### Optimization of production process of epoxidized soybean oil with high oxygen content through response surface methodology

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The epoxidation process of soybean oil (SBO) and peracid produced by 50 wt% hydrogen peroxide ( $H_2O_2$ ) and formic acid (FA) was studied with sulfuric acid ( $H_2SO_4$ ) as a catalyst. Three reaction parameters, including reaction temperature, FA-to-SBO ratio, and  $H_2O_2$ -to-SBO ratio, were investigated, along with the combined effect on oxirane value (OV). Based on response surface methodology (RSM), the Box-Behnken design (BBD) was used to optimize the process parameters. According to the results, the calculated OV (7.34%) and the experimental OV (7.31%) were significantly in agreement. The product was confirmed as epoxidized soybean oil (ESO) by IR and NMR characterization methods. These results demonstrated the reliability of RSM to optimize the SBO reaction to produce ESO with high oxygen content.

Keywords: soybean oil; response surface methodology; epoxidation; oxirane value.

### **INTRODUCTION**

Unsaturated triglycerides are the main constituents of soybean oil (SBO), and their epoxidation is one of the most commercially important reactions, as SBO epoxides have a wide variety of applications<sup>1</sup>. Epoxy rings exhibit a high reactivity, therefore they can be used not only as intermediates for polyols, alkanol amines, polyure-thanes, and epoxy resins but also directly as solubilizers, plasticizers, lubricants, toughening agents and so on<sup>2, 3</sup>. Industrially, the epoxidation of SBO is currently performed with percarboxylic acids, which are usually produced by oxidizing carboxylic acids with hydrogen peroxide (H<sub>2</sub>O<sub>2</sub>) *in situ*. Catalysts for this reaction are usually inorganic acids or acidic ion exchange resins<sup>4</sup>. And the schematic diagram of the SBO epoxidation process is shown in Fig. 1.



Figure 1. Schematic illustration of the epoxidation of SBO

The production of the high oxygen content of epoxidized soybean oil (ESO) is strongly influenced by several reaction variables. In addition to the main reaction of epoxy ring formation, many side reactions directly lead to a decreased oxirane value (OV), the ring-opening reactions of epoxy groups are one example. In 2005, Campanella et al.<sup>5</sup> studied epoxy ring-opening reactions for the liquid–liquid system, which was comprised of acetic acid,  $H_2O_2$ , and peroxyacetic acid. As a result, the ring-opening reactions of the oxirane ring by  $H_2O_2$  in the presence of acid are fast. In 2008, Cam-

panella et al.<sup>6</sup> further found that the rate of ring-opening reaction caused by acetic acid and peracetic acid was the fastest compared to other ring-opening reactions in solution. In 2018, Cai et al.<sup>7</sup> found since acetic acid is continuously regenerated in the reaction solution, its concentration is always higher than peracetic acid's, so the effect of carboxvlic acid on the ring-opening of oxirane ring is greater than that of percarboxylic acid. Although acetic acid has a lower ring-opening rate for oxiranes than FA because of its mild nature, it also inhibits double-bond epoxidation. In 2020, Santacesaria et al.8 conducted a detailed investigation of SBO epoxidation when FA and acetic acid were used as oxidants, and established their respective kinetic models, which showed that during epoxidation reactions in which formic acid (FA) served as the oxidant, the double bond conversion and epoxidation yield were higher than those of acetic acid so that short reaction times could be achieved. In conclusion, under suitable reaction conditions, FA provides more advantages as an oxidant for the epoxidation of SBO than are offered by acetic acid.

Normally, the researchers prefer to use the parameterat-a-time method when process parameters need to be optimized. Though this method has some obvious limitations, for example, the interaction between parameters cannot be displayed, and the number of experiments increases as more parameters are measured<sup>9</sup>. The response surface methodology (RSM) could solve these problems easily, and it is used to efficiently optimize experimental parameters<sup>10</sup>. Box-Behnken design (BBD) is a class of rotatable or almost rotatable second-order designs based on a three-level incomplete factorial design<sup>11</sup>. Compared with other response surface designs (central composite, Doehlert matrix and three-level full factorial design), BBD has two main advantages<sup>12, 13</sup>. One advantage is that BBD has a higher response efficiency, and the other advantage is that it does not contain all combinations of factors at the highest or lowest level at the same time, in other words, unsatisfactory experimental results under extreme conditions can be avoided. So far, there are few reports on the application of RSM to optimize the process of vegetable oil

epoxidation reactions. Moreover, the application of BBD in SBO epoxidation has not yet been reported.

In this study, the RSM was used to optimize the reaction process parameters of FA and high-concentration  $H_2O_2$  as oxidants to generate high oxygen content ESO in a short time. Moreover, to obtain the optimum conditions for producing the maximum oxygen content ESO, the significance of the three sensitive reaction parameters, including the reaction temperature, FA-to-SBO ratio and  $H_2O_2$ -to-SBO ratio, and their combined effects on the OV of epoxidized soybean oil were investigated.

### **EXPERIMENTAL**

### Materials

Soybean oil (SBO), FA (85%), and  $H_2O_2$  (50%) was kindly provided by Shengjun New Material Technology Co., Ltd. Sulfuric acid (98%), acetone (AR), hydrochloric acid and ethanol were purchased from Guangzhou Chemical Reagent Co., Ltd. All numbers are given as mass fraction %.

### **Epoxidation procedure**

The epoxidation of SBO was performed *in situ* with FA using a literature method<sup>8</sup>. In a 250 mL three-necked flask, placed in a temperature-controlled oil bath, SBO (50 g) was mixed with an appropriate amount of FA. Subsequently, the mixed solution of  $H_2O_2$  and  $H_2SO_4$  was added to the reaction mixture at the same rate within 15 mins. In addition, the reaction solution was stirred at 600 rpm to obtain a sufficient dispersion of SBO. The oil phase sample was collected after 3 hours of reaction, which was washed with  $Na_2CO_3$  and distilled water to eliminate the influence of free acids. And then dried with the addition of anhydrous  $Na_2SO_4$ . Finally, all dried samples were analyzed for IV and OV.

### Response surface methodology (RSM)

A RSM consists of statistical and mathematical methods that define the optimum parameter values by

Table 1. The range and levels of experimental variables

determining a desirable response or set of responses<sup>14</sup>. The RSM principle is to optimize the response through a series of experiments. In other words, the purpose of the RSM is to obtain a sufficient relationship between a series of independent variables  $(x_n)$  and a response of interest (y). And the purpose of RSM is to optimize the variables within a specific area. Hypothesis testing in RSM is a statistical tool to determine whether a factor is significant <sup>2</sup>. This relationship is usually represented using a low-degree polynomial model. A quadratic regression model was used to fit the experimental data, which can be represented by the following equation (1), as follows<sup>15</sup>:

$$\gamma = \beta_0 \pm \sum_{i=1}^n \beta_i X_i \pm \sum_{i=1}^n \beta_{ii} X_i^2 \pm \sum_{i>j}^n \sum_{j=1}^n \beta_{ij} X_i X_j + e$$
(1)

where  $\gamma$  is the response of interest,  $\beta_0$  and  $\beta_i$  are the intercept and the first-order regression coefficient, respectively.  $\beta_{ii}$  is the quadratic coefficient,  $\beta_{ij}$  is the regression coefficient for the interaction of *i* and *j*,  $X_i$  and  $X_j$  are independent variables, *n* and *e* represent the number of factors and random error, respectively.

#### **Design of experiments**

A Box-Behnken design (BBD) method was used to analyze and optimize the variables of the SBO epoxidation reaction<sup>2</sup>. In this paper, the OV was selected as the response obtained from the epoxidation of SBO. And the three process variables that have the greatest influence on the OV values were selected as A: reaction temperature, B: FA-to-SBO ratio, and C:  $H_2O_2$ -to-SBO ratio. In previous work, Single-factor experiments were done for each of these three independent variables, and the results showed no linear relationship between the variables and the response value (OV). And the appropriate range and level of independent parameters were determined from these results as shown in Table 1. Table 2 shows the detailed experimental design of the 17 experiments which were designed with the BBD method.

Factor	Description	Unit	Minimum	Middle	Maximum
A (Numeric)	Reaction Temperature	°C	60	70	80
B (Numeric)	FA : SBO	mol : mol	0.4 : 1	0.8 : 1	1.2 : 1
C (Numeric)	$H_2O_2$ : SBO	mol : mol	1:1	5:1	9:1

Table 2. Experimental parameters were employed for the SBO epoxidation process and the OV obtained through this reaction

Run	Reaction temperature	FA : SBO (mol / mol)	$H_2O_2$ : SBO (mol / mol)	OV (%)	
1	60	0.8	9	6.46 ± 0.06	
2	80	0.4	5	6.56 ± 0.06	
3	60	0.4	5	6.83 ± 0.06	
4	70	1.2	9	5.82 ± 0.06	
5	70	0.8	5	6.79 ± 0.06	
6	80	1.2	5	5.15 ± 0.06	
7	60	0.8	1	$2.46 \pm 0.06$	
8	70	0.8	5	6.79 ± 0.06	
9	60	1.2	5	6.20 ± 0.06	
10	70	0.8	5	6.79 ± 0.06	
11	70	0.8	5	6.78 ± 0.06	
12	70	0.8	5	6.80 ± 0.06	
13	80	0.8	1	1.70 ± 0.06	
14	80	0.8	9	5.56 ± 0.06	
15	70	0.4	1	2.87 ± 0.06	
16	70	1.2	1	1.17 ± 0.06	
17	70	0.4	9	6.10 ± 0.06	

### **Analytical methods**

A standard HBr-acetic acid and Hanus method were used to determine the OV and IV of each sample, respectively<sup>16</sup>. The OV and IV values were calculated by Eq. (2) and Eq. (3), respectively:

$$OV = \frac{(V_1 - V_2) \times N \times 0.016}{W} \times 100$$
 (2)

where  $V_1$  and  $V_2$  denote the volume of NaOH solution to titrate the blank sample and the test sample (ml), respectively; N denotes the concentration of NaOH solution (mol/L); and W denotes the mass of the test sample (g).

$$IV = \frac{(V_1 - V_2) \times N \times 0.1269}{G} \times 100$$
(3)

where  $V_1$  and  $V_2$  denote the volume of Na<sub>2</sub>S<sub>2</sub>O<sub>3</sub> solution to titrate the blank sample and the test sample (ml), respectively; *N* denotes the concentration of Na<sub>2</sub>S<sub>2</sub>O<sub>3</sub> solution (mol/l); and *G* denotes the mass of the test sample (g).

The SBO's conversion was calculated by Eq. (4):

$$Conversion(\%) = \frac{IV_0 - IV}{IV_0} \times 100\%$$
(4)

Where  $IV_0$  and IV refer to the iodine value of SBO (g/100 g of oil) and the iodine value of the product (%), respectively.

An FT-IR spectrometer (Thermo Scientific Nicolet iS20, USA) was used to record infrared spectra between 4000–400 cm<sup>-1</sup>. Nuclear magnetic resonance (NMR) spectra (<sup>1</sup>H NMR and <sup>13</sup>C NMR) were recorded using Bruker AVANCE NEO 500 NMR spectrometers. <sup>1</sup>H and <sup>13</sup>C NMR spectra were recorded at frequencies of 500 and 125 MHz, respectively.

### **RESULTS AND DISCUSSION**

The experimentally measured initial iodine value (IV<sub>0</sub>) of the SBO was 128.8  $\pm$  2.0 g meaning 0.50  $\pm$  0.008 mol of double bond per 100 g of SBO. And the main components and contents of SBO are shown in Table 3 <sup>17, 18</sup>.

#### Statistical analysis of data for epoxidation of SBO

The results of the OV are shown in Table 4. The quadratic equation for the OV () according to the coded variables (A, B and C as reaction temperature, FA-to-SBO ratio, and H<sub>2</sub>O<sub>2</sub>-to-SBO ratio) are stated by Eq. (5).

Table 3. Characterization of the SBO

Companyitien	Contont		
Composition	Content		
lodine value	128.8 ± 2.0 g/(100g of SBO)		
[H <sub>2</sub> O]	160 ppm		
Monoglycerides	0.84 wt%		
Diglycerides	0.12 wt%		
Triglycerides	0.12 wt%		
Acidity	0.11 wt%		
C16:0 (palmitic)	9.93 wt%		
C18:0 (stearic)	3.83 wt%		
C18:1 (oleic)	21.1 wt%		
C18:2 (linoleic)	57.7 wt%		
C18:3 (linolenic)	7.06 wt%		
C20:0 (arachidic)	0.29 wt%		

 $\gamma = 6.79 \cdot 0.3725A \cdot 0.5025B + 1.9675C \cdot 0.195AB \cdot 0.035AC +$  $+ 0.355BC \cdot 0.275A^2 \cdot 0.33B^2 \cdot 2.47C^2$ (5)

The statistical analysis for epoxidation of SBO was done using analysis of variance (ANOVA)<sup>19</sup>. In addition, ANOVA was also used to quantify the independent input variables' effects on the output. Both P-values and Fvalues are very important in evaluating the significance of regression coefficients to obtain a statistically significant regression model. The variance of the data relative to the mean is defined based on the F-value of the ratio of the mean square of the group variance to the error. A high F-value indicates variation in the response in the regression equation, but the significance of independent variables is confirmed by P < 0.05. Table 4 shows the ANOVA results for SBO epoxidation with regard to the oxirane value. The results indicated that the regression model was statistically significant because the F-value was 3189.46 and the *P*-value < 0.0001. Therefore, this regression model demonstrates that reaction variables and oxirane values are actually related. The results of regression analysis proved the linear model terms A, B, C, AB, BA,  $A^2$ ,  $B^2$  and  $C^2$  have significant effects on OV. In addition, the regression model also investigates the coefficient of determination  $R^2$  and adjusted  $R^2$ . Based on the high  $R^2$  value (0.9998), the experimental data and the model are statistically well-fit, and also show a variability of 99.98% in this experimental data. However, to avoid this undesirable effect, an adjusted  $R^2$  value is used since the  $R^2$  value increases as the number of variables predicted by the model increases. It plays an important role in illustrating the consistency between the model and the experimental data.

Source of variation	DF	Sum of	Mean	F-Value	P-Value (Prob >	
Model	9	62 13	6.9	3189.46	< 0.0001	Significant
A - reaction temperature	1	1.11	1.11	512.89	< 0.0001	olgimount
B - FA : SBO	1	2.02	2.02	933.36	< 0.0001	
C - H <sub>2</sub> O <sub>2</sub> : SBO	1	30.97	30.97	14308.85	< 0.0001	
AB	1	0.1521	0.1521	70.28	< 0.0001	
AC	1	0.0049	0.0049	2.26	0.1761	
BC	1	0.5041	0.5041	232.92	< 0.0001	
$A^2$	1	0.3184	0.3184	147.13	< 0.0001	
$B^2$	1	0.4585	0.4585	211.86	< 0.0001	
$C^2$	1	25.69	25.69	11869.04	< 0.0001	
Residual	7	0.0152	0.0022			
Lack of Fit	3	0.0150	0.0050	99.67	0.0003	Significant
Pure Error	4	0.0002	0.0000			
Cor Total	16	62.14				
<i>R</i> -squared	0.9998					
Adj <i>R</i> -Squared	0.9994					

Table 4. Process and product parameters of SBO epoxidation in the literature



Figure 2. Residual plots: (a) plot of actual OV versus predicted OV, (b) normal probability distribution of studentized residuals, (c) plot of predicted OV versus studentized residual, (d) Plot of experiment trials versus studentized residuals

Figure 2 (a) indicates that there is an acceptable consistency between the predicted and the actual OV. Moreover, there is a linear relationship between the experimental and calculated OVs based on the similarity between the predicted  $R^2$  value (0.9961) and the adjusted  $R^2$  value (0.9994). The results demonstrated that the model gave good predictions for the responses within the studied range. In addition, the accuracy of the model is confirmed by analyzing residual plots.

Residuals are defined as the deviation between the actual response and the calculated response<sup>19</sup>, so there is one residual for each experimental data. The calculation method for studentized residuals is to divide the normalized residuals by their standard deviations, where the residuals are fitted with a normal distribution function. Figure 2 (b) shows that the residuals have a normal distribution because the normal plot of residuals shows a straight line. Therefore, the errors associated with the quadratic model are normally distributed and negligible.

Figure 2 (c) shows the predicted OV versus studentized residuals. According to Fig. 2 (c), the original experimental observations are independent of the response, as evidenced by the scattered distribution of data points. Hence, it can be concluded that the regression model adequately describes the reaction process. Figure 2 (d) is a graph of experimental data plotted against residual values, indicating the degree of deviation between the actual values and predicted values through the residual values for each operation. The fitted regression model approximates the response surface well because the residuals are in the range of  $\pm$  4. The random distribution of the regression model.

The regression model also shows the lack of fit test, which indicates that the model can adequately describe the relationship between input variables and responses. In this study, the lack of fitting parameters in the model shows that the *F*-value is 99.67 and the *P*-value is 0.0003.

From the results, it was indicated that the quadratic regression model fits the experimental data perfectly.

## Interactive effect of reaction temperatures and FA-to-SBO ratio on OV

Reaction temperatures and FA-to-SBO ratio are critical processing variables that influence OV the most. In addition, both of these variables have an important effect in controlling the reaction rate. The peroxidation of  $H_2O_2$ is a reversible reaction. By increasing the amount of FA, the production of performic acid is more favorable. However, increasing the amount of carboxylic acid also accelerates the epoxy ring-opening reaction<sup>8</sup>. Therefore, the FA-to-SBO ratio was increased from 0.4:1 to 1.2:1 (mol:mol) and reaction temperatures were varied between 60 and 80 °C to investigate their combined effect on OV. Figure 3 shows the interactive effects that were observed between the FA-to-SBO ratio and reaction temperature while fixing all other parameters. This result illustrates that OV decreases with the increase of reaction temperature and the amount of FA, which is consistent with a previous report in the literature<sup>20</sup>. Because the epoxy ring opening reaction takes place on the contact surface between the SBO phase and the water phase, the increase in the amount of FA will increase the contact area of the two phases under the action of stirring. In addition, the increase in temperature will also accelerate the mass transfer rate of the reaction solution, resulting in the opening of more epoxy rings. Similar results were observed by Zaher and coworkers<sup>21</sup>.

# Interactive effect of reaction temperatures and $H_2O_2$ -to-SBO ratio on OV

The 3D and outline plot of the interactive effect of reaction temperature and  $H_2O_2$ -to-SBO ratio on OV is shown in Fig. 4. The  $H_2O_2$ -to-SBO ratio has a critical influence on the OV, as it provides active oxygen as an oxidant in the reaction system. According to the literature, the decomposition rate of  $H_2O_2$  will increase at higher temperatures<sup>22</sup>. Therefore, it is observed from Fig. 4 that the OV decreases in response to an

increasing reaction temperature, resulting from a lower oxidant concentration because of the decomposition of  $H_2O_2$ . On the other hand, the OV first increased and then decreased in response to a greater amount of  $H_2O_2$ . A hydrogen peroxide molecule attacks an epoxy ring similar to a hydrolysis reaction, but it will attack the epoxy ring at a very fast rate when the solution is acidic<sup>23</sup>. In addition, it can be indicated from contour plots that the amount of hydrogen peroxide has a greater effect on the ring opening of the epoxy ring than that of temperature.

# Interactive effect of FA-to-SBO ratio and $H_2O_2$ -to-SBO ratio on OV

An illustration of the mutual interaction between the FA-to-SBO ratio and the  $H_2O_2$ -to-SBO ratio is shown in Fig. 5. Also, the interactive effects of these two variables on the OV are similar to the above. The results showed that the OV reached the maximum with an increasing amount of  $H_2O_2$ , and then gradually decreased; However, the OV decreases directly in response to an increasing amount of FA. As shown in Fig. 5, the optimum values of the molar ratio of FA-to-SBO and the molar ratio of  $H_2O_2$ -to-SBO are 0.576 and 6.28, respectively. In addition, the OV will also decrease if the two variables are increased beyond the optimum level.

### **Optimization of production parameters of ESO**

In order to maximize the OV, the process independent variables were optimized by the RSM arithmetic equation. The most favorable calculated OV from the model was 7.34% at a reaction temperature of 66.2 °C, the molar ratio of FA-to-SBO molar ratio is 0.576 : 1 and the molar ratio of H<sub>2</sub>O<sub>2</sub>-to-SBO is 6.28 : 1 as shown by Fig. 6. Several experiments were performed under the optimized process parameters. The average OV obtained from these experiments was 7.31%, which was consistent with the value predicted by the model, and the conversion rate of the double bond was 100%.



Figure 3. The 3D and outline plot of the relationships of the effect of A (reaction temperature) and B (FA-to-SBO ratios) on OV at  $n(H_2O_2)$ : n(SBO) = 5 : 1,600 rpm and 3 h reaction time



Figure 4. The 3D and outline plot of the relationships of the effect of A (reaction temperature) and C ( $H_2O_2$ -to-SBO ratios) on OV at n(FA) : n(SBO) = 0.8 : 1,600 rpm and 3 h reaction time



Figure 5. The 3D and outline plot of the relationships of the effect of *B* (FA-to-SBO ratios) and *C* ( $H_2O_2$ -to-SBO ratios) on OV at 70 °, 600 rpm and 3 h reaction time



Figure 6. Production process optimization plot

### DISCUSSION

Traditional optimization methods, such as the parameter-at-a-time method, are cumbersome in their experiments, misleading in the analysis of experimental results, and unable to investigate the interactions between different factors. The combination of the orthogonal array method and ANOVA can be used to investigate the assignment of experimental factors in a range of experimental tests, but it is unable to fit the results to a regression equation to reach an optimal level by calculating the independent variables<sup>24</sup>. RSD-based BBD is an efficient statistical technique that can perfectly solve the above problems. Compared with orthogonal design and ANOVA, BBD can calculate intermediate-level responses that have not been studied. Therefore, we used three-level BBD to determine the optimal process by the minimum number of experiments in this paper.

The OV predicted by the model was in strong agreement with the experimentally tested results, and the ANOVA and the lack of fitting parameters of the model indicated the accuracy and generalizability of the model. In this study, the optimal reaction temperature, the molar ratio of FA-to-SBO and the molar ratio of  $H_2O_2$ -to-SBO for the production of high oxygen content ESO by SBO epoxidation were predicted perfectly. Moreover, we investigated the literature for process and product parameters of ESO production by SBO epoxidation under different catalysts, as shown in Table 5. The highest OV (6.99%) of ESO produced by SBO in the literature was lower than the OV (7.31%) in this paper, which indicated the superiority of optimizing the reaction conditions using RSM.

This paper also aims to realize the production of high OV epoxidized SBO on an industrial scale. The exothermic heat generated by the SBO epoxidation reaction is negligible in laboratory studies, but the exothermic heat at the industrial scale level leads to an increase in the temperature of the system. This undoubtedly leads to the fact that the SBO epoxidation process cannot always be secured under optimal process conditions. Therefore, the conventional kettle reactor is unsuitable for this process condition since a rapid heat dissipation capability is unavailable. However, the currently popular microreactors provide perfect heat dissipation capability. This study is of great importance for the application of SBO epoxidation reaction on microreactors.

### **FT-IR** analysis

It is reported<sup>27</sup> that the infrared characteristic peaks corresponding to double bonds in SBO appear at 3008 cm<sup>-1</sup>, and the infrared characteristic peaks of epoxy groups in ESO appear at 823 cm<sup>-1</sup>. The characteristics of the FTIR spectra of ESO from Fig. 7 show that the double bond has disappeared and the epoxy group has



Figure 7. The FTIR spectra of the sample of SBO and ESO

appeared. Therefore, these findings confirm that epoxidation has taken place.

### NMR analysis

The <sup>1</sup>H NMR spectra of virgin SBO and ESO are shown in Fig. 8(a). The conversion of the alkene groups to epoxy groups is evident in the <sup>1</sup>H NMR spectra. Moreover, in the spectrum of SBO, olefin protons are visible in the range of 5.3–5.4 ppm, while in the spectrum of ESO, olefin protons are not observed, but epoxy protons are observed in the range of 2.9–3.0 ppm. On the other hand, from the <sup>13</sup>C NMR spectrum of Fig. 8(b), sp<sup>2</sup> hybridized carbons in olefinic groups in SBO appeared in the range of 125–135 ppm, but it is not observed in the spectrum of ESO. Instead, peaks in the 50–60 ppm range indicate that it is an epoxy carbon atom. In summary, the characteristic peaks of the NMR spectrum indicate the process of epoxidation of SBO to form ESO.



Figure 8. <sup>1</sup>H NMR spectra (a) and 13C NMR spectra (b) of ESO (top) and SBO (bottom)

Table 5. Process and product parameters of SBO epoxidation in the literature

Entry	Catalyst	Reaction conditions	Conversion <sup>a</sup> (%)	OV <sup>a</sup> (%)	Ref.
1	H <sub>2</sub> SO <sub>4</sub> /H <sub>2</sub> O <sub>2</sub> /HCOOH	60 °C, 3.5 h	97	6.99	25
2	Bis(acetyl-acetone) dioxo-molybdenum (VI) [MoO₂(acac)₂]	110 °C, 2 h	70	4.31	26
3	Amberlite IR-120H	65 °C, 10 h	98	6.97	27
4	Amberlite 16	55 °C, 3 h	98	6.40	28
5	Peroxopolyoxometalate [PW <sub>4</sub> O <sub>24</sub> ] <sup>3-</sup> with dicationic long-chain	60 °C, 4 h	97	6.59	29

<sup>a</sup> The results are calculated by the titrimetric method as described in the analytical part.

### CONCLUSIONS

In this paper, using RSM-based BBD, the reaction parameters were optimized to produce ESO with maximum OV. The ANOVA and the lack of fitting parameters indicated that the regression model for the production of ESO was statistically significant. In addition, experiments were carried out under the optimal reaction parameters, and the results verified the parameters' optimality, and the product's OV reached 7.31%. Finally, IR and NMR were used to analyze the products, and the results indicated that ESO was produced by the epoxidation reaction of SBO. The results of this study will help to improve the efficiency of SBO epoxidation reaction to produce high oxygen content ESO, especially for applications on microreactors.

### **DECLARATION OF COMPETING INTEREST**

The authors declare no competing financial interest.

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