Optimization for Fire Performance of Ultra-low Density Fiberboards Using Response Surface Methodology

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The optimization of the process conditions for fire retardant ultra-low density fiberboards (ULDFs) was investigated using response surface methodology (RSM). Three parameters, namely those of Borax-Zinc-Silicate-Aluminum (B-Zn-Si-Al), chlorinated paraffin (CP), and chloride-vinyl chloride emulsions (PVDC) were chosen as variables. The considerably high R^2 value (99.98%) indicated the statistical significance of the model. The optimal process conditions for the limiting oxygen index (LOI) were determined by analyzing the response surface's three-dimensional surface plot and contour plot, and by solving the regression model equation with Design Expert software. The Box-Behnken design (BBD) was used to optimize the process conditions, which showed that the most favorable dosages of B-Zn-Si-Al, CP, and PVDC were 800 mL, 46.47 mL, and 35.64 g, respectively. Under the optimized conditions, the maximum LOI was 48.4.

Keywords: Ultra-low density; Optimization; Fire performance; Response Surface Methodology; Box-Behnken design; Limiting oxygen index

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INTRODUCTION

With the critical lack of wood reserves in China, consuming agricultural wastes as raw materials has become progressively more important to the wood industry (Gu and Gao 2002). Agricultural wastes are the most promising fibrous raw materials that will support the sustainable development of the wood composite industries in China (Thomas *et al.* 2011; Chen *et al.* 2013, 2014, 2015a,b, 2016b). Ultra-low density fiberboards (ULDFs) that are produced by biodegradable, inexpensive, sustainable, abundant, and environmentally friendly plant fibers have recently attracted increased attention (Xie *et al.* 2008a,b, 2011). They can be used as architectural heat preservation materials and buffering packaging materials.

However, natural fibers have a low limiting oxygen index (LOI) indicating that they have a high flammability and could be easily ignited in the presence of air. Inorganic flame retardants, such as Al-Si compounds (Niu *et al.* 2014), Borax-Zinc-Silicate-Aluminum (B-Zn-Si-Al) compounds (Wu *et al.* 2016b), aluminum trihydroxide (Liang *et al.* 2013; El Hage *et al.* 2014), and magnesium hydroxide (Hoffendahl *et al.* 2015), have been extensively used in fire retardant polymeric materials because they have good thermal-stability, as well as smoke-suppressing and toxic-free additives. Chlorinated paraffin (CP) (Chen *et al.* 2016a) and poly(vinylidene chloride-vinyl chloride) emulsions (PVDC) (Wu *et al.* 2016a) are also used in wood fiber products for their low cost, convenience, and excellent compatibility. Nevertheless, very high doses of inorganic retardants must be added in products for them to show excellent fire retardant capabilities. Once the loading level in a product is high, its physical and mechanical properties would decrease. Moreover, halogen retardants could release smoke to the air. Therefore, the need to seek a cheaper, safer, and more efficient compounded fire retardant for ULDFs is significant.

Response surface methodology (RSM) combines the advantages of statistical and mathematical methods. It can evaluate the effects of several independent variables for the optimization of complex multi-variable processes in one experiment system (Box and Draper 1987). The method of RSM has been used widely in many industries because it is practical and derived from experimental methodology (Baş and Boyacı 2007). It can be used to depict the overall effects of the response parameters, including the interactions between independent experimental factors and response parameters (Guo *et al.* 2011).

In this paper, a compounded fire retardant made with B-Zn-Si-Al compounds, CP, and PVDC is used in ULDFs. There is a synergistic effect between them and CP and PVDC are cheap and efficient, which can reduce the needed dose of fire retardants. The B-Zn-Si-Al compounds can suppress the smoke produced from CP and PVDC. Response surface methodology was used to optimize the processing conditions of ULDFs.

EXPERIMENTAL

Materials

Kraft pulp (KP, spruce-pine-fir; Tembec Inc., Quebec, Canada) was used to fabricate the ULDFs. Sodium silicate, aluminum sulfate, borax, and zinc sulfate were purchased from Tianjin Fuchen Chemical Reagents Factory (Tianjin, China) and were utilized in the preparation of B-Zn-Si-Al compounds. The CP and PVDC were supplied by the Changzhou Fengshuo Chemical Company, Ltd. (Changzhou, China). Sodium dodecylbenzene sulfonate was purchased from the Jiangsu Qingting Washing Products Co., Ltd. (Jiangsu, China).

Methods

Manufacture of ultra-low density fiberboards

The ULDFs were manufactured using the same method described in Xie *et al.* (2011) with a target bulk density of 50 kg/m^3 to 70 kg/m^3.

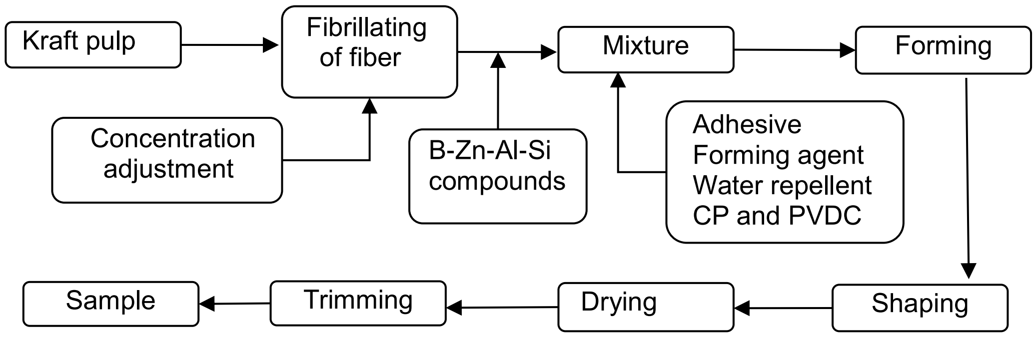

Fig. 1. The preparation of flame-retardant specimens (adapted from Wu *et al.* (2016b))

Twenty mL of adhesive (25 wt.% polyvinyl alcohol, 25 wt.% gelatinized starch, and 0.25 wt.% polyacrylamide), 40 mL of surfactant (20 wt.% sodium dodecyl benzene sulfate), 50 mL of water repellent (alkyl ketene dimmer), CP, and PVDC were added during the mixing stages. The B-Zn-Al-Si compounds were prepared as described in Wu *et al.* (2016b). The detailed process is shown in Fig. 1.

Experimental design and statistical analysis

In this study, the limiting oxygen index (LOI) was used to optimize the conditions for manufacturing fire retardant ULDFs with the help of the Box-Behnken experimental design (BBD) using RSM. The factors and the levels were chosen by one-factor experiments. The selections of variables are shown in Table 1.

Table 1. Codes and Levels of Factors Chosen for the Trials

Symbol	Variable	Codes and Levels		
		-1	0	1
A (mL)	Dose of B-Zn-Si-Al	600	700	800
B (g)	Dose of CP	30	40	50
C (mL)	Dose of PVDC	40	60	80

The software program Design Expert 8.0.6 (Stat-Ease Inc., Minneapolis, USA) was utilized to execute the optimization and mathematical modeling of the fire retardant ULDFs fabricating conditions. The BBD was used to model the RSM. Three independent variables, namely the dose of B-Zn-Si-Al compounds, CP, and PVDC, were chosen in the BBD design. Table 1 shows the variables that were coded as A, B, and C, and their levels were coded as -1, 0, and +1. The variables and levels came from the results of the single-factor experiments. The LOI value was picked as the response variable. An analysis of variance (ANOVA) was used to analyse the response surface model. In deciding the significance and adequacy of the quadratic polynomial model, ANOVA plays an important role.

Limiting oxygen index (LOI) test

The LOI was selected as the criterion by which to assess the flame resistant property of ULDFs. In accordance with GB/T 2406.2 (2009), experiments were implemented using a HC-2 limiting oxygen index instrument (Jiang Ning County Analysis Instrument Factory, Nanjing, China). Test specimens were sawn to the sizes of $150 \times 10 \times 10$ mm^3 ($L \times W \times H$). The shaped specimens were put in the specimen holder surrounded by a transparent glass tube, and the mixed gas fluent (O_2 and N_2) was regulated until the mixed gas stream near the specimen was steady. Every specimen on the top surface of the square pillar was lit and burned downwards. Afterwards, the lowest oxygen concentration that supported combustion was noted for this specimen. The final results were gathered by determining the mean value of five parallel experiments.

RESULTS AND DISCUSSION

Experimental Design Matrix and Results

The BBD experimental design of 17 experiments and the corresponding results of LOI are shown in Table 2.

Table 2. Experimental Designs and Response Values

No.	A	B	C	LOL	
				Experimental Values	Predicted Values
1	-1	-1	0	35.7	35.6
2	1	-1	0	44.5	44.5
3	-1	1	0	41.9	41.9
4	1	1	0	45.4	45.5
5	-1	0	-1	35.1	35.1
6	1	0	-1	40.7	40.6
7	-1	0	1	38.8	38.9
8	0	0	1	45.8	45.8
9	0	-1	-1	38.1	38.2
10	0	1	-1	43.6	43.6
11	0	-1	1	44.5	44.5
12	0	1	1	46.4	46.3
13	0	0	0	47.5	47.4
14	0	0	0	47.3	47.4
15	0	0	0	47.4	47.4
16	0	0	0	47.4	47.4
17	0	0	0	47.4	47.4

Model Fitting

The correlations between the test results and variances were analyzed by different fitting models of RSM. The results are shown in Table 3.

Table 3. Sequential Model Sum of Squares

Source	Sum of Squares	df	Mean Square	F-value	P-value	
Mean *vs.* Total	31994.49	1	31994.49	-	-	-
Linear *vs.* Mean	144.28	3	48.09	4.34	0.0251	-
2FI *vs.* Linear	10.75	3	3.58	0.27	0.8462	-
Quadratic *vs.* 2 FI	133.1	3	44.37	4969.34	<0.0001	Suggested
Cubic *vs.* Quadratic	0.043	3	0.014	2.83	0.1701	Aliased
Residual	0.020	4	0.005	-	-	-
Total	32282.69	17	1898.98	-	-	-

The significance of the fitting model depends on the “P-value.” Generally, if the “P-value” is more than 0.05, it indicates that the model is not significant. In contrast, if the “P-value” is less than 0.05, it is significant; if the “P-value” is less than 0.01, it is extremely significant. As shown in Table 3, the models of “2FI *vs*. Linear” and “Cubic *vs.* Quadra” were not significant, while the model of “Linear *vs.* Mean” was significant and the model of “Quadratic *vs.* 2 FI” was very significant. In accordance with the sequential model sum of squares, the models were chosen in accordance with the maximum order polynomials, and should not be aliased. The additional terms in this polynomial were significant.

The model(s) suggested are picked *via* the Whitcomb Score. The default is the model with the highest Score1. If one model is highest on Score1 and a different model is highest on Score2, then both models will be "Suggested". The experimenter must choose between them. Model Score is defined as:

Score1 = (M)(L)(Predicted R^2)
Score2 = (M)(L)(Adjusted R^2)
Where: M is the Sequential Model Sum of Squares score (Table 3):
M=1, if $p \leqslant 0.05$ (p=p-value);
M=0.05/p, if $p > 0.05$;
M=0; if model is aliased.
L is the Lack of Fit score (Table 4):
L=1, if $p \geqslant 0.10$ (or if Lack of Fit not present)
L=p/0.10, if $p < 0.10$

In this case, the model of "Quadratic vs. 2 FI" is selected since it gets the highest value in both score1 and score2.

The model summary statistics are shown in Table 4. These statistics focused on the model's maximum "Predicted R^2" and the "Adjusted R^2" values. A suitable model should obtain the highest "Predicted R^2" and a lowest prediction sum of squares (PRESS). As shown in Table 4, the Quadratic model was similar with the Cubic model, but both of them had higher Adjusted R^2 values than the model of Liner and 2FI. The "Predicted R^2" of the Quadratic was the highest and the PRESS was the lowest. Based on above analysis, the suitable model was the quadratic polynomial model.

Table 4. Model Summary Statistics

Source	Lace of Fit p-value	R^2	Adjusted R^2	Predicted R^2	PRESS	-
Linear	0.0001	0.5006	0.3854	0.2248	223.42	-
2FI	0.0001	0.5379	0.2607	-0.2511	360.57	-
Quadratic	0.1701	0.9998	0.9995	0.9975	0.71	Suggested
Cubic	-	0.9999	0.9997	-	+	Aliased

In deciding the significance and adequacy of the quadratic polynomial model, an analysis of variance (ANOVA) played an important role, and Table 5 lists the summary of the ANOVA. Due to the model's F-value of 3585.77, it can be inferred that this model was significant. It was merely a 0.01% probability that one Model "F-Value" would happen because of noise. Values of the "P-value" < 0.05 suggested the model terms were significant. In this case A, B, C, AB, AC, BC, A^2, B^2, and C^2 were significant model terms. When the values were > 0.05 it implied the model terms were not significant. Lack of fit is measuring how well the model fits the data. Strong lack of fit ($p<0.05$) is an undesirable property, because it indicates that the model doesn't fit the data well. It is desirable to have an insignificant lack of fit ($P>0.1$). The P-value of the "lack-of-fit" was 0.1701 and was > 0.1, which indicated that the P-value was not significant. The model fit well and could be used for practical prediction in this experiment. Table 6 demonstrates the analysis of credibility on this model. The value "Predicted R^2" of 0.9975 was in reasonable harmony with the "Adjusted R^2" of 0.9995. "Adequate precision (AP)" assessed the signal to noise ratio (Wang and Lu 2005). An AP ratio greater than 4 was satisfactory (Bowerman 1991). Concurrently, the low value of the coefficient of variation (COV) (0.22%) indicated good accuracy and dependability of the tests as recommended by Ahmad *et al.* (2005) and Whitcomb (1994). In the present case, an AP ratio of 169.895 suggested an adequate signal. As a result, the model could be utilized to navigate this design space.

Table 5. The ANOVA for the Response Surface Quadratic Polynomial Model

Source	Sum of Squares	DF	Mean Square	F-value	P-value Prob. > F	
Model	288.14	9	32.02	3585.77	< 0.0001	Significant
A	77.50	1	77.50	8680.14	< 0.0001	-
B	26.28	1	26.28	2943.50	< 0.0001	-
C	40.50	1	40.50	4536.00	< 0.0001	-
A B	7.02	1	7.02	786.52	< 0.0001	-
A C	0.49	1	0.49	54.88	0.0001	-
B C	3.24	1	3.24	362.88	< 0.0001	-
A^2	77.40	1	77.40	8668.87	< 0.0001	-
B^2	6.45	1	6.45	722.18	< 0.0001	-
C^2	38.21	1	38.21	4279.65	< 0.0001	-
Residual	0.062	7	0.0089	-	-	-
Lack of Fit	0.042	3	0.014	2.83	0.1701	Not Significant
Pure Error	0.02	4	0.005	-	-	-
Cor. Total	288.20	16	-	-	-	-

Table 6. Analysis of Credibility on this Model

Std. Dev	Mean	R^2	Adjusted R^2	Predicted R^2	COV	Adequate Precision (AP)
0.094	43.38	0.9998	0.9995	0.9975	0.22%	169.895

The final equation in terms of the coded factors was as follows,

$$Y = +47.40 + 3.11 * A + 1.81 * B + 2.25 * C - 1.33 * A * B + 0.35 * A * C - 0.90 * B * C - 4.29 * A^2 - 1.24 * B^2 - 3.01 * C^2 \tag{1}$$

where *Y* is the LOI of ULDFs and *A*, *B*, and *C* are the coded variables for the dose of B-Zn-Si-Al compounds, CP, and PVDC, respectively.

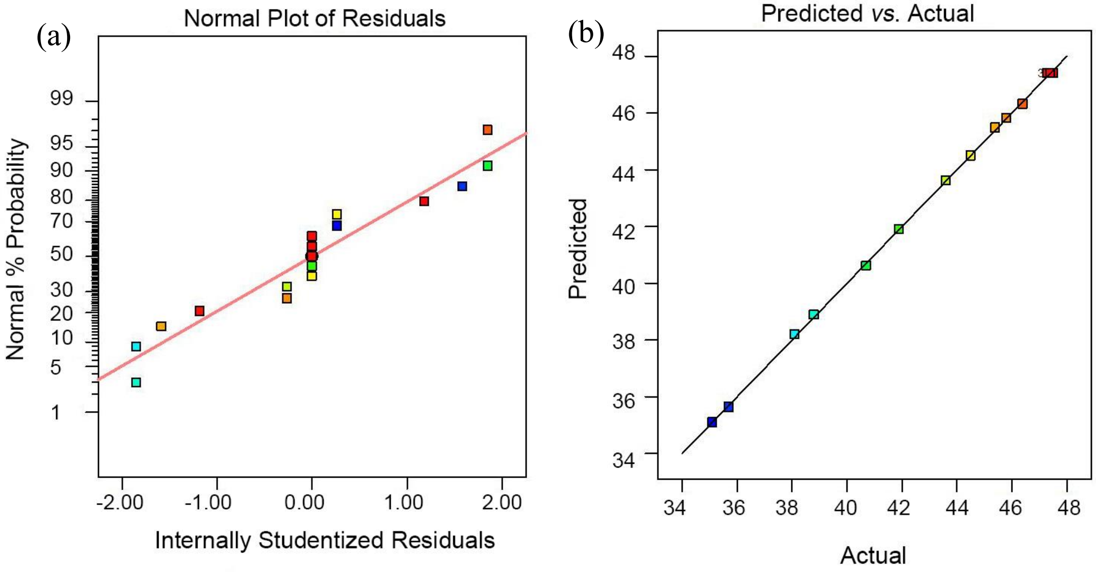

Fig. 2. (a) Normal probability plot of residual dilution; (b) Correlation between experimental values and predicted values

Figure 2 (a) shows the normal probability plot of the residual dilution, which illustrated that the residuals fell near the straight line, which depicted that the errors were normally dispersed. In Table 2, each of the experimental values was contrasted with the predicted values from the model. Each experimental value assessed by the predicted values is shown in Fig. 2 (b). All of the facts mentioned above indicated an outstanding sufficiency of the regression model.

Optimization of LOI

The fitted response surface for LOI of ULDFs by the above empirical model was generated using Design Expert software and is given in Figs. 3, 4, and 5.

As shown in Fig. 3, the LOI of ULDFs increased first, then decreased with increased doses of B-Zn-Si-Al. The amounts of B-Zn-Si-Al had an important influence on the response value *Y*. The LOI of ULDFs was at the lowest point (35.1), when the addition of B-Zn-Si-Al reached 600 mL. This may have been because the low amount of B-Zn-Si-Al could not promote the formation of a three-dimensional network structure. As a result, a massive loss of wood fibers and fines that were drained out with the water led to an increase in the white water concentration and deteriorating flammability (Wu *et al.* 2016b). With increased amounts of B-Zn-Si-Al, the LOI rose to the maximum then began to drop. This was because the B-Zn-Si-Al would cause agglomeration between the particles, which undermined the function of the adsorption bridging action and sweep flocculation. The LOI had an ascending trend that changed to a smooth trend with increased CP. The contour lines were near elliptical, which indicated that the interaction effects of B-Zn-Si-Al and CP were significant.

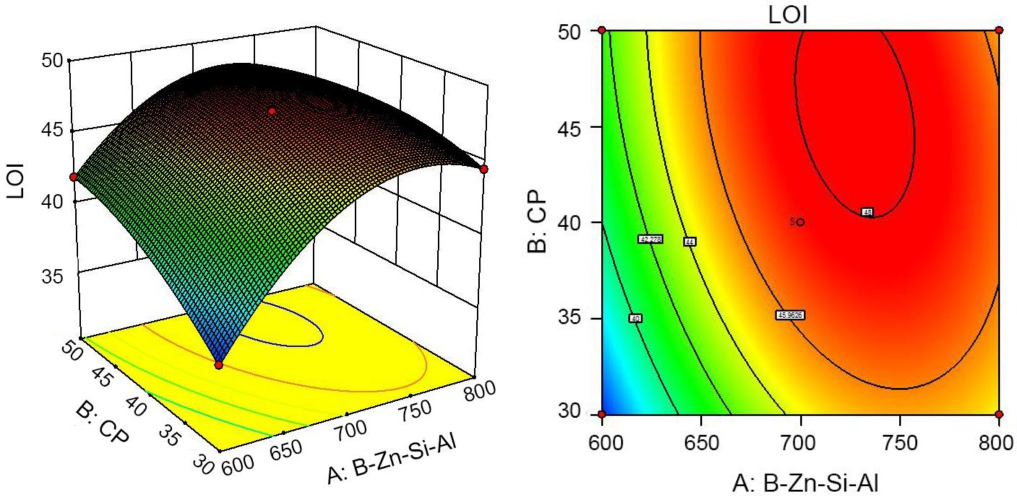

Fig. 3. 3D Response surface plots and contour lines showing the effects of the amounts of B-Zn-Si-Al and amounts of CP on the LOI of specimens

From Fig. 4, the LOI of ULDFs increased in the beginning, then it declined with the increase of both PVDC and B-Zn-Si-Al. The LOI reached the highest point when these two variances remained close to zero. The reason may have been that PVDC's weak acidity could change the film-forming environment of B-Zn-Si-Al, which could weaken absorption and bridge effects, and caused a massive loss of flame retardance when more PVDC was added. The contour lines appeared round, which indicated that the interaction effects of B-Zn-Si-Al and CP were not significant.

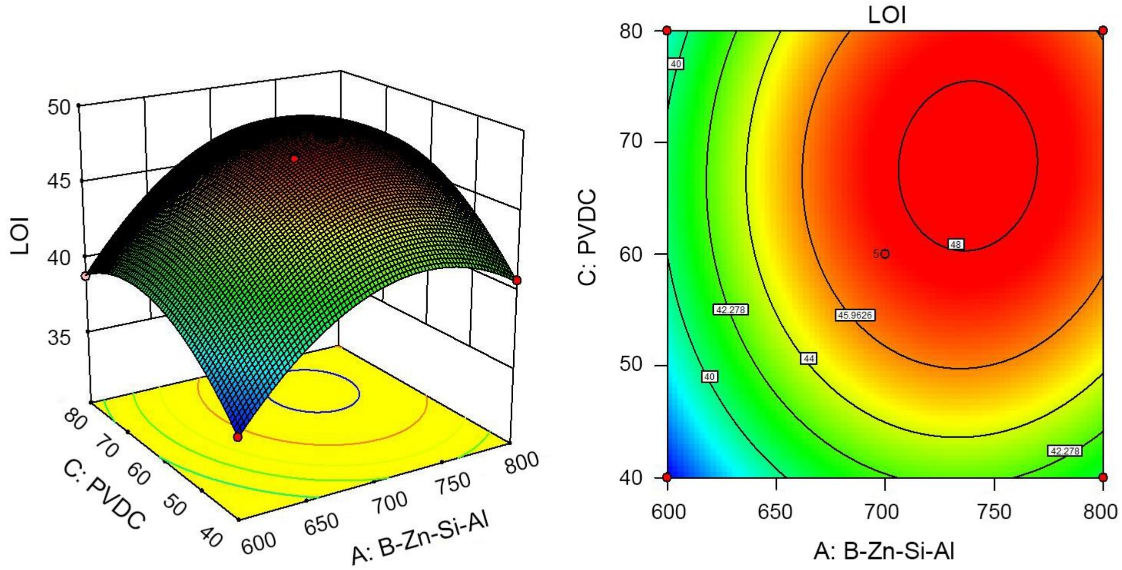

Fig. 4. 3D Response surface plots and contour lines showing the effects of the amounts of B-Zn-Si-Al and amounts of PVDC on the LOI of specimens

Figure 5 shows the effect of the interaction of CP and PVDC on the LOI. It was evident that the LOI experienced growth with increased CP, while the LOI increased in the first stage, and then decreased with the increase of PVDC. The contour lines took on an elliptical shape, which indicated that the interaction effects of PVDC and CP were significant.

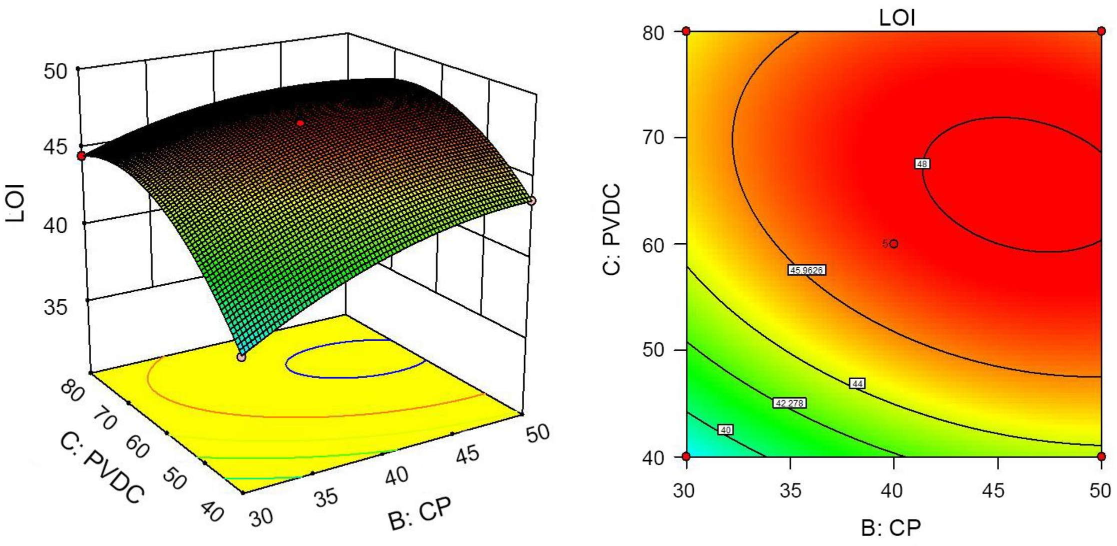

Fig. 5. 3D Response surface plots and contour lines showing the effects of the amounts of CP and amounts of PVDC on the LOI of specimens

Validation of the Models

The process parameters of the fire retardant ULDFs were studied and optimized by RSM. The optimum technological conditions were as follows: the usage of B-Zn-Si-Al, PVDC, and CP were 800 mL, 46.47 mL, and 35.64 g, respectively. The LOI of the average of 10 parallel experiments of the final product was 48.4, under the optimum conditions. The result was approximately the predicted value in Eq. 1, which indicated that the model of Eq. 1 was accurate, and further verified the practicality of this optimum strategy.

CONCLUSIONS

Based on the three factors and three levels of the Box-Behnken Design, RSM was utilized to optimize the manufacturing conditions that enhanced the LOI.

1. The results indicated that RSM provided a practical and useful method for fire retardant optimization. From the results, the technique not only assisted in discovering the prime levels of the most noteworthy factors considered with the least resources and time, but also proved to be effective and reasonable in this process-optimizing experiment.
2. The optimal usage of B-Zn-Si-Al, PVDC, and CP were 800 mL, 46.47 mL, and 35.64 g, respectively. In this situation, the maximum LOI was 48.4.

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