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# Optimization of coagulation–flocculation process for combined sewer overflow wastewater treatment using response surface methodology

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ABSTRACT

A different type of combined sewer overflow (CSO) problem in dry weather, with characteristic of high organic pollutant loads and wide variations, has been becoming one of the most serious urban river pollution problems in China. The performance of coagulation–flocculation process in this type of CSO wastewater treatment was investigated in this study, using polyaluminum ferric chloride sulfate (PAFCS) as coagulant. A $2^3$ full-factorial central composite design and response surface methodology were applied to evaluate the effects and interactions for the chemical oxygen demand (COD) removal efficiency by three factors including initial COD concentration, initial suspended solid concentration, and coagulant dosage. A quadratic model was obtained and the analysis of variance results indicated clearly that experimental data could fit the equation well with a $R^2$ of 95.15%. There is a significant interaction between the initial COD concentration and coagulant dosage for COD removal efficiency. The experimental data and model predictions agreed well. The quadratic model was demonstrated to be an appropriate approach in prediction of the coagulant dosage or COD removal efficiency in this type CSO wastewater treatment using coagulation–flocculation process.

*Keywords:* Combined sewer overflow; Coagulation–flocculation process; Quadratic model; Response surface methodology

## 1. Introduction

Combined sewer overflow (CSO) has been becoming a major environmental concern in cities with a combined sewer system around the world. These overflows contain not only storm water but also untreated human and industrial waste, toxic materials, and debris, and could adversely affect water quality in the urban areas [1]. This type of CSO events has been reported in many countries, such as the United States, Canada, Spain, France, Japan, and Korea [2–7]. However, a different type of CSO was noticed in China recently. A part of untreated wastewater was discharged causally and directly to nearby rivers via the storm water pipes in a separate sewer system even during a dry weather period. It mainly occurs at the following two situations: (1) In some rapidly developing urban areas, the

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construction of a new separate sewer system where the construction of a wastewater system falling behind a storm water system; (2) In some old areas with buildings in high density, the old combined sewer system was not transformed to a separate sewer system completely, which resulted in some domestic or industrial sewer pipes being connected to the storm water pipes intentionally or unintentionally [8]. Although there are interception pumps at the end of the storm sewer system, when the wastewater volume exceeds the capacity of the pump transmission capacity, the excessive wastewater has to be discharged directly into the nearby rivers even during a dry weather. The pollutant concentrations of this CSO event are much higher than those of the traditional CSO events that occurred during heavy rainfall period. The chemical oxygen demand (COD) and suspended solid (SS) concentration of this CSO event was 310.37 ± 130.25 mg/L and 293.29 ± 175.64 mg/L, and the maximum values could reach 1,205.8 and 1,321.5 mg/L, respectively, in China [9–11]. In contrast, the means of COD and SS concentration were 92 and 165 mg/L in United States [12], 229 and 182 mg/L in Spain [13], 150 and 142 mg/L in Italy [14], and 143 and 107 mg/L in Japan [15], respectively. This type of CSO events has become serious water pollution issues with higher pollution loads and greater variation. It needs a feasible treatment to safeguard the river water quality.

### 1.1. Coagulation–flocculation process

Coagulation–flocculation process has been widely used in CSO wastewater treatment both in bench-scale and pilot-scale applications, as it is efficient and easily to be operated [16–19]. This process was also successfully used in engineering applications [20,21]. The optimization of the influence factors, such as the type and dosage of the coagulants, pH, mixing speed and time, temperature, and retention time, was studied by many researchers [22–25]. But most of the studies were based on the same wastewater quality conditions, especially with the fixed initial COD and SS concentrations. However, the initial COD and SS concentration may be very different at different situations as reported in Guida's study [26]. Since the CSO wastewater quality varied, it is difficult to determine the optimal and economic coagulant dosage or to get a stable efficiency under fixed condition by coagulation–flocculation process.

### 1.2. Response surface methodology

Response surface methodology (RSM) is a mathematical/statistical method which is useful in analyzing the effects of several independent variables on the responses and determining the optimal conditions of desirable responses [27]. It overcomes the limitation of the conventional method which only changes one variable and keeps the others constant each time. The conventional method does not depict the combined effect of all the variables and are also time-consuming because they require a great number of experiments to determine the optimum levels. RSM is a collection of statistical and mathematical techniques useful for developing, improving, and optimizing processes. RSM method has been used in coagulation–flocculation process for different wastewater to optimize the coagulation conditions [28–31].

The optimization of coagulation–flocculation process for the CSO wastewater treatment was investigated in this paper. Central composite design (CCD) and RSM were applied to develop a mathematical correlation for the COD removal, with the initial COD, initial SS concentration, and coagulant dosage as variables, and to evaluate the effects and interactions of the 3 factors. The objective was to provide a useful and reasonable method to predict the coagulant dosage or COD removal efficiency in coagulation–flocculation process for CSO wastewater treatment.

## 2. Materials and methods

### 2.1. Chemicals

Polyaluminum ferric chloride sulfate (PAFCS) was purchased from Changzhou Jianghu Chemical Co. Ltd, Jiangsu China. The $Al_2O_3$ content is 28%. The other reagents were purchased from Sinopharm Chemical Reagent Co. Ltd., Shanghai, China. All the chemicals were analytical grade and used without further purification.

### 2.2. Characterization of CSO wastewater

The water samples employed in coagulation experiments were collected from a municipal pump station in Shanghai. The COD, SS, ammonia nitrogen ($NH_4^+$-N), and total phosphorus (TP) were determined according to standard methods [32].

### 2.3. Jar test

The experiments were performed in a TS6-1 jar-test apparatus with six square beakers (Wuhan Hengling Technology Ltd, China). After adding wastewater and coagulants to 1-L beakers, rapid mixing was applied for 1 min at 250 rpm followed by slow mixing for

30 min at 50 rpm. After 30 min settling, the supernatant was withdrawn from a sampling point located in the middle of the beaker to determine the residual COD concentrations, so that the effect of coagulants could be determined. The initial and treated samples were analyzed repeatedly in order to validate the results. The analytical errors were controlled less than ±5%.

### 2.4. Experimental design and data analysis

A preliminary study on the effect of type and dosage of coagulants, mixing speed and time, and retention time of the coagulation–flocculation process was carried out in order to select the best coagulant and the optimal reaction conditions. The experiments were designed and carried out by single-factor and orthogonal experimental design. The first single-factor experiments were carried out as a preliminary coagulants screening test, and later orthogonal experiments were performed finding the optimal coagulation conditions (coagulant dosage, mixing speed, mixing time, and retention time). Six coagulants were employed in this study, and PAFCS was selected as the experiment coagulant by multi-evaluation, which takes the COD removal efficiency, sludge volumes, and pH variation as evaluation indexes. After the orthogonal experiments, the data were analyzed using analysis of variance (ANOVA), and the statistical significance of the factors toward the selected response of the process was determined by means of an *F*-test. On the basis of the study, COD, SS, and coagulant dosage were selected as the variable factors for optimization. The ranges of COD and SS concentration were determined from CSO wastewater quality results (Table 1) and the coagulant dosage was determined by pre-experiments.

CCD method is one of the most popular classes of second-order designs. It was suitable for fitting a quadratic surface, which usually works well for the process optimization [33]. A $2^3$ full-factorial design of CCD with six replicates at the central points was employed to fit the second-order polynomial models and to obtain an experimental error in this study. The range and levels of experimental variables investigated in this study are shown in Table 2. Twenty runs were required for a complete set of the experimental design and are shown in Table 3. The COD removal efficiency after coagulation–flocculation process was taken as the response. The quadratic equation model is expressed by Eq. (1).

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

where $Y$ is the response (dependent variable); $\beta_0$ is constant coefficient; $\beta_i$, $\beta_{ii}$, and $\beta_{ij}$ are coefficients for the linear, quadratic, and interaction effect; $X_i$ and $X_j$ are factors (independent variables). Design expert software (Version 7.1.3, Stat-Ease, Inc., USA) was used for response surface and counter plotting. Adequacy of the proposed model is then revealed using the diagnostic checking tests provided by ANOVA. The quality of the fit polynomial model was expressed by the coefficient of determination $R^2$. The $R^2$ values provide a measure of how much variability in the observed response values can be explained by the experimental factors and their interactions. These analyses are done by means of Fisher's *F*-test and *p*-value (probability). Model terms were evaluated by the *p*-value with 95% confidence level.

Table 1
Characteristics of CSO wastewater

| Parameters | Unit | Concentration (mean ± S.D.[a]) |
|---|---|---|
| COD | mg/L | 290.24 ± 87.37 |
| SS | mg/L | 142.60 ± 85.52 |
| $NH_4^+$-N | mg/L | 16.46 ± 5.47 |
| TP | mg/L | 4.36 ± 1.36 |
| pH | – | 6.87 ± 0.58 |

[a] Standard deviation.

## 3. Results and discussion

### 3.1. COD removal efficiency

The three-level experiments were carried out according to the CCD experimental plan (Table 3), and the average of COD removal efficiency as the response obtained from the experiments are shown in Table 4. It showed that the COD removal efficiency was different when the variables values were different. The COD removal efficiency varied from 30% to over 50%, and the maximum removal efficiency of COD is about 52%.

### 3.2. Development of regression model equation and validation of the model

Using the experimental results, the following second-order polynomial equation was fitted to the results and obtained in terms of actual factors:

Table 2
Experimental range and levels of the independent variables

| Variables | Range and levels | | | | |
|---|---|---|---|---|---|
| | −alpha | −1 | 0 | +1 | +alpha |
| $A$: initial COD concentration (mg/L) | 171.6 | 200 | 290 | 380 | 408.4 |
| $B$: initial SS concentration (mg/L) | 33.1 | 60 | 145 | 230 | 256.9 |
| $C$: coagulant dosage (mg/L) | 42.10 | 50 | 75 | 100 | 107.9 |

Table 3
CCD design results for the three experimental variables in coded units

| Standard | Factor 1 $A$: initial COD | Factor 2 $B$: initial SS | Factor 3 $C$: coagulant dosage |
|---|---|---|---|
| 1 | −1 | −1 | −1 |
| 2 | 1 | −1 | −1 |
| 3 | −1 | 1 | −1 |
| 4 | 1 | 1 | −1 |
| 5 | −1 | −1 | 1 |
| 6 | 1 | −1 | 1 |
| 7 | −1 | 1 | 1 |
| 8 | 1 | 1 | 1 |
| 9 | −1.316 | 0 | 0 |
| 10 | 1.316 | 0 | 0 |
| 11 | 0 | −1.316 | 0 |
| 12 | 0 | 1.316 | 0 |
| 13 | 0 | 0 | −1.316 |
| 14 | 0 | 0 | 1.316 |
| 15 | 0 | 0 | 0 |
| 16 | 0 | 0 | 0 |
| 17 | 0 | 0 | 0 |
| 18 | 0 | 0 | 0 |
| 19 | 0 | 0 | 0 |
| 20 | 0 | 0 | 0 |

$$\begin{aligned}Y_{\mathrm{COD}} = &-40.41 + 0.39A + 0.21B + 0.45C - 0.00011AB \\ &- 0.00104AC - 0.00029BC - 0.00057A^2 \\ &- 0.00051B^2 + 0.00004C^2 \quad (R^2 = 95.15\%)\end{aligned} \tag{2}$$

The results of the second-order response surface model for COD removal efficiency were shown in Table 5. The quadratic regression model demonstrates that the model is highly significant as the Fisher $F$-test ($F_{\mathrm{model}}$, mean square regression/mean square residual = 21.90) with a very low probability value [$(P_{\mathrm{model}} > F) < 0.0001$]. The main effect of initial COD ($A$), initial SS ($B$), and coagulant dosage ($C$), the second-order effect of initial COD ($A^2$) and initial SS ($B^2$), and the two-level interaction of initial COD concentration and coagulant dosage ($AC$) are the significant model terms. Other model terms are not significant. The fit of the model was checked by the determination coefficient ($R^2$). In this case, $R^2 = 0.9515$ indicates that only 4.85% of the total variation is not explained by the model. The value of the adjusted determination coefficient (adjusted $R^2 = 0.9078$) is also high to advocate a high significance of the model [33]. A higher value of the correlation coefficient ($R = 0.9754$) justifies an excellent correlation between the independent variables. Simultaneously, a relatively low value of the coefficient of variation (CV = 3.92%) indicates good precision and reliability of the experiments [34].

The ANOVA results indicated that there was a significant interaction between the initial COD concentration and coagulant dosage for COD removal efficiency ($P_{AC} = 0.0032$). It was reported that increasing the carbon fraction within a floc leads to a significant change in floc structure [35]. Walker found that natural organic matter (NOM) leads to form smaller flocs when added to iron oxide suspensions flocculated with ionic salts

Table 4
Experiment results for three experimental variables in actual units

| Standard | Factor 1<br>*A*: initial COD | Factor 2<br>*B*: initial SS | Factor 3<br>*C*: coagulant dosage | Response<br>COD removal (%) |
|---|---|---|---|---|
| 1 | 200.00 | 60.00 | 50.00 | 33.67 |
| 2 | 380.00 | 60.00 | 50.00 | 36.10 |
| 3 | 200.00 | 230.00 | 50.00 | 43.72 |
| 4 | 380.00 | 230.00 | 50.00 | 36.10 |
| 5 | 200.00 | 60.00 | 100.00 | 44.73 |
| 6 | 380.00 | 60.00 | 100.00 | 37.70 |
| 7 | 200.00 | 230.00 | 100.00 | 50.76 |
| 8 | 380.00 | 230.00 | 100.00 | 37.70 |
| 9 | 171.55 | 145.00 | 75.00 | 42.77 |
| 10 | 408.45 | 145.00 | 75.00 | 37.33 |
| 11 | 290.00 | 33.13 | 75.00 | 39.96 |
| 12 | 290.00 | 256.87 | 75.00 | 42.76 |
| 13 | 290.00 | 145.00 | 42.10 | 45.41 |
| 14 | 290.00 | 145.00 | 107.90 | 51.87 |
| 15 | 290.00 | 145.00 | 75.00 | 49.72 |
| 16 | 290.00 | 145.00 | 75.00 | 45.41 |
| 17 | 290.00 | 145.00 | 75.00 | 49.72 |
| 18 | 290.00 | 145.00 | 75.00 | 48.28 |
| 19 | 290.00 | 145.00 | 75.00 | 49.00 |
| 20 | 290.00 | 145.00 | 75.00 | 48.58 |

Table 5
ANOVA for response surface quadratic model

| Source | Sum of squares | df | Mean square | *F*-value | *p*-value | |
|---|---|---|---|---|---|---|
| Model | 571.57 | 9 | 63.51 | 21.79 | <0.0001 | Significant |
| *A* | 111.93 | 1 | 111.93 | 38.40 | 0.0001 | |
| *B* | 14.47 | 1 | 14.47 | 4.97 | 0.05 | |
| *C* | 86.06 | 1 | 86.06 | 29.53 | 0.0003 | |
| *AB* | 5.36 | 1 | 5.36 | 1.84 | 0.2048 | |
| *AC* | 43.38 | 1 | 43.38 | 14.89 | 0.0032 | |
| *BC* | 3.21 | 1 | 3.21 | 1.10 | 0.3184 | |
| $A^2$ | 148.18 | 1 | 148.18 | 50.84 | <0.0001 | |
| $B^2$ | 94.91 | 1 | 94.91 | 32.56 | 0.0002 | |
| $C^2$ | 0.0053 | 1 | 0.0053 | 0.0018 | 0.9669 | |
| Residual | 29.14 | 10 | 2.91 | | | |
| Lack of fit | 16.33 | 5 | 3.27 | 1.27 | 0.3984 | Not significant |
| Pure error | 12.82 | 5 | 2.56 | | | |
| Total | 600.71 | 19 | | | | |
| Std. dev. | 1.71 | | $R^2$ | 0.9515 | | |
| Mean | 43.54 | | Adj. $R^2$ | 0.9078 | | |
| C.V. % | 3.92 | | Pred. $R^2$ | 0.7447 | | |
| PRESS | 153.37 | | Adeq. precision | 13.626 | | |

[36]. Moreover, Amal found that the formed flocs were more small when using iron oxide in the presence of humic and fulvic acids [37]. And it indicated that the floc structure significantly affects the organic matter removal in coagulation process [38]. Guan's research showed that NOM with adjacent carboxylic and adjacent phenol groups are more easily removed from the liquid state with the flocs precipitation [39]. It also demonstrated in Table 5 that there has been no significant interaction between initial COD and initial SS concentration ($P_{AB}$ = 0.2048), and between initial SS concentration and coagulant dosage ($P_{BC}$ = 0.3184).

For a model to be reliable, the response should be predicted with a reasonable accuracy by the model when compared with the experimental data. Fig. 1 compares experimental COD removal efficiency with the predicted values obtained from the model. The figure indicated good agreements between the experimental and predicted values of COD removal efficiency. (The observed points on both of these plots reveal that the actual values are distributed relatively near to the straight line.)

### 3.3. 3D surface and counter plotting for evaluation of variables

For a better explanation of the independent variables and their interactive effects on COD removal, 3D surface plots and its corresponding contour plots are drawn as a function of two factors at a time, holding all other factors at fixed levels (normally at the zero level), represented in Figs. 2–4.

It is clear from the Fig. 2 that at a fixed coagulant dosage (75 mg/L), the COD removal increased steadily at first and then decreased with increasing the COD and SS concentration. In other words, the residual COD concentration will be higher for higher initial COD and SS concentrations. This could be ascribed to the accompanying increase in COD aggregation and depletion of accessible hydrolysis products of the coagulant.

It was shown in Figs. 3 and 4 that the COD removal efficiency steadily increased with increasing coagulant dosage, and there is no tendency to reach maximum removal efficiency in this experiment with the applied range of coagulant dosage. Then additional single-factor experiments were carried out with coagulant dosage at 25, 50, 75, 100, 125, and 150 mg/L, all the experiment conditions keep consistent with the center of CCD experiments. The result showed that the COD removal efficiency increased steadily at first and then stabilized at a maximum value (55%) after 100 mg/L coagulant dosages, despite the coagulant doses kept increasing. Amuda’s research groups observed similar phenomenon in COD removal by coagulation–flocculation process for the treatment of beverage industrial wastewater and abattoir wastewater. The results showed that the COD removal efficiency increased rapidly at first and up to 73% with 300 mg/L dose of ferric chloride in beverage industrial wastewater treatment. Addition of the coagulant above 300 mg/L caused the removal efficiency to appear constant [40]; and the COD removal efficiency reached a maximum of 65, 63, and 65% using alum, ferric chloride, and ferric sulfate,

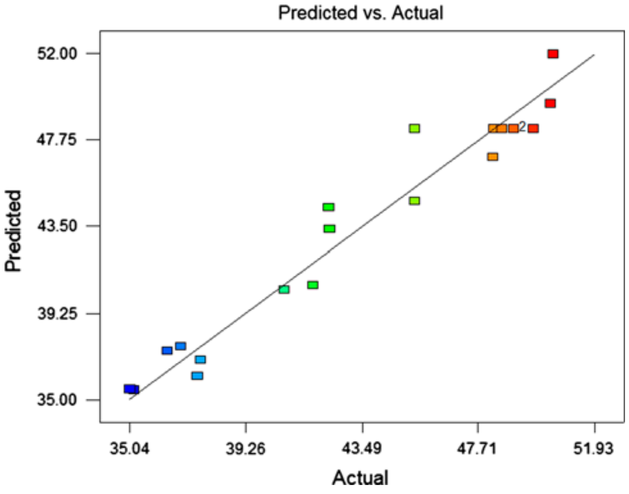

Fig. 1. Predicted vs. actual data for COD removal efficiency.

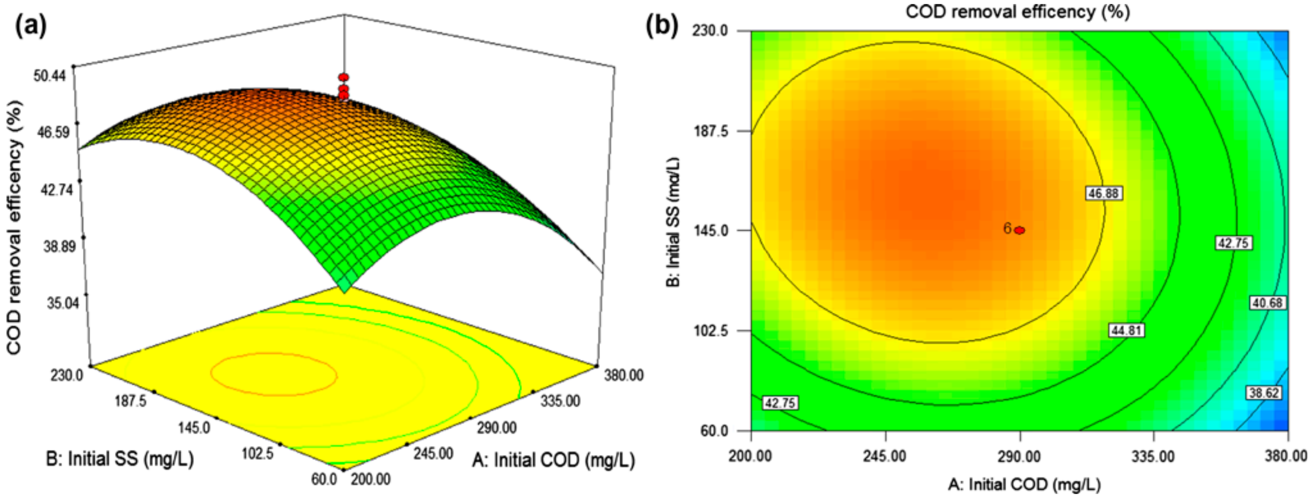

Fig. 2. The effect of initial COD and SS concentration for COD removal: (a) 3D surface graph (b) Contour plots.

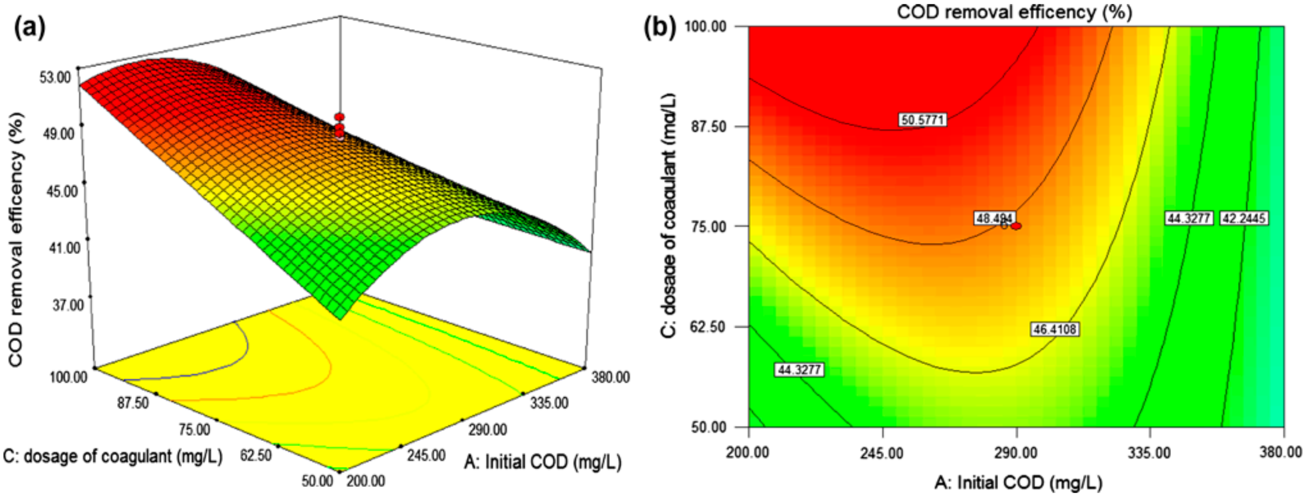

Fig. 3. The effect of initial COD concentration and coagulant dosage for COD removal: (a) 3D surface graph (b) Contour plots.

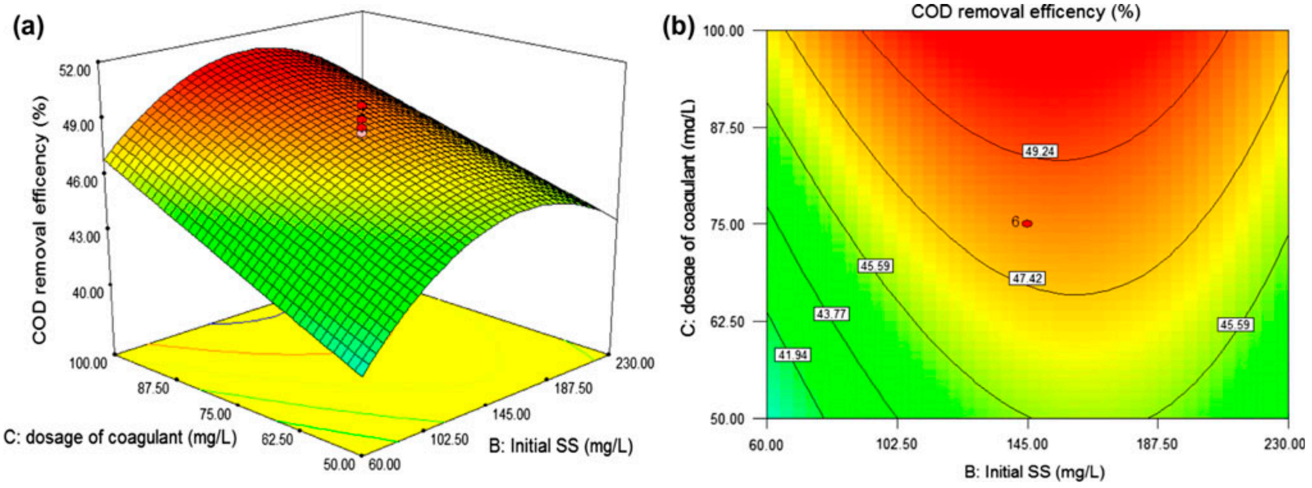

Fig. 4. The effect of initial SS concentration and coagulant dosage for COD removal: (a) 3D surface graph (b) Contour plots.

respectively, at doses of the coagulants (750 mg/L) in abattoir wastewater treatment [41].

### 3.4. Confirmation experiments

In order to confirm the validity of the statistical model, additional confirmation experiments were conducted. The means of measured and calculated efficiency were 49.39 and 51.18 with standard deviation of 4.07 and 4.56, respectively. The difference between measured and calculated removal efficiency was evaluated by PASW statistics 17.0. The *T*-test result showed that there is no obvious difference between measured and calculated removal efficiency (Sig. = 0.248). It is confirmed that the quadratic model can be used to optimize the coagulant dosage or COD removal efficiency in the treatment of CSO wastewater by coagulation–flocculation process.

## 4. Conclusions

In this study, coagulation–flocculation process was employed to remove organic matters and SSs in CSO wastewater treatment with PAFCS as coagulant. Statistical optimization method (CCD method coupled with RSM) was successfully employed to obtain a quadratic model while the interactions between variables were demonstrated. The mathematic model quantitatively exhibited the COD removal influenced by initial COD, SS concentration, and coagulant dosage. The ANOVA results of the model showed that experimental data could fit the equation with an $R^2$ of 95.15%. There is a significant interaction between initial COD concentration and coagulant dosage for COD removal efficiency. The experimental data and model predictions agreed well. It was found that the COD removal efficiency increased steadily at first and then stabilized at a maximum value (55%) after 100 mg/L coagulant dosages, despite the coagulant doses kept increasing. Finally, the mathematic model was demonstrated to be an appropriate approach to optimize the coagulant dosage or COD removal efficiency in the CSO wastewater treatment by coagulation–flocculation process.

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