts have been performed to enhance water resistance of soy-based adhesive. Most researchers have tried to unfold soy proteins initially with the exposure of hydrophobic subunits and subsequently employed methods such as grafting modification or blending with other resins [27, 28]. Besides the abovementioned methods, cross-linking modification is considered to be more affordable and convenient [29, 30] because cross-linking agents can be directly mixed with pristine soy-based adhesive prior to the curing process, which can achieve high performance and meet the requirements of relevant standards with simple preparation procedures [27, 31]. Our research group has studied various methods especially cross-linking modification to enhance the performance of soy-based adhesive [30, 32, 33]. In our recent study, epoxy prepolymer was prepared and blended with soy-based adhesive as the cross-linking agent. It was proved to be effective in the preparation of multilayer plywood, which had excellent bonding strength and water resistance [34]. However, further strategies for particleboard manufacturing, especially with reed straw as the raw material, lie in the optimized manufacturing conditions, such as adhesive amount, hot-pressing temperature, or hot-pressing time.

Response surface methodology (RSM) represents a typical experimental optimization method with high efficiency. This methodology can explore the interactions between independent variables and more than one dependent variable with further prediction of their responses under specified series of experiments [35, 36].

Consequently, whether soy-based adhesive could be used to prepare reed straw particleboard that met the requirements of Type P6 (furniture-grade particleboard under humid conditions) according to Chinese Standard GB/T 4897-2015, RSM was employed to test and optimize the manufacturing conditions. The interactions between independent variables (adhesive amount X_1, hot-pressing temperature X_2, and hot-pressing time X_3) and the dependent variable (wet internal bonding strength Y) were investigated, and the optimum conditions for reed straw particleboard were obtained, which was also verified by validation experiments. Moreover, the physical and mechanical properties of reed straw particleboard prepared under optimum conditions were also tested to further validate the accuracy and reliability of the optimization model.

2. Materials and Methods

2.1. Materials. Soy flour was purchased from Xianglin Food Co., Ltd., China, with 52% of protein and 10% of moisture. Reed straw was brought from Taohuajiang State-Owned Forest Farm and ground to the average particle sizes ranging from 5 to 20 mm. The characterization of reed straw is displayed in Table 1.

Before particleboard preparation, the reed straw was pretreated with 1 wt.% sodium hydroxide solutions at 60°C for 30 min to break the wax and inorganic layer of the reed straw epidermis. After this period, the reed straw was dried in an oven until achieving constant weight. Chemicals used in this study were provided by Sinopharm Chemical Reagent Co., Ltd., China, with analytical grade.

2.2. Preparation of Soy-Based Adhesives and Particleborad. The preparation of soy adhesives used in this paper could be referred to our previous work [32]. The procedure was as follows: defatted soy flour, urea, and distilled water were blended together to prepare the soy adhesive emulsion. A 300 ml three-neck flask equipped with a mechanic stirrer, a condenser, and a thermometer was charged with PPGDGE and TETA. Then, the mixture was heated to 50°C and kept for 30 min. After heating, the mixture was cooled to room temperature and slowly added to the soy adhesive emulsion with rapid agitation. This slurry was stirred for another 1 h to prepare the modified soy adhesive. The modified soy-based adhesive was assigned the name SBA.

Table 1: Proximate and chemical analysis of raw material, dry basis (wt.%).

	Ash	Moisture content	Lignin	Cellulose
Reed straw	3.72	8.2	20.12	57.91

Reed straw was mixed with SBA in a blender (B-20 Blender, Guangzhou Panyu Lifeng Food Machinery, Co., Ltd.) at room temperature for 20 min. The mixture was put in a $30 \times 30\ cm^2$ steel mold with stops that allowed reaching a thickness of 0.6 cm. The amount of mixture was weighed and calculated to obtain a target density of $0.75\ g/cm^3$. The mold with the mixture was then placed in the preheated hot press machine (SDR100 × 90, Qingdao Guosen Machinery, Co., Ltd.) under 4.5 MPa at 138°C for 30 min. The mold was taken out from the press machine in order to remove the particleboard when the machine was cooled to room temperature. The panels were stored at room temperature for at least 24 h before tests.

2.3. Wet Internal Bonding Strength (WIB) Test of Reed Straw Particleboard. Wet internal bonding strength (WIB) represents the property of water resistance of soy-based adhesive used in this research. The pretreatment and test of particleboards were implemented specified by Chinese National Standard GB/T 17657. Before tests, all particleboard samples should initially be immersed into boiling water for 120 min and subsequently soaked in water at 20°C for 60 min. After this period, these samples were dried in an oven at 70°C for 16 h. The measurements of WIB were performed on 5 cm × 5 cm square blocks. The crosshead speed of tension-testing machine (Jinan Shijin Co. Ltd., China) was 2.0 mm/min.

2.4. Single-Factor Experimental Design for WIB. Single-factor experiments were designed to optimize the preliminary range of the wet IB for the RSM design. Based on the literatures [37, 38], the key factors on WIB of particleboards were adhesive amount, hot-pressing temperature, and hot-pressing time. Therefore, these factors were selected to be performed in single-factor experiments, and the range of the adhesive amount, hot-pressing temperature, and hot-pressing time were 10%–35% (dry basis), 110–150°C, and 12–36 min, respectively.

2.5. Experimental Design of Response Surface Methodology (RSM). Derived on the above results of single-factor experiments, the software Design-Expert (Version 10.0.1.0, Stat-Ease Inc., USA) was used for experimental design. The Box–Behnken design (BBD) was applied to test the effects of three independent variables (X_1, adhesive amount; X_2, hot-pressing temperature; and X_3, hot-pressing time) at three levels on the dependent variable (Y, WIB). The independent variables and their levels are presented in Table 2. Moreover, the results of the whole design including 17 experimental points performed in a randomized order are presented in Table 3.

Table 2: Variables and levels used in RSM design.

Coded and uncoded variables	Levels		
Factors	−1	0	1
Adhesive amount (X_1, %)	15	20	25
Hot-pressing temperature (X_2, °C)	120	130	140
Hot-pressing time (X_3, min)	18	24	30

The following second-order polynomial model was used to describe the relationship between the independent variables and the dependent variable [39]:

$$Y = \beta 0 + \sum_{j=1}^{k} \beta_j X_j + \sum_{j=1}^{k} \beta_{jj} X_j^2 + \sum \sum_{i<j} \beta_{ji} X_i X_j, \qquad (1)$$

where Y was the predicted response; β_0 was a constant; β_j, β_{jj}, and β_{ji} were the linear, quadratic, and interactive coefficients, respectively; X_i and X_j were the independent coded variables ($i \neq j$; i and j ranging from 1 to k); and k was the number of independent parameters (in this study, $k = 3$).

The fit quality of the polynomial model equation was assessed by both the coefficient of determination (R^2) and ANOVA analysis. The significance of the regression coefficient was evaluated by checking F value and p value [40, 41].

2.6. Thickness Swelling and Mechanical Properties. The physical and mechanical properties, including thickness swelling, modulus of rupture (MOR), and modulus of elasticity (MOE) of reed straw particleboards prepared under optimized conditions, were tested according to the methods referred to Chinese National Standard GB/T 17657-2013. Particleboard samples with dimension of 50 mm × 50 mm were immersed in distilled water at ambient temperature. The weight and thickness of these samples were measured at 2 h, 12 h, 24 h, 36 h, 48 h, and 60 h to evaluate the short and long-term changes during the immersion. The modulus of rupture (MOR) and modulus of elasticity (MOE) were tested on a panel with the dimensions of 14 cm × 5 cm × 0.6 cm (length × width × thickness). The crosshead speed was set to 2.5 mm/min. There were at least six replicates recorded in all these tests.

3. Results and Discussion

3.1. Analysis of Single Factor Experiment

3.1.1. Effect of Adhesive Amount on WIB. The effect of adhesive amount on WIB is displayed in Figure 1. The hot-pressing temperature and hot-pressing time were set to 120°C and 20 min, respectively. When the adhesive amount reached 15%, the WIB was 0.09 MPa, which could meet the requirement of Type P6 particleboard. The WIB was increasing with the rising adhesive amount and could be up to 0.15 MPa with 35% of the adhesive amount. Specifically, the more the adhesive added, the larger the WIB could be. Insufficient adhesive will make the reed straw not fully bonded together, which induces poor WIB.

Table 3: Experimental scheme and results.

Run number	Adhesive amount (X_1, %)	Hot-pressing temperature (X_2, °C)	Hot-pressing time (X_3, min)	WIB (Y, MPa)
1	20	130	24	0.15
2	20	130	24	0.15
3	20	140	30	0.17
4	20	130	24	0.16
5	15	130	18	0.1
6	20	120	18	0.13
7	20	140	18	0.15
8	25	130	18	0.15
9	15	130	30	0.13
10	25	140	24	0.18
11	20	120	30	0.14
12	25	130	30	0.17
13	25	120	24	0.16
14	20	130	24	0.15
15	15	120	24	0.11
16	20	130	24	0.15
17	15	140	24	0.12

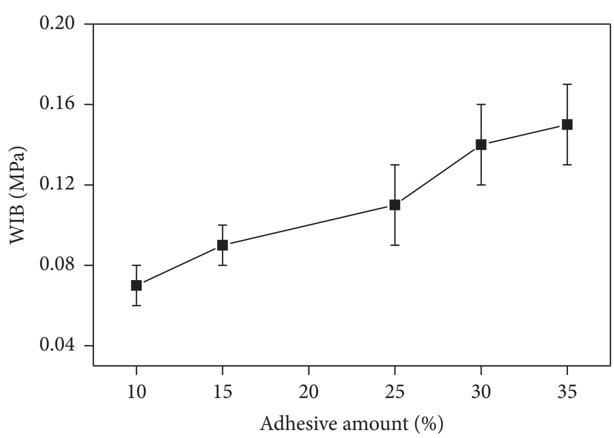

Figure 1: Effect of adhesive amount on WIB.

Therefore, based upon synthetic consideration of the cost and effects, the range from 15%–25% of the adhesive amount was optimal and selected for RSM experiments.

3.1.2. Effect of Hot-Pressing Temperature on WIB. Hot-pressing temperature is a key factor for the curing process of soy-based adhesive. The effect of hot-pressing temperature from 110 to 150°C on WIB of reed straw particleboard was investigated with the adhesive amount of 20% and hot-pressing time of 20 min. As illustrated in Figure 2, the WIB exceeded 0.09 MPa and was up to 0.1 MPa when the hot-pressing temperature was 120°C. Moreover, the WIB reached the peak value to 0.13 MPa when the hot-pressing temperature reached 120°C. However, further rise in hot-pressing temperature had an adverse effect on WIB, which decreased to 0.12 MPa compared with that of 140°C. The curing of soy-based adhesive requires a certain temperature, which usually exceeds 100°C. Over this temperature, the soy-based adhesive can be cured rapidly. However, excessive temperature (> 150°C) can also make the soy-based adhesive cure but with the decomposition of the reed straw fiber [42], which is detrimental to WIB. Consequently, the optimal range of hot-pressing temperature for RSM experiments was from 120 to 140°C.

3.1.3. Effect of Hot-Pressing Time on WIB. Hot-pressing time is crucial for the curing process of soy-based adhesive. The adhesive cannot be cured completely if the time is not enough. For investigating the effect of hot-pressing time on WIB, the adhesive amount and hot-pressing temperature were set to 20% and 120°C, respectively. As displayed in Figure 3, the WIB increased as the hot-pressing time was rising. When the hot-pressing time reached 18 min, the WIB was 0.09 MPa and met the requirement of Type P6 particleboard. Moreover, the trend of WIB under increasing hot-pressing time was the same as the adhesive amount. The longer the hot-pressing time was, the larger the WIB reached. There were interactions between hot-pressing time and hot-pressing temperature. The hot-pressing pressure was fixed in this study due to the fixed thickness of the reed straw particleboard. The pressure can compress the reed straw particle and remove the porosity of the material [43, 44]. With the heating, the water can be evaporated from the mixture and the particle can be glued together by the soy-based adhesive. If the hot-pressing time is too short, the water cannot be fully evaporated, resulting in incomplete curing of the adhesive. Therefore, considering both the cost and practical effects, the hot-pressing time should be selected from 18 min to 30 min for RSM experiments.

3.2. Statistical Analysis and Model Fitting. The results of BBD experiments are shown in Table 3. The coefficient of the independent variables (X_1, X_2, and X_3) for the dependent variable (Y) could be expressed by the second-order polynomial equation illustrated as the follows:

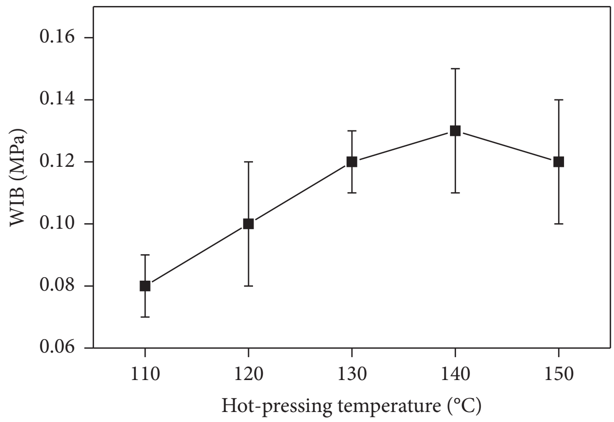

Figure 2: Effects of hot-pressing temperature on WIB.

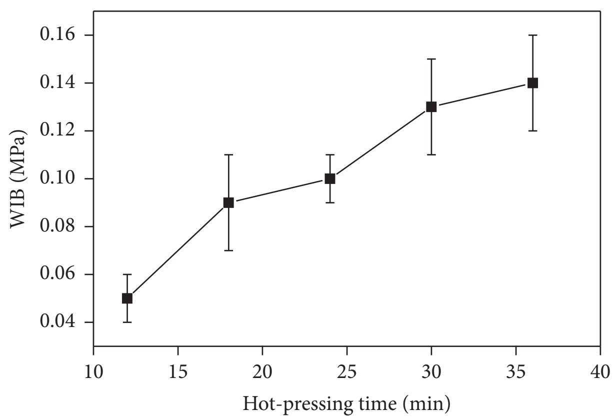

Figure 3: Effect of hot-pressing time on WIB.

$$Y = 0.15 + 0.025X_1 + 0.01X_2 + 0.01X_3 + 2.5 \times 10^{-3}X_1X_2 - 2.5 \times 10^{-3}X_1X_3 + 2.5 \times 10^{-3}X_2X_3 - 9.75X_1^2 + 2.5 \times 10^{-4}X_2^2 - 4.75 \times 10^{-3}X_3^2, \quad (2)$$

where a positive or negative coefficient indicated a synergetic and antagonistic effect, respectively [45].

ANOVA analysis was performed to determine the significance of the second-order polynomial model. The results of variance and fitness of the optimization model are displayed in Table 4. The p value of the model was 0.0002 that was far less than 0.05, which suggested that the second-order polynomial model was fit to describe the effects of the independent variables (X_1, X_2, and X_3) on WIB. Moreover, the value of the determination coefficient (R^2) was 0.969, which indicated 96.9% of the total variation in WIB attributed to the experimental variables. In addition, the adjusted R^2 was 0.9292, which was in good agreement with R^2. Therefore, it could come to the conclusion that the model was accurate enough to predict the WIB in the response. The lack of fit was employed to evaluate the validity of the model. The F value of the lack of fit was 2.5, and the p value was 0.1985 that was more than 0.05, indicating the lack of fit of this model was not significant. It was credible for the model to explain the relationship between the dependent variable and independent variables.

The independent variables all had very significant effects on WIB as illustrated in Table 4. The p values of these variables were less than 0.01. Meanwhile, the interaction term (X_1X_2) also had a very significant level ($p < 0.01$), suggesting that the interaction between the adhesive amount and hot-pressing temperature had very significant effect on WIB. Moreover, the interaction term (X_1X_3) and the quadratic term (X_1^2) had a significant level ($p < 0.05$). However, the interaction term (X_2X_3) and the quadratic term (X_2^2, X_3^2) failed to have a significant level. Consequently, the effects of the independent variables and their interactions on WIB could be considered as not simply linear relationship [46, 47]. In accordance with the sum of squares, the effects of these independent variables on the response should be in the order displayed as follows: adhesive amount (X_1) > hot-pressing temperature (X_2) > hot-pressing time (X_3).

3.3. Analysis of Response Surface Plots. The 3D response surface plots are displayed in Figure 4. Two independent variables were depicted in one plot with the other variables fixed at its center point value. It was shown that the three independent variables all had a significant effect on the response discussed in the previous section, which could be further verified, because the WIB increased with each independent variable increasing according to the illustrations of Figures 4(a)–4(c). The adhesive amount, hot-pressing temperature, and hot-pressing time all had significant effects ($p < 0.01$) on WIB of reed straw particleboard. This was also consistent with relevant studies reported [48, 49]. The adhesive amount was an important factor for particleboard preparation, which directly affects the other variables. Lack of adhesive resulted in the reduction of WIB because there were not enough adhesive between the surface of adjacent reed straw fibers, which could not be bonded together. The interaction terms (X_1X_2, $p = 0.0012 < 0.01$) and (X_1X_3, $p = 0.046 < 0.05$) displayed a significant level indicating these terms had significant effects on WIB with steeper surface plots. However, the combined effects (X_2X_3, $p = 0.432 > 0.05$) of hot-pressing temperature and hot-pressing time were not significant to the response. The reason could be that the combined effect (X_2X_3) on WIB was little when the adhesive amount (X_1) was determined.

3.4. Optimization of Preparation Conditions and Model Validation. The optimal conditions optimized through RSM experiments were obtained by the software and shown as follows: adhesive amount of 24.5%, hot-pressing temperature of 138.35°C, and hot-pressing time of 26.79 min. The predicted optimum WIB of reed straw particleboard manufactured under the predicted conditions was 0.18 MPa. In order to validate the accuracy of the optimal conditions, a set of tests including six parallel experiments were implemented by using modified optimal conditions: 25% of adhesive amount, 138°C of hot-pressing temperature, and 27 min of hot-pressing time. The measured average WIB was 0.17 ± 0.02 MPa, and the percentage error between the

Table 4: ANOVA of RSM experiments.

Source	Sum of square	Df	Mean square	F value	p value	Significance
Model	7.194×10^{-3}	9	7.993×10^{-4}	24.33	0.0002	**
X_1	5×10^{-3}	1	5×10^{-3}	152.17	0.0001	**
X_2	8×10^{-4}	1	8×10^{-4}	24.32	0.0017	**
X_3	8.01×10^{-4}	1	7.98×10^{-4}	24.45	0.0018	**
X_1X_2	2.48×10^{-5}	1	2.5×10^{-5}	0.766	0.0012	**
X_1X_3	2.5×10^{-5}	1	2.5×10^{-5}	0.754	0.046	*
X_2X_3	2.5×10^{-5}	1	2.5×10^{-5}	0.762	0.432	—
X_1^2	4.003×10^{-4}	1	4.003×10^{-4}	12.18	0.0101	*
X_2^2	2.632×10^{-7}	1	2.632×10^{-7}	8.09×10^{-3}	0.9312	—
X_3^2	9.5×10^{-5}	1	9.5×10^{-5}	2.89	0.1329	—
Residual	2.3×10^{-4}	7	3.286×10^{-5}	—	—	—
Lack of fit	1.5×10^{-4}	3	5×10^{-5}	2.5	0.1985	—
Pure error	8×10^{-5}	4	2×10^{-5}	—	—	—
Total correlation	7.424×10^{-3}	16	—	—	—	—
R^2	0.969	$\sqrt{R^2}$	0.9292	—	—	—

*Significant at the 5% level ($p < 0.05$); **significant at the 1% level ($p < 0.01$).

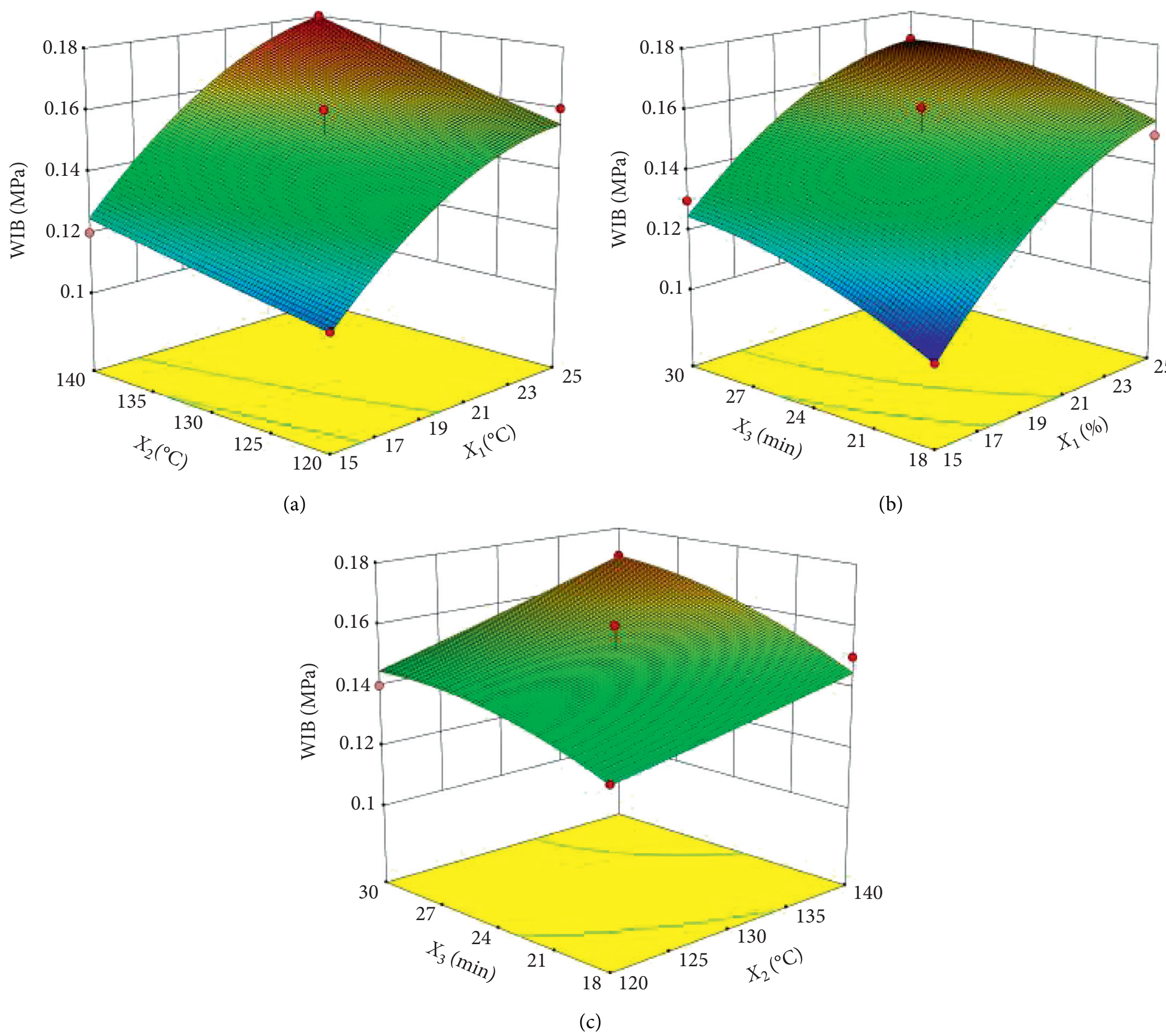

Figure 4: Response surface plots for the effects of dependable variables on WIB.

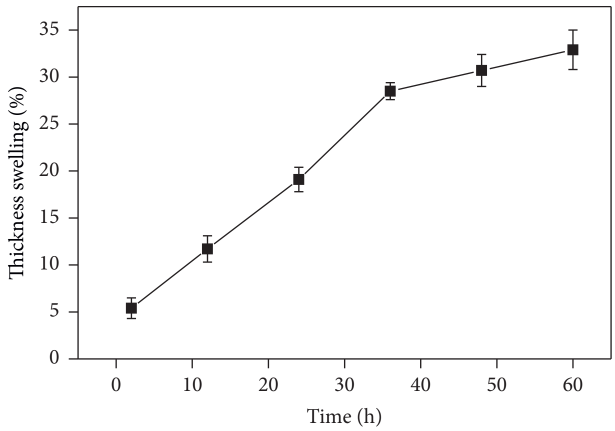

Figure 5: Effects of soaking time on thickness swelling of reed straw particleboard prepared under optimal conditions.

Table 5: Variables and levels used in RSM design.

Mechanical properties	Strength	Type P6 requirement
MOR (MPa)	15.1 ± 2.4	14
MOE (GPa)	2.05 ± 0.8	1.9

measured and predicted WIB was within the value of 6%, demonstrating that the accuracy of this model is reasonable within 94% of the prediction interval. The validity and accuracy of the model were confirmed by this result.

3.5. Thickness Swelling and Mechanical Properties. The effects of soaking time on thickness swelling of reed straw-based particleboard under optimal conditions are illustrated in Figure 5. The thickness swelling of reed straw particleboard was 19.1%, which met the requirements of Type P6 particleboard (20%). In addition, with the raising soaking time, the thickness swelling of reed straw particleboard was increasing sharply. However, when the soaking time reached 40 h, the thickness swelling was growing slowly. The mechanical properties including MOR and MOE are shown in Table 5. Both the MOR and MOE of reed straw particleboard prepared under optimal conditions derived from RSM experiments all exceeded the values required by Chinese National Standard GB/T 4897-2015. Therefore, these results further verified the accuracy and validity of the RSM optimization model.

4. Conclusions

In this study, optimal manufacturing conditions were obtained by using RSM. The effects and interactions of adhesive amount, hot-pressing temperature, and hot-pressing time on wet internal bonding strength of reed straw particleboard with SBA as the binder were investigated. RSM was proved to be a suitable method to the optimization of manufacturing conditions for achieving maximum WIB of the reed straw particleboard by SBA. Through single-factor experiments, the preliminary range for RSM was determined. The regression model for manufacturing conditions was significant ($p = 0.0002 < 0.01$). In addition, the determination coefficient (R^2) and the adjust determination coefficient ($\sqrt{R^2}$) of this model were 0.969 and 0.9292, respectively. The value of R^2 was in reasonable agreement with $\sqrt{R^2}$. The optimal conditions predicted by the model were 25% of adhesive amount, 138°C of hot-pressing temperature, and 27 min of hot-pressing time. Under the optimal conditions, validation tests were performed, and the average value of parallel experiments was 0.17 ± 0.02 MPa. The percentage error between the measured and predicted WIB was within the value of 6%, which was in good agreement with the predicted one. Moreover, the thickness swelling of water absorption after 24 h soaking and mechanical properties (MOE and MOR) of the samples prepared under optimized conditions were further measured, which all met the requirements of Type P6 particleboard. It could provide an efficient method for massive production of reed straw particleboard.

Data Availability

The data used to support the findings of this study are available from the corresponding author upon request.

Conflicts of Interest

The authors declare that they have no conflicts of interest.

Authors' Contributions

Yingjie Zhang and Yong Wang contributed equally to this work.

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