Optimization of Pretreating Poplar Wood Shavings with Cellulase to Produce Binderless Fiberboard Using Response Surface Methodology

Jianguo Wu,[a,b,*] Yuanyuan Wang,[a] Zhao Zhang,[a] Xuanxian Lu,[a] Chi Wang,[a] Zhiqing Zhong,[a] Dong Qian,[a] and Tong Zhang [a,b]

Wood processing waste, poplar wood shavings, were used for fiberboard production, and the pretreatment conditions using cellulase were studied using response surface methodology (RSM). After single factors optimization, central level of temperature, dosage, and liquid/solid ratio (LSR) of cellulase pretreatment conditions were obtained. Further optimization to study the influence of the factors was carried out using Box-Behnken design of experiments. A second-order polynomial equation was obtained, and the low *p*-value (<0.007) implied that the model was highly significant by analysis of variance (ANOVA). The optimized cellulase pretreatment conditions for maximum bending strength (BS) of the fiberboard were determined by ridge analysis as 44.4 °C of temperature, 1.23 U/g of dosage, 4.2 of LSR, and 5 h of pretreatment time. Under the optimized conditions, the BS of the fiberboard reached 25.12 ± 0.35 MPa by validation experiment, which was twice that of the fiberboard without pretreatment. Thus, the cellulase pretreatment should be a good choice to produce high-strength binderless fiberboard.

Keywords: Fiberboard; Binderless; Cellulase; Pretreatment; Poplar wood shavings; Response surface methodology; Optimization

Contact information: a: Jiangsu Key Laboratory for Eco-Agricultural Biotechnology around Hongze Lake, Jiangsu Key Construction Laboratory for Food Safe and Nutritional Function, School of Life Science, Huaiyin Normal University, Changjiang West Road 111, Huai'an 223300, China; b: Jiangsu Collaborative Innovation Center of Regional Modern Agriculture and Environmental Protection, Huaiyin Normal University, Changjiang West Road 111, Huai'an 223300, China;
** Corresponding author: jgwu_21@sina.cn*

INTRODUCTION

As traditional fiberboard production widely employs formaldehyde- and other aldehyde-based adhesives, toxic and harmful gases that are released during the production seriously pollute the environment and endanger human health (Bertrand *et al.* 2007; Lithner *et al.* 2011; Piekarski *et al.* 2017; Pang *et al.* 2018). Consequently, eco-friendly binderless fiberboard production technology has been growing in recent years. Geng *et al.* (2006) found that the fiberboards made from alkaline-treated bark showed lighter color, higher internal bonding strength, higher modulus of rupture, and higher modulus of elasticity in comparison with the control panels. Nikhom and Okuma (2000) used oil palm frond as raw material to produce viscous substances by high temperature steam explosion, and they further studied the binderless fiberboard production technology. Binderless fiberboards using *Triarrhena sacchariflora* residue, corn stalk, and poplar wood shavings by bio-pretreatment with white-rot fungus *Trametes hirsute* or *Trametes versicolor* have been studied (Wu *et al.* 2011, 2016, 2020). However, physical and chemical methods had the

disadvantages of high pollution and energy consumption, while bio-pretreatment methods had the disadvantages of long pretreatment time. Hence, more attention has been paid to enzyme pretreatment (Widsten and Kandelbauer 2008).

Kharazipour and Mai (1998) first pretreated wood fiber with laccase without any traditional adhesive, and then they obtained fiberboard with certain mechanical strength by dry pressing and wet pressing, respectively. Felby *et al.* (2002) and Euring *et al.* (2011) pretreated raw materials by laccase, and they carried out small-scale factory production experiments of medium density fiberboard. Ibrahim *et al.* (2013) used laccase to modify kraft lignin to formulate adhesives combined with polyethyleneimine, chitosan, and soy protein, respectively. Wu *et al.* (2016) found that there was a positive correlation between the content of hydroxyl group and the mechanical strength of fiberboard. Cellulase could break the β-1,4-glycoside bond of cellulose into amorphous cellulose, cello-oligosaccharide, and small molecule sugars, which are rich in the hydroxyl group. Therefore, the cellulase pretreatment should be also a potential binderless fiberboard production method (Yu *et al.* 2003). However, still there are no relevant research reports available.

In China, the poplar wood shavings (PWS), the waste from poplar wood processing, are very easily available. PWS could be used for production of fiberboard, lipids, xylitol and bioenergy (Chen *et al.* 2016; Dalli *et al.* 2017; Li *et al.* 2019; Wu *et al.* 2020), but there have been no reports about binderless fiberboard using PWS by cellulase pretreatment. In this study, cellulase was firstly used to pretreat PWS for binderless fiberboard production. The cellulase pretreatment conditions were optimized using response surface methodology (RSM) (Gunny *et al.* 2013; Wang *et al.* 2018; Mohammed *et al.* 2020). The result would provide technical support for the production of binderless fiberboard by cellulase pretreatment.

EXPERIMENTAL

Materials and Agents

Well-distributed shavings of PWS collected from a wood processing factory in Huai'an (China), were dried at 60 °C in an oven to a constant weight. Cellulase was purchased from Huai'an (Bio-mass Green Bioenergy Co. Ltd, China). The filter paper activity (FPA), carboxymethylcellulose activity (CMC), β-glucosidase activity, and xylanase activity of the cellulase were about 350 U/g, 4000 U/g, 60 U/g, and 20000 U/g, respectively. Acetate buffer (pH 4.8) was prepared by mixing 12 mL of sodium acetate solution (1.0 M) and 8 mL of acetic acid solution (1.0 M) in 1 L purified water. Acetic acid and sodium acetate used were all of analytical grade and purchased from the Sinopharm Chemical Reagent Co. Ltd., China.

Cellulase Pretreatment

Approximately 60 g of PWS was immersed in 300 mL of acetate buffer with 60 U of cellulase (FPA). The pretreatment temperature was controlled at 50 °C by a thermostat (uncertainty of ± 0.1 °C) in the shaker. After 2 h of pretreatment, the PWS were loaded into six layers of gauze and squeezed by hands to about 60% moisture content to prepare fiberboard by hot pressing.

Single Factor Optimization

The initial single factor conditions used was 50 °C of temperature (*T*), 1.0 FPA U/g of PWS of enzyme dosage, liquid/solid ratio (LSR) of 5, and 2 h of pretreatment time (*t*). The single factor optimization method is shown in Table 1.

Table 1. Single Factor Optimization Method

Single factor	Level			
	T (°C)	Dosage (U/g)	LSR	*t* (h)
T	35, 40, 45, 50, 55	1	5	2
Dosage	50	0.5, 0.75, 1, 1.25, 1.5	5	2
LSR	50	1	3, 4, 5, 6, 7	2
t	50	1	5	1, 2, 3, 4, 5

Response Surface Methodology Optimization

Factor T, dosage, and LSR were optimized by response surface methodology (RSM) and the Box-Behnken design of experiments as shown in Table 2.

Table 2. Level of Variables for the Box-Behnken Design of Experiments

Factor	Code	Level		
		-1	**0**	**1**
T (°C)	x_1	40	45	50
Dosage (U/g)	x_2	1	1.25	1.5
LSR	x_3	3	4	5

Preparation of Fiberboard

The well-distributed PWS were manually loaded into a stainless mold (100 mm × 100 mm × 3 mm), then pressed flat into a fiberboard by a R-3202 Hot-Press Model (Wuhan Qien Science and Technology Development Co. Ltd., Hubei, China). Hot pressing was carried out at 170 °C under 17 MPa of pressure for 10 min.

Property of Fiberboard

The length, width, and thickness of fiberboard were measured by a micrometer, and the mass of fiberboard was measured by electronic balance (Model BSA124S, Sartorius, Gottingen, Germany), so the density of fiberboard was calculated by dividing the fiberboard mass (kg) by its volume (m^3). The fiberboard was cut into specimens of 100 mm in length and 20 mm in width. The bending strength (BS) of fiberboard specimens was determined by performing the three point flex test with 60 mm of span length at 3 mm/min crosshead speed using a micro-computer controlled electron universal testing machine (Model ETM104B, Wance Group, Shenzhen, China). The BS could be calculated as Eq. (1). The water swelling ratio was measured from the different thickness (h) before and after immersing in water for 24 h, and the water swelling ratio could be calculated as Eq. (2). Detailed procedures were carried out as shown in National Standard (GB/T 11718-2009) (Standardization Administration of China 2009).

$$BS = \frac{3FL}{2bh^2} \quad (1)$$

$$\text{Water swelling ratio } (\%) = \frac{h_2 - h_1}{h_1} \times 100\% \quad (2)$$

where F is the maximum force to break the fiberboard specimens (N); L is the span length, L = 60 mm; b is the width of fiberboard specimens (mm); h is the thickness of fiberboard specimens (mm); h_2 is the final thickness of fiberboard after immersing in water (mm); and h_1 is the initial thickness of fiberboard before immersing in water (mm).

Statistical Analysis

Test Pilot software (Wance Group, Shenzhen, China) was used to calculate BS, ORIGIN PRO 8.0 (OriginLab Corp., Wellesley, MA, USA) was used to analyze the data and draw bar graphs, and Statistical Analysis System 9.4 (SAS 9.4) (SAS Institute Inc., North Carolina, USA) was used to design experiment and analyze the data of RSM. The error bars in bar graphs correspond to standard errors of three tests.

RESULTS AND DISCUSSION

Single Factor Optimization of Cellulase Pretreatment Conditions

The results of enzyme dosage, LSR, temperature, and pretreatment time for BS of fiberboard are shown in Fig. 1. After PWS was pretreated by cellulase, it was made into fiberboard without adhesive. With the increase of dosage, LSR, and temperature, the BS of fiberboard increased first and then began to decrease. At a dosage of 1.25 U/g, LSR of 4, and at 45 °C, the BS of fiberboard indicated a peak value. The pretreatment time of cellulase had a positive correlation with the BS of fiberboard within 5 hours of pretreatment. The BS of fiberboard reached the maximum at 5 h of pretreatment time, and it decreased slightly after prolonging the pretreatment time.

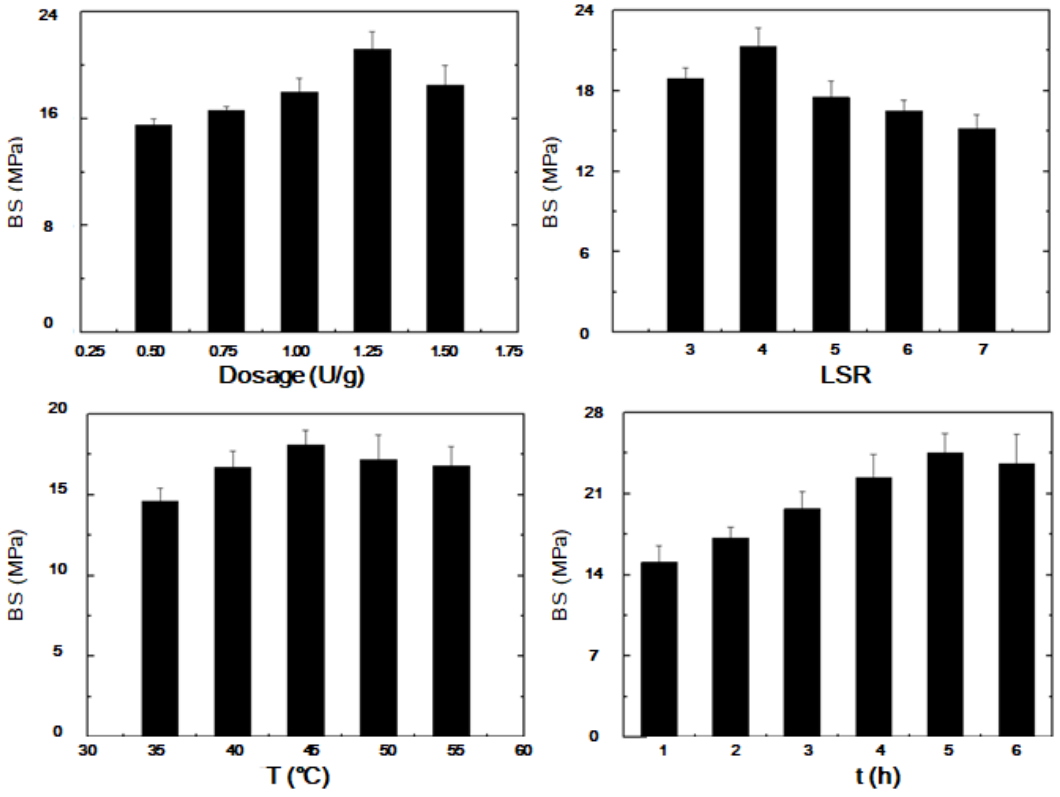

Fig. 1. Sing factor optimization of cellulase pretreatment conditions

The interaction effect of dosage, LSR, and temperature might appear in the process of cellulase pretreatment. It was necessary to optimize these three factors by RSM. To ensure the pretreatment effect, the pretreatment time was kept for 5 h.

Optimization by Response Surface Methodology

Response surface methodology using Box-Behnken design (BBD) (Li *et al.* 2018) was made to determine the optimal levels of the three selected factors (*T* for x_1, dosage for x_2, LSR for x_3). The experimental design and results are shown in Table 3.

Table 3. Box-Behnken Design with BS as Response

Run	Variables Level			BS (MPa)
	T (°C)(x_1)	Dosage (U/g)(x_2)	LSR(x_3)	
1	40	1	4	22.38±1.53
2	40	1.5	4	22.05±1.35
3	50	1	4	20.18±1.47
4	50	1.5	4	21.31±1.16
5	45	1	3	22.88±1.33
6	45	1	5	24.10±1.58
7	45	1.5	3	23.53±1.46
8	45	1.5	5	22.78±1.39
9	40	1.25	3	21.85±1.58
10	50	1.25	3	21.59±1.19
11	40	1.25	5	22.28±1.25
12	50	1.25	5	21.67±0.89
13	45	1.25	4	24.34±1.42
14	45	1.25	4	25.04±1.51
15	45	1.25	4	24.26±1.44

The relationships between BS and the tested factors were obtained by application of RSM. By employing multiple regression analysis on the experimental data, BS and the tested factors were related by the following second-order polynomial equation as Eq. (3). The 3D response surfaces and 2D contour plots could be generated by SAS 9.4 based on the Eq. (3).

$$Y = -179 + 7.785 \cdot x_1 + 26.638 \cdot x_2 + 6.799 \cdot x_3 - 0.091 \cdot x_1^2 + 0.292 \cdot x_1 \cdot x_2 - 0.018 \cdot x_1 \cdot x_3 - 12.733 \cdot x_2^2 - 1.97 \cdot x_2 \cdot x_3 - 0.428 \cdot x_3^2 \quad (3)$$

where *Y* was BS of the fiberboard, x_1 was temperature (*T*), x_2 was dosage, and x_3 was LSR.

Table 4. Analysis of Variance (ANOVA) for the Second-Order Polynomial Model

Source	Degree of Freedom	Sum of Square	Mean Square	*F*-Value	*p*-Value
Model	9	24.107	2.679	11.406	0.007
Error	5	1.174	0.235	-	-
(Lack of Fit)	3	0.806	0.269	1.459	0.431
(Pure Error)	2	0.368	0.184	-	-
Total	14	25.281	-	-	-
R^2 = 0.954					

The analysis of variance (ANOVA) data for the selected quadratic polynomial model is listed in Table 4. The high model *F*-value (11.406) and low *p*-value (<0.007) implied that the model was highly significant. The coefficient of determination R^2 was calculated as 0.954, which implied that the model was reliable for the BS of fiberboard.

Table 5. Regression Results of the Box-Behnken Design

Variables	Estimate	Standard Error	*t*-Value	*p*-Value
x_1	-0.476	0.171	-2.780	0.039
x_2	0.016	0.171	0.095	0.928
x_3	0.123	0.171	0.715	0.507
$x_1 * x_1$	-2.271	0.252	-9.004	0.000
$x_1 * x_2$	0.365	0.242	1.506	0.192
$x_1 * x_3$	-0.088	0.242	-0.361	0.733
$x_2 * x_2$	-0.796	0.252	-3.156	0.025
$x_2 * x_3$	-0.493	0.242	-2.033	0.098
$x_3 * x_3$	-0.428	0.252	-1.698	0.150

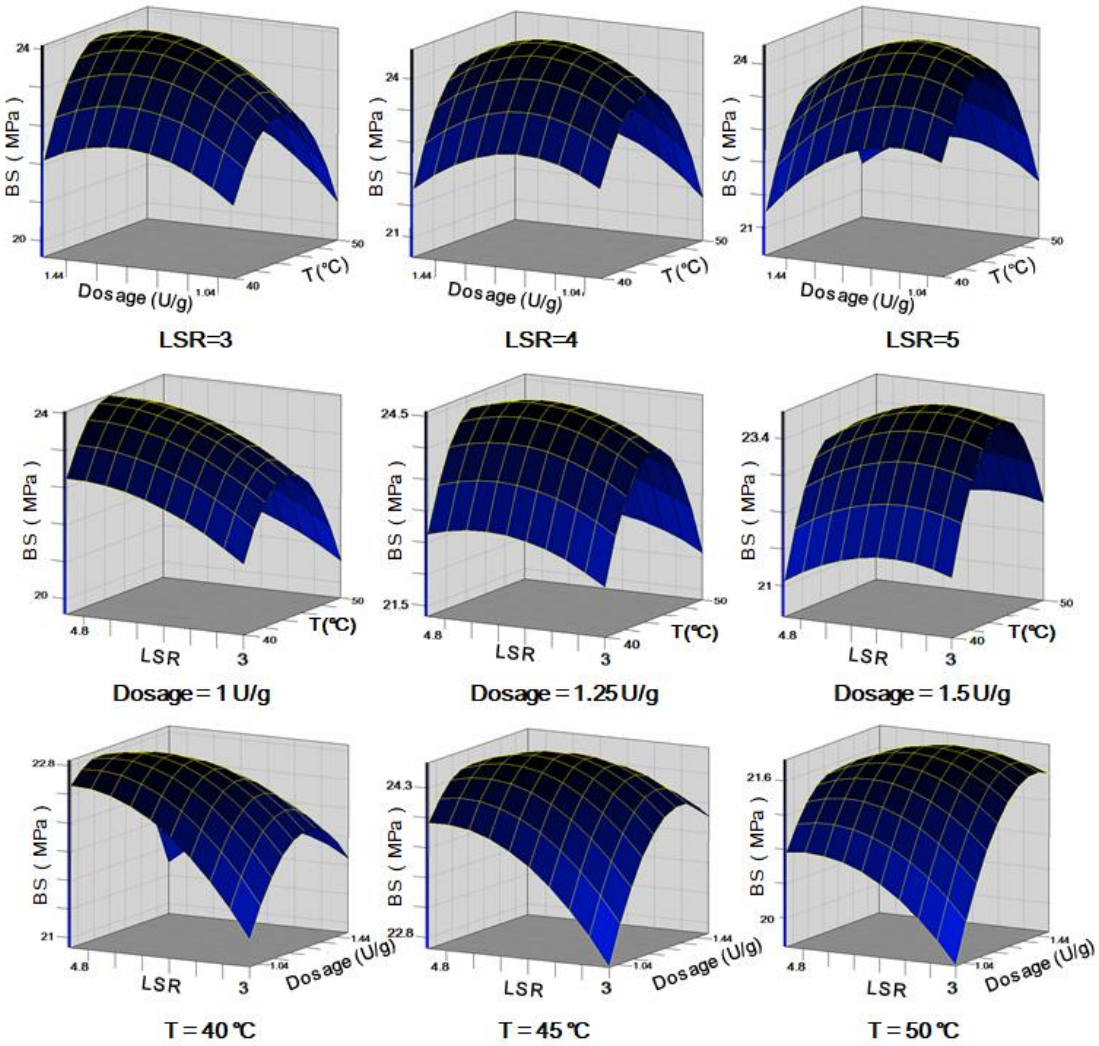

Fig. 2. 3D Response surfaces showing the effects of temperature (*T*), Dosage, and LSR on the BS of fiberboard

The coefficient estimates of model equation, along with the corresponding *p*-values, are presented in Table 5. The *p*-value was employed to check the significance of each coefficient, which also indicated the interactions between the variables (Liu *et al.* 2003; Sakr *et al.* 2019). The corresponding coefficient was more significant as the *p*-value was smaller. Temperature (x1) was highly significant with *p*-values less than 0.05, indicating that temperature was the most effective factor (Souri *et al.* 2018).

The 3D response surfaces and 2D contour plots (Figs. 2 and 3) generated by SAS 9.4 were the graphical representations of the regression equation (Eq. 3). These plots visualized the relationship between the response and each variable, and the interactions between two tested variables. The 3D response surfaces and 2D contour plots could also locate the optimum values of the factors for the maximum of the response. The maximum predicted response was indicated by the surface confined in the smallest ellipse in the contour diagram (Muralidhar *et al.* 2001; He *et al.* 2013; Zuorro *et al.* 2019). Peaks were found in 3D response surfaces (Fig. 2), and the smallest ellipse was also found in 2D contour plots (Fig. 3), which indicated that there was indeed an optimal condition to get the maximum BS of the fiberboard.

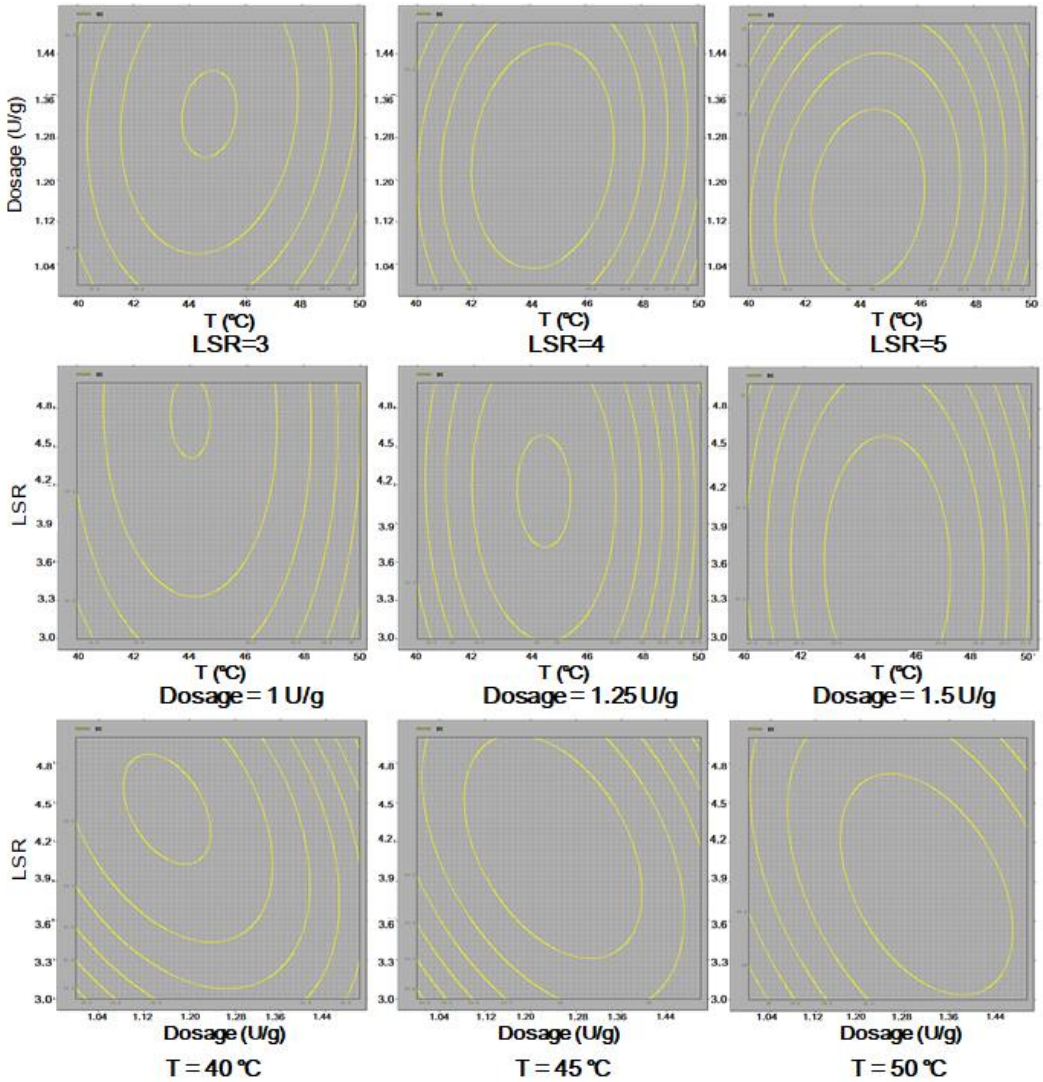

Fig. 3. 2D Contour plots showing the effects of temperature (*T*), Dosage, and LSR on the BS of fiberboard

Optimization Results and Verification

The optimum conditions for cellulase pretreatment could be determined by ridge analysis (Kim and Rhee 2015), and the results are shown in Table 6. Based on ridge analysis, the maximum BS of fiberboard could reach to 24.586 ± 0.275 MPa, when temperature was 44.4 °C, dosage was 1.23 U/g, and LSR was 4.20. To verify the predicted results, the validation experiments were performed in triplicate. Under the optimized condition, the BS of fiberboard reached 25.12 ± 0.35 MPa with implying that experimental and predicted values (24.586 MPa) were in good agreement. The BS of the fiberboard using PWS pretreated under the optimized cellulase pretreatment conditions was twice that of the fiberboard without pretreatment (12.35 MPa).

The density of fiberboard was 952 kg/m^3, which indicated that the fiberboard belonged to high density fiberboard based on the National Standard of the People's Republic of China (GB/T 31765-2015) (Standardization Administration of China 2015). However, BS of the fiberboard was lower than that of national standard (37 MPa). Water swelling ratio of the fiberboard was only 11.8%, which reached the national standard (<12%). Comparing the national standard of medium density fiberboard (GB/T 11718-2009) (Standardization Administration of China 2009), water swelling ratio of the fiberboard was better than that of the national standard of medium density fiberboard (35%), and BS of the fiberboard reached the national standard (25 MPa). In addition, the BS of the transversely cutting specimen, longitudinally cutting specimen, and 45° obliquely cutting specimen were 25.3 MPa, 25.0 MPa, and 24.8 MPa, respectively. The fiberboard could be approximated as an in-plane isotropic material. Consequently, the cellulase pretreatment should be a good choice to produce high-strength binderless fiberboard, and the fiberboard could be used as high quality medium density fiberboard. Hydrolysis of cellulose by cellulase could increase the content of hydroxyl group in material, which was beneficial to the strength of fiberboard. Steam explosion or acid hydrolysis could also increase the hydroxyl group availability in materials. Whether it can also be used to improve the strength of fiberboard? That will be an interesting research topic.

Table 6. Ridge Analysis for BS as Response

Radius	*T* (°C)(x_1)	Dosage (U/g)(x_2)	LSR (x_3)	Predicted Response	Standard Error
0	45.000	1.250	4.000	24.547	0.280
0.1	44.598	1.247	4.058	24.577	0.279
0.2	44.457	1.236	4.158	24.585	0.276
0.3	44.392	1.224	4.254	24.585	0.273
0.4	44.345	1.212	4.346	24.577	0.268
0.5	44.304	1.201	4.438	24.564	0.263
0.6	44.267	1.189	4.528	24.545	0.260
0.7	44.231	1.177	4.618	24.519	0.259
0.8	44.196	1.166	4.708	24.488	0.262
0.9	44.163	1.154	4.797	24.451	0.271
1	44.129	1.143	4.886	24.407	0.288

CONCLUSIONS

The optimization condition of cellulase pretreatment was obtained as 44.4 °C of temperature, 1.23 U/g of dosage, 4.2 of LSR, and 5 h of pretreatment time. Under the optimized condition, the BS of fiberboard was reached 25.12±0.35 MPa, which was twice that of the fiberboard without pretreatment. Therefore, cellulase pretreatment should be a good choice to produce high strength binderless fiberboard.

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