

## Research Article

# Optimization of Deep Oxidative Desulfurization Process Using Ionic Liquid and Potassium Monopersulfate

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Response surface methodology (RSM) was selected to optimize a desulfurization process with metal based ionic liquids ([Bmim]Cl/CoCl<sub>2</sub>) and potassium monopersulfate (PMS) together to remove benzothiophene (BT) from octane (simulating oil). The four experimental conditions of PMS dosage, [Bmim]Cl/CoCl<sub>2</sub> dosage, temperature, and reaction time were investigated. The results showed that the quadratic relationship was built up between BT removal and four experimental variables with 0.9898 fitting coefficient. The optimal conditions were 1.6 g (20 wt%) PMS solution, 3.2 g [Bmim]Cl/CoCl<sub>2</sub>, 46°C, and 23 min, which were obtained based on RSM and experimental results. Under the optimal condition, predicted sulfur removal rate and experimental sulfur removal rate were 96.7% and 95.4%, respectively. The sequence of four experimental conditions on desulfurization followed the order temperature > time > [Bmim]Cl/CoCl<sub>2</sub> dosage > PMS solution dosage.

## 1. Introduction

Over the past fifteen years, many developing countries have been suffering from severe haze which seriously affects people's health. One of the reasons is attributed to burning the sulfur-containing fuels and producing sulfur oxide which can form the haze and acid rain to pollute environment [1]. In order to control environmental pollution caused by sulfur oxide, almost all countries tightly limit the sulfur content in fuel oil. In 2016, China enacted the national law to control the sulfur content in fuel oil that must be less than 10 ppm, the same as USA and European countries [2].

In order to remove organic sulfur compound in oil, there are some effective technologies like hydrodesulfurization, adsorption, extraction, oxidation, and so on. Nowadays, ionic liquid attracts more attention in desulfurization field [3] because of its green solvent characteristics [4, 5] and catalytic activity. Zhang et al. [6] used [HDBN]Cl/ZnCl<sub>2</sub> ionic liquid and H<sub>2</sub>O<sub>2</sub> to treat model oil and obtained 98.8% dibenzothiophene (DBT) removal. Safa et al. [7] used acidic ionic liquid loaded on silica gel as catalyst for desulfurization and 75.7% of sulfur removal of real diesel was obtained.

The response surface methodology (RSM) is a mathematical statistical method which is always applied to the process optimization. In conventional multifactor experiments, optimal process is usually carried out by varying one factor and fixing all other factors (i.e., one factor at a time). Alternatively, the RSM enables one to determine the influence of individual factors as well as their interactive influences with a limited number of planned experiments. Thus, the RSM is widely used in various industrial, agricultural, and other research fields [8].

Chen et al. [9] prepared [C<sub>4</sub>mim]Cl/ZnCl<sub>2</sub> ionic liquid and used it to investigate the oxidative removal of sulfur compounds from diesel fuels, with 30 wt% H<sub>2</sub>O<sub>2</sub> solution as oxidant. In this study, the temperature, IL dosage, H<sub>2</sub>O<sub>2</sub> dosage, and reaction time were investigated for desulfurization. Chi et al. [10] also synthesized a functional ionic liquid combined with 30 wt% H<sub>2</sub>O<sub>2</sub> solution for desulfurization and IL dosage, H<sub>2</sub>O<sub>2</sub> dosage, temperature, and reaction time were investigated.

In this study, we prepared a functional [Bmim]Cl/CoCl<sub>2</sub> ionic liquid (IL) with potassium monopersulfate (PMS) to remove benzothiophene (BT) from octane. The effects of

four independent variables of PMS dosage, [Bmim]Cl/CoCl<sub>2</sub>, temperature, and reaction time for desulfurization were investigated based on response surface methodology (RSM) and real experiment.

## 2. Experimental Methods

**2.1. Desulfurization Process.** The [Bmim]Cl/CoCl<sub>2</sub> ionic liquid (IL) was prepared by mixing 0.1 mol CoCl<sub>2</sub>·6H<sub>2</sub>O and 0.1 mol 1-n-butyl-3-methylimidazolium chloride ([Bmim]Cl) at 110°C for 48 h. The model oil was prepared by dissolving benzothiophene (BT) in octane maintaining sulfur content of 500 ppm. The desulfurization tests were carried out in a 40 mL two-necked flask equipped with a stirrer and a condenser. The desulfurization procedure was run as follows: firstly, [Bmim]Cl/CoCl<sub>2</sub> and the simulating oil were added to the flask and the mixture was vigorously stirred for 30 min until the extraction equilibrium; secondly, 20 wt% PMS solution was added to the flask and quickly diffused into the ionic liquid phase. Subsequently, the mixture was vigorously stirred to complete the desulfurization reaction. All the chemicals in this study were purchased from Aladdin chemical reagent company. After reaction, aliquots of the upper phase (model fuel) were obtained. The remaining compounds were determined by GC (Agilent 7820A GC, USA) with a flame ionization detector (FID) and HP-1 capillary column (19091Z-443, 30 m × 0.32 mm × 0.25 μm). The S-removal efficiency was calculated according to (1) as follows:

$$\text{S-removal efficiency (\%)} = \left(1 - \frac{[S]_t}{[S]_0}\right) \times 100, \quad (1)$$

where [S]<sub>0</sub> was the initial S-content and [S]<sub>t</sub> was the S-content at time *t*.

**2.2. Experimental Design for Desulfurization Process.** Experimental design, mathematical modeling, and optimization process were performed using Design-Expert 8.0.6 software. Box-Behnken Design (BBD), one of the most common design methods, was used for the response surface methodology (PMS). The levels of the variables for desulfurization process are shown in Table 1. The experimental data were analyzed by the response surface regression procedure to fit the following second-order polynomial model [11]:

$$Y = \beta_0 + \sum_{i=1}^k \beta_i X_i + \sum_{i=1}^k \beta_{ii} X_i^2 + \sum_{i=1}^k \sum_{j=1}^k \beta_{ij} X_i X_j. \quad (2)$$

Here, *Y* is the predicted response (sulfur removal, %); *X<sub>i</sub>* and *X<sub>j</sub>* are variables (experimental conditions); β<sub>0</sub> is the constant coefficient; and β<sub>*i*</sub> is the coefficient that determines the influence of parameter *i* in the response (linear term), which refers to the effect of the interaction among independent variables.

In this study, [Bmim]Cl/CoCl<sub>2</sub> ionic liquid and PMS were investigated to remove benzothiophene (BT) from octane under different conditions such as initial PMS (20% solution) (1-2 g), [Bmim]Cl/CoCl<sub>2</sub> (2-4 g), temperature (40-60°C),

TABLE 1: Levels of the variables for BBD experimental design.

Variables	Code	Unit	Levels		
			-1	0	1
PMS	A	g	1	1.5	2
[BMIM]Cl/CoCl <sub>2</sub>	B	g	2	3	4
Temperature	C	°C	40	50	60
Time	D	min	5	15	25

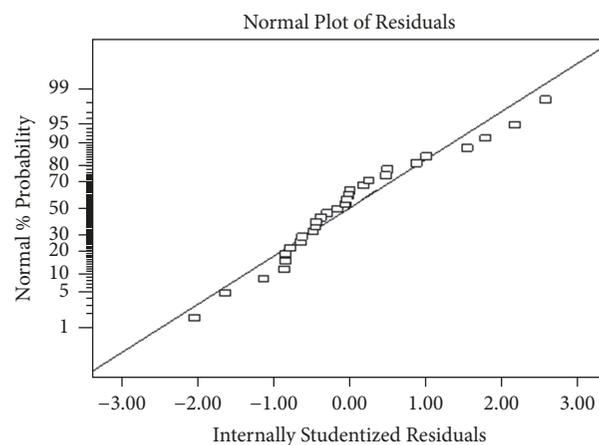


FIGURE 1: Normal probability of residuals.

and time (5-25 min). Based on Box-Behnken Design, the design matrix of coded values for the factors and the response in terms of sulfur removal for all 29 experimental runs are shown in Table 2, in which the amount of PMS, IL, temperature, and time are denoted as *A*, *B*, *C*, and *D*, respectively.

## 3. Results and Discussion

**3.1. ANOVA Analysis.** The relationship between S-removal and variables (*A*, *B*, *C*, and *D*) was obtained and exhibited in (3) by Design-Expert 8.0 software calculation. The experimental results were verified by comparing them with the calculated data (i.e., the predicted values) which were shown in Table 2.

$$\begin{aligned} \text{S-removal percentage} &= 91.94 + 4.23A + 4.57B - 10.27C + 7.72D \\ &\quad - 1.88AB + 5.07AC + 0.70AD + 1.35BC \\ &\quad + 2.37BD + 0.075CD - 16.58A^2 - 9.60B^2 \\ &\quad - 11.83C^2 - 6.55D^2. \end{aligned} \quad (3)$$

Figures 1 and 2 show the diagnostics of the fitted models in terms of comparison between the normality plot of residuals and the predicted and actual values. The residuals were defined as the differences between the experimental and predicted values. As can be seen from Figure 1, the residuals are in a normal distribution because a lot of data are near -1

TABLE 2: Experimental design matrix and results for process optimization.

Run	A	B	C	D	S-removal	S-removal
	PMS/g	IL/g	T/°C	Time/min	Experimental results/%	Predicted values/%
1	1	2	50	15	54.3	55.1
2	2	2	50	15	65.9	67.3
3	1	4	50	15	68.6	68.0
4	2	4	50	15	72.7	72.7
5	1.5	3	40	5	75.6	76.2
6	1.5	3	60	5	55.5	55.5
7	1.5	3	40	25	90.7	91.5
8	1.5	3	60	25	70.9	71.1
9	1	3	50	5	57.2	57.6
10	2	3	50	5	62.1	64.6
11	1	3	50	25	72.7	71.6
12	2	3	50	25	80.4	81.5
13	1.5	2	40	15	76.6	77.6
14	1.5	4	40	15	84.6	84.0
15	1.5	2	60	15	52.3	54.3
16	1.5	4	60	15	65.7	66.2
17	1	3	40	15	74.1	74.6
18	2	3	40	15	75.2	73.0
19	1	3	60	15	43.9	44.0
20	2	3	60	15	65.3	62.6
21	1.5	2	50	5	69.1	65.9
22	1.5	4	50	5	70.5	70.3
23	1.5	2	50	25	78.5	76.6
24	1.5	4	50	25	89.4	90.4
25	1.5	3	50	15	91.9	91.9
26	1.5	3	50	15	93.7	91.9
27	1.5	3	50	15	92.4	91.9
28	1.5	3	50	15	91.2	91.9
29	1.5	3	50	15	90.7	91.9

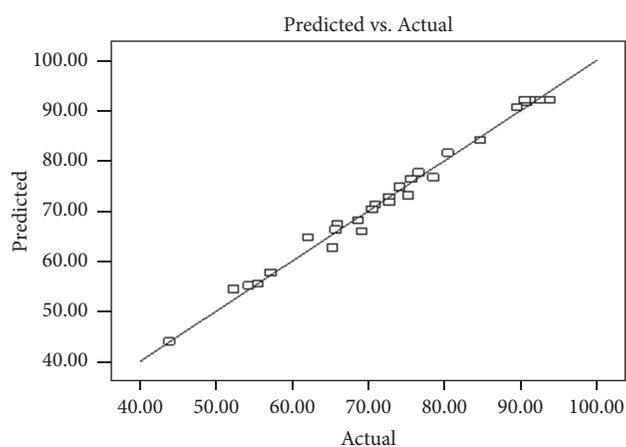


FIGURE 2: Plots of actual response versus predicted response.

to 1 of the  $x$ -axis. From Figure 2, all the data are near the line which meant the predicted values are close to experimental values, indicating that the model is successful in capturing the

correlations between the influencing parameters and sulfur removal.

Table 3 presents the ANOVA results for response surface quadratic model. The significance of each coefficient is determined by  $F$ -value. For the operating parameters, values of “Prob >  $F$ ” are less than 0.05, which indicates model term is significant. In this case,  $A$ ,  $B$ ,  $C$ ,  $D$ ,  $AC$ ,  $BD$ ,  $A^2$ ,  $B^2$ ,  $C^2$ , and  $D^2$  are significant model terms, indicating that all the single parameters have a great impact on desulfurization. The  $AC$  is significant which means that the interaction effect between PMS and temperature exhibits a great influence on desulfurization. The interaction between IL and time is also very strong impact on desulfurization because “Prob >  $F$ ” value of  $BD$  value is lower than 0.05 [12, 13]. In contrast,  $AB$ ,  $AD$ ,  $BC$ , and  $CD$  have negligible effect on the sulfur removal because the “Prob >  $F$ ” values are bigger than 0.05. Based on the SS values of four conditions, the desulfurization efficiencies follow the order: temperature > time > IL > PMS. The Lack of Fit is an important data to evaluate the reliability of the model. If the model is significant, it is not good for fit and needs to be adjusted. If the model is not significant, it is

TABLE 3: Analysis of variance (ANOVA) for response surface quadratic model.

Source	SS	df	MS	F value	Prob. > F
Model	5075.6	14	362.5	96.9	<0.0001
A	215.1	1	215.1	57.5	<0.0001
B	250.2	1	250.2	66.9	<0.0001
C	1264.9	1	1264.9	338.1	<0.0001
D	714.6	1	714.6	191	<0.0001
AB	14.1	1	14.1	3.76	0.0730
AC	103.0	1	103.0	27.5	0.0001
AD	2.0	1	2.0	0.52	0.4811
BC	7.3	1	7.3	1.95	0.1845
BD	22.6	1	22.6	6.03	0.0277
CD	0.02	1	0.02	0.006	0.9393
A <sup>2</sup>	1782.8	1	1782.8	476.5	<0.0001
B <sup>2</sup>	598.2	1	598.2	159.8	<0.0001
C <sup>2</sup>	907.5	1	907.5	242.6	<0.0001
D <sup>2</sup>	278.6	1	278.6	74.5	<0.0001
Residual	52.4	14	3.7		
Lack of fit	46.5	10	4.7	3.13	0.1412
Pure error	5.9	4	1.5		
Total	5128.0	28			

\* CV% = 2.63;  $R^2 = 0.9898$ ; Adj- $R^2 = 0.9796$ .

simulated in an acceptable way. “Prob. > F” is 0.1412 which implies that the “Lack of Fit” is not significant. The “Lack of Fit” could occur which is attributed to noise [14].

The correlation coefficient ( $R^2$ ) [15] can be used to describe degree of correlation between dependent variable (S-removal) and independent variables (A, B, C, and D), which is obtained by Design-Expert 8.0.6 software. The  $R^2$  value in this study is 0.9898, which indicates the correlation model (see (3)) is right. In addition, the  $R^2$  value and adjusted- $R^2$  value (0.9796) are similar which means there is a little chance that nonsignificant term has been included in the model. The coefficient of variance (CV) is another statistic to estimate the ratio of the standard deviation and the averages for observed results and measure the degree of variation for each observation. It also is a kind of evaluation profile of model reproducibility. As a general rule, a model can be considered reasonably reproducible when CV is not greater than 10% [15, 16]. The CV for this model is only 2.63%, showing a good precision and reliability of the experimental runs.

**3.2. Response Surface Plot and Contour Plot.** The three-dimensional response surface plot is useful to determine the individual and cumulative effects of one variable or the mutual interaction between two variables. The response surface analyzes the geometric nature of surface, the maxima and minima of the response, and the significance of the coefficients of the canonical equation [17]. The contour plots help to identify the type of interactions between tested variables. Each contour curve represents an infinite amount of sulfur removal under variation of two tested variables with other variables at the respective zero level.

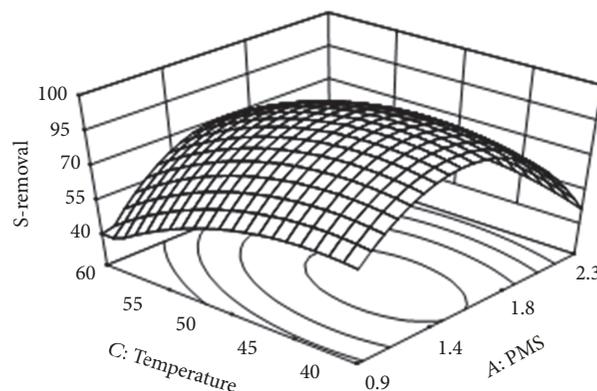


FIGURE 3: The response surface plot about effect of PMS and temperature interaction on S-removal.

Figures 3–6 show three-dimensional response surface plots and contour plots about the relationship between sulfur removal and four variables. From the ANOVA analysis, two experimental variables of PMS solution dosage (A) and temperature (C) could produce interaction effect on S-removal. The ionic liquid (B) and time (D) together also exhibit interaction effect. So, Figure 3 shows the response surface plot which expresses the desulfurization effect of PMS solution dosage (A) and temperature (C) under zero level of ionic liquid (B) and time (D). Similarly, Figure 5 shows the response surface plot on desulfurization effects of ionic liquid (B) and time (D) under zero level of PMS solution dosage and temperature. Figures 4 and 6 show the contour plots.

It can be seen from Figure 3 that sulfur removal increases with the rise of PMS dosage. However, when the PMS

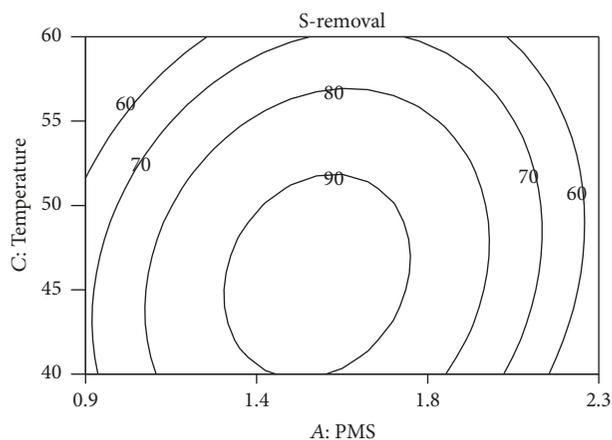


FIGURE 4: The contour plot about effect of PMS and temperature interaction on S-removal.

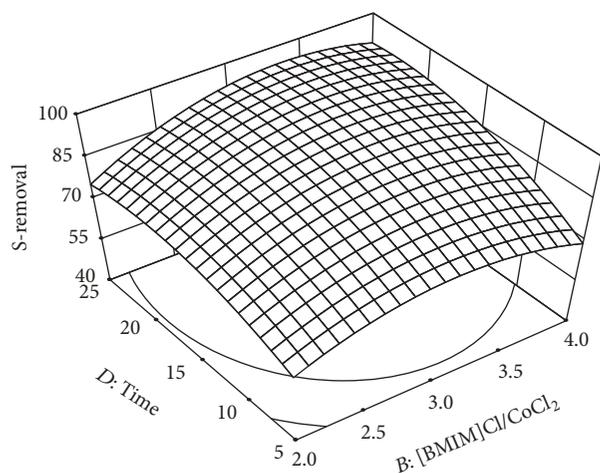


FIGURE 5: The response surface plot about effect of [BMIM]Cl/CoCl<sub>2</sub> dosage and time interaction on S-removal.

dosage is up to a certain value, continuing to increase the PMS dosage will inhibit the desulfurization process and result in sulfur removal down. The reason is that excess peroxosulphate ( $\text{HSO}_5^-$ ) species could act as sulfate radical scavengers decreasing the desulfurization effectiveness [18]. From Figure 3, the temperature also has the optimal value. The reason is that high temperatures are not beneficial for sulfur removal because they negatively affect the catalytic oxidative and extraction processes. Thus, desulfurization is hindered at high temperatures since PMS is unstable and decomposes at temperatures as low as 65°C. PMS loses its oxidative capacity above 60°C. However, high temperatures help reduce the ionic liquid viscosity which is beneficial for achieving a good mixing between the ionic liquid and the fuel. At low temperatures, the viscosity of the ionic liquid is very high, thereby significantly hindering the mixing process between the ionic liquid and the fuel. From the contour plot (Figure 4), contour plot exhibits a closed oval contour curve which means the biggest sulfur removal is at the core of oval. If the temperature is fixed under a certain value,

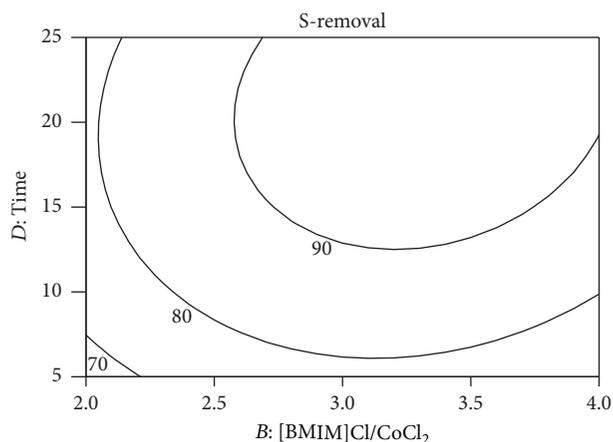


FIGURE 6: The contour plot about effect of [BMIM]Cl/CoCl<sub>2</sub> dosage and time interaction on S-removal.

S-removal exhibited a tendency of increase first and then decrease during the rise of PMS dosage. The same tendency also occurs during the rise of temperature under fixed PMS dosage value. Therefore, the PMS dosage and temperature exist the optimal values.

From Figure 5, it also can be seen that [BMIM]Cl/CoCl<sub>2</sub> (IL) dosage has the optimal value. The reason is that at these high IL loading conditions, cobalt ions in ionic liquid phase might react with PMS to produce more sulfate radical species to oxidize BT molecules. Theoretically, the S-removal efficiency should increase with the IL dosage. However, if ionic liquid dosage exceeds the optimal value, excess cobalt ions can act as free radical scavengers consuming sulfate radicals and resulting in a poor desulfurization performance [19, 20]. In Figure 6, the contour plot exhibits S-removal which expresses a tendency of first increase and then decrease during rise of IL dosage under fixed time. Reaction time is also an important condition that should be investigated. From Figure 5, the S-removal would have almost no change after a certain time under a fixed ionic liquid.

The optimal experimental conditions can be rapidly determined from the response surface plots under only 29 experiment points. The RSM is a convenient method to help us to search the optimal condition.

### 3.3. Desulfurization Mechanism and Process Optimization.

The possible mechanism of desulfurization is that firstly BT is extracted by [BMIM]Cl/CoCl<sub>2</sub> from octane phase to IL phase. Secondly, BT is oxidized in IL phase by sulfate radical which is from the reaction of  $\text{Co}^{2+}$  and PMS. The optimized conditions and predicted sulfur removal are shown in the Table 4. Using this optimized experimental condition, the experimental run for IL and PMS oxidation was conducted according to verification and an experimental value obtained is 95.4%. The optimized sulfur removal is 96.7% according to RSM. The result implies that the experimental value obtained is close to the value calculated from the model and the error rate is only 1.3%, which consequently verifies the model capability.

TABLE 4: Optimization criteria for sulfur removal.

Reaction condition	Value
PMS/g	1.6
IL/g	3.2
Temperature/°C	46
Time/min	23
Predicted sulfur removal/%	96.7
Experimental sulfur removal/%	95.4

#### 4. Conclusion

A functional [Bmim]Cl/CoCl<sub>2</sub> ionic liquid (IL) together with PMS exhibited a high sulfur removal. The quadratic relationship between benzothiophene removal and four experimental conditions was built up based on Box-Behnken Design (BBD) using RSM with 0.9898 fitting coefficient. The optimum conditions were 1.6 g (20% wt) PMS solution, 3.2 g [Bmim]Cl/CoCl<sub>2</sub>, 46°C, and 23 min with 96.7% predicted sulfur removal and 95.4% experimental sulfur removal. The order of influence of experimental conditions is temperature > time > [Bmim]Cl/CoCl<sub>2</sub> dosage > PMS solution dosage.

#### Conflicts of Interest

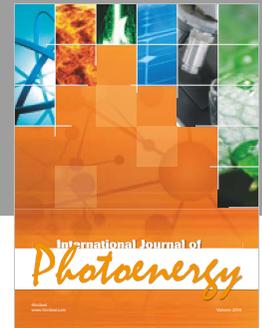
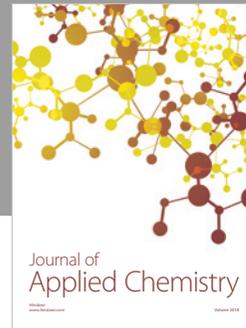
The authors declare that they have no conflicts of interest.

#### Acknowledgments

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