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Efficiency of $FeSO_4.7H_2O$ as a Coagulant on Chromium Hexavalent Removal Using Coagulation-Flocculation Process: Optimization Using Response Surface Methodology

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ABSTRACT

Response Surface Methodology-Central Composite Design (RSM-CCD) is widely employed in the process of optimizing the applications of coagulants for wastewater treatment. The experiment aims to evaluate the effect of the $FeSO_4.7H_2O$ concentration and settling time on removing the chromium hexavalent (Cr (VI)) from wastewater by coagulation-flocculation using RSM-CCD. This experiment was carried out based on the results of the model and optimization using the RSM-CCD to obtain the removal efficiency of Cr (VI) as well as determine the influence of the concentration of $FeSO_4.7H_2O$ (X1) and settling time (X2). The RSM-CCD experimental design uses a two-factor and two-level design with 13 experimental runs and 5 center points. Inter-variable regression coefficients are also produced with the elimination of Cr (VI). The ANOVA test results showed a fairly significant figure for the assembled model. The model validation results show that the proposed model is compatible with the experimental results. RSM optimization shows optimum conditions based on experimental $FeSO_4.7H_2O$ coagulant concentration variables and coagulation time at 150 mg/L coagulant concentration and 60 minutes of time, and the prediction results based on RSM-CCD optimization using Design Expert show the most optimum condition at 165,562 mg/L coagulant concentrations and 60,527 minutes of time.

Keywords: coagulation-flocculation, $FeSO_4.7H_2O$, Response Surface Methodology, central composite design.

1. INTRODUCTION

The supply of clean water is one of the needs for various activities carried out by humans. Water sources provide essential nutrients for aquatic life, but human and industrial activities destroy most of the water supply for broad human needs [1]. One type of pollutant caused by human and industrial activities is the presence of heavy metal pollution, such as chromium, in waters [2]. Chromium exists in two forms in nature: trivalent chromium (Cr(III)) in chromium oxides and hydroxides, and hexavalent chromium (Cr(VI)) in chroma salts [3–6]. Chromium exposure can have a number of detrimental impacts on the human body, including digestive, respiratory, reproductive, and immune system issues [5,7]. Hexavalent chromium is employed in a variety of sectors, including leather tanning, electroplating, and metallurgy [2,8]. The coagulation-flocculation method is one of the procedures used to eliminate or reduce the concentration of hexavalent chromium as a contaminant [9–11]. This approach is said to be an efficient way to remove particles or pollutants from water [12]. Coagulants formed from inorganic chemicals as well as synthetic organic chemicals, generally referred to as natural coagulants, can be used to achieve an effective coagulation process [13–15]. The chemical composition of the coagulant used is crucial in influencing the efficacy of the coagulation process because it can boost or decrease the treatment's effectiveness

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[16,17]. Coagulant dosage, pH, stirring speed, temperature, and settling time are all parameters that influence coagulant performance and efficiency [18,19]. Because the majority of the suspended solids in sewage have very small particle sizes and a negative charge, these particles must collect and form bigger flocs during the sedimentation process [15,20]. Coagulants can be used to stop the particle destabilization process [21]. The coagulation-flocculation process involves four particle destabilizing processes: electrostatic double-layer pressure, adsorption and neutralizing charge, adsorption and inter-molecular bridges, and adsorption and inter-molecular bridges, Immunization of sediment [15,22]. The coagulation-flocculation process has the benefit of being more affordable and efficient in wastewater treatment on a big scale [23]. $FeSO_4.7H_2O$ is a common coagulant used in the coagulation-flocculation process [24–26]. $FeSO_4.7H_2O$ was chosen as a coagulant because it is extremely soluble in water, acidic in nature, can reduce Cr (VI) to Cr (III), and will precipitate in the form of hydroxide at a particular pH. As a result, $FeSO_4.7H_2O$ not only act as an efficient coagulant but also as a reducing agent [24].

Typically, laboratory experiments are used to establish the optimal dose of a coagulant. However, due to limits in the working scale and fluctuations in water quality, the coagulation process is usually not ideal enough to assess this, hence modeling programs such as the Response Surface Methodology are required to optimize the process [27]. Response surface methodology (RSM) is a technique for analyzing cross-factor interactions in order to find an ideal response with the fewest number of experiments [28,29]. This method is frequently combined with factorial designs such as Box-Behnken Design (BBD) and Central Composite Design (CCD). CCD is frequently used to optimize the usage of coagulants as a wastewater treatment application [27,29–31]. Several studies regarding the optimization of RSM-CCD in the coagulation process were carried out by Usefi and Asadi-Ghalhari [32] which show that the RSM-CCD model can describe the main behavior of the turbidity removal process in stone cutting industry waste using PAC and alum coagulants. In addition, research conducted by Asadi-Ghalhari et al. [33] showed that the RSM-CCD model can be used in the process of removing turbidity in wastewater with a removal percentage of 96.6%.

Based on this context, the objective of this experiment is to evaluate the influence of coagulant concentration $FeSO_4.7H_2O$ and stirring time as independent variables on the process of removing Cr (VI) from wastewater via the coagulation-flocculation method using the RSM-Central Composite Design.

2. RESEARCH METHODS

2.1 MATERIALS

Jar Test (Cyclone), Spectrophotometer UV-Vis (*Shimadzu*), Analytic balance, pH meter (*Lutron*), Beaker Glass (*Iwaki*), $FeSO_4.7H_2O$ (*Merck, Germany*), $K_2Cr_2O_7$ (*Merck, Germany*), NaOH (*Merck, Germany*), and distilled water

2.2 COAGULANT PREPARATION

The coagulant concentration was varied to determine the effect of concentration on Cr (VI) removal in wastewater. This experiment's coagulant was made by dissolving $FeSO_4.7H_2O$ in distilled water at three distinct concentrations: 125 mg/L, 150 mg/L, and 175 mg/L.

2.3 COAGULATION-FLOCCULATION PROCESS

The coagulation-flocculation process was carried out using a jar test using various coagulant concentrations (125-175 mg/L) and flocculation time (45-75 minutes). Stirring speed was divided into two stages, quick stirring for 5 minutes (200 rpm), and slow stirring (60 rpm) and sedimentation time for 60 minutes. The pH of the solution was adjusted at pH=8 by adding NaOH. The results then analyzed using a UV-Vis Spectrophotometer (*Shimadzu*) with standard method procedures (SNI 6989.71:2009)

Based on the test results using a UV-Vis Spectrophotometer, the efficiency of Cr (VI) removal can be calculated using equation (1):

$$\%\,\text{Efficiency} = \frac{\text{(Co-Ct)}}{\text{Co}} \times 100\% \qquad (1)$$

with: Co = Initial concentration of Cr (VI) (mg/L), And Ct = concentration of Cr (VI) at a certain time (mg/L).

2.4 DATA ANALYSIS

This experiment was carried out based on the model results and optimization using RSM-Central Composite Design (CCD) with Design Expert 13.0.5.0 software to obtain the percentage of Cr (VI) removal efficiency, determine the effect of $FeSO_4.7H_2O$ concentration (X1) as a coagulant, and determine the effect of time (X2). The RSM-CCD experimental design used a two-factor and two-level design with 13 experimental runs and five center points. Regression coefficients between variables were also generated on the elimination of Cr (VI) ($Y_{Cr(VI)}$). The RSM-CCD experimental design is shown in Table 1. In RSM-CCD design, experimental data is collected by running experiments at particular points around a certain experimental condition. The data is then utilized to develop a regression model that can interpolate or extrapolate the expected response values at other times. Prediction data is obtained by utilizing the regression model to forecast the response at new points.

The percentage removal of Cr (VI) is calculated using a second-order equation, which can be seen in equation (2):

Table 1. Independent factors and levels in RSM-CCD design.

Variables	**Level**		
	-1	**0**	**1**
$FeSO_4.7H_2O$ (mg/L)	125	150	175
Time (Minutes)	45	60	75

$$Y = B_o + \sum_{i=1}^{n} B_i X_i + \sum_{ipj}^{n} B_{ij} X_i X_j + \sum_{i=1}^{n} B_{ii} X_i^2 \qquad (2)$$

The value of Y is the estimated value of the response to a decreasing value of Y (Cr (VI)) in the $FeSO_4.7H_2O$ coagulation process. The regression coefficients are Bo, Bj, Bij, and Bii; n is the number of coded variables; and Xi and Xj are the independent variables. The determination of the lowest and maximum values of the independent variables' levels tries to bring the experimental set offered by the software within the intended range. ANOVA was used to identify a significant regression model and to test for lack of fit at a substantial confidence level ($p < 0.01$). The R^2 value is used to evaluate the suitability level of each model. Response surface and contour plots are used to describe the optimization results and model fit from the optimization process using $FeSO_4.7H_2O$ as a coagulant in Cr (VI) removal.

3. RESULTS AND DISCUSSION

3.1 OPTIMIZATION USING RSM-CCD DESIGN

The RSM-CCD design was used for this investigation. The aim of this experiment is to provide advice for model selection and optimizing the coagulation-flocculation process conditions to obtain the highest efficiency level. Table 2 shows the findings of the Design Expert 13.0.5.0 program's analysis, which reveals the optimal efficiency model. This model is based on the value of R^2, which shows the ratio of the variation ratio to the overall ratio and indicates the model's applicability based on the predicted and experimental values.

This study suggests a Quadratic model, as shown in the results of Table 2. The results of data processing with the Design Expert program produce an R^2 value of 0.8523 and an Adj-R^2 value of 0.7468 at a 95% confidence level. Experimental values and predicted values are presented in Table 3. Equation (3) shows the relationship between efficiency and the variables that affect this experiment.

Table 2. Statistical models of coagulation-flocculation using $FeSO_4.7H_2O$ as coagulant.

Source	Std. Dev.	R^2	Adjusted R^2	Predicted R^2	PRESS
Linear	2.64	0.4987	0.3985	0.1412	119.61
2FI	2.78	0.5022	0.3363	0.0113	137.69
Quadratic	**1.71**	**0.8523**	**0.7468**	**0.1475**	**118.72**
Cubic	1.54	0.9153	0.7968	-2.1912	444.42

$$Y = -37.22019 + 1.29640 *X_1 + 0.978562 *X_2 - 0.000932 *X_1 *X_2 - 0.003744 *X_1^2 - 0.006808 *X_2^2 \quad (3)$$

with X_1: $FeSO_4.7H_2O$ Concentration and, X_2: Stirring Time

The data in Table 3 show that the values obtained from the experiments and those predicted have a good correlation with no significant differences. Based on these results, the effectiveness of the FeSO4.7H2O coagulant for reducing Cr (VI) in wastewater reached 99.945%, with a predicted efficiency of 98.81%, when the coagulant concentration was 150 mg/L and the stirring time was 60 minutes. At 60 minutes, the lowest coagulant content was 114.645 mg/L, with a projected efficiency value of 89.98%.

Table 4 shows the results of the ANOVA based on the selected model. An ANOVA is a data analysis method used to determine the suitability of the model and response variable. The F-Model is 8.08 with a very low probability value of 0.0080, indicating that this model is statistically significant. A p value greater than 0.1000 indicates that the model is not statistically significant [34]. Furthermore, the Adeq precision (AP) value can be utilized as an indicator that the model can be implemented based on the signal-to-noise ratio measurement. For the model to be accepted, the AP value must be greater than 4 [35]. The AP value obtained in this experiment was 7.575, indicating an appropriate signal for this model to explore the design space. Coefficient Variation (CV) is another metric that may be used to determine whether or not a model is significant by comparing the estimated standard error to the average value of the experimental model. The CV value should not be greater than 10% in order for the model to be considered significant [36]. The fewer values of CV value, correlates with the measurement precision of the analysis technique. Based on the data obtained in this experiment, the CV value obtained was 1.78%, so the model compiled was significant.

According to the ANOVA results in Table 4, the concentration of the coagulant $FeSO_4.7H_2O$ (X1) produces a p-value of 0.0019, whereas the parameter of stirring time (X2) provides a p-value of 0.6062. When the resulting p-value for a parameter is 0.05, it is deemed to be important in the process. A p-value smaller than 0.05 indicates the test results are statistically significant [37,38]. Based on the results of this experiment, it can be concluded that the parameters that affect the decrease in Cr (VI) in the coagulation-flocculation process are the concentration of the coagulant $FeSO_4.7H_2O$ (X1), with a p-value smaller than 0.05, while the time (X2) produces a p-value greater than 0.05, which shows that the influence of the time is not particularly significant. The magnitude of the replication error and model error can also be estimated using the lack of fit test value. This value divides the total number of errors or residuals into two parts: pure errors based on measurement replicates and model performance [35].

Table 3. Experiment and predicted of FeSO4.7H_2O efficiency as a coagulant for Cr (VI) removal.

Run	Design variable		Real variable		Response	
	X1	X2	X1	X2	Experiment	Prediction
1	-1	+1	125	75	92.510	92.68
2	+1	+1	175	75	97.765	97.84
3	0	0	150	60	99.914	98.81
4	0	0	150	60	97.751	98.81
5	0	-1.41421	150	38.7868	92.876	95.28
6	+1.41421	0	185.355	60	97.274	98.27
7	-1	-1	125	45	93.250	91.33
8	0	+1.41421	150	81.2132	96.761	96.20
9	+1	-1	175	45	99.903	97.88
10	0	0	150	60	99.945	98.81
11	0	0	150	60	98.765	98.81
12	0	0	150	60	97.653	98.81
13	-1.41421	0	114.645	60	89.129	89.98

Table 4. ANOVA results for Cr (VI) removal using coagulation-flocculation method.

Source	Sum of Squares	df	Mean Square	F-value	p-value	
Model	118.69	5	23.74	8.08	0.0080	significant
A-FeSO4	68.60	1	68.60	23.34	0.0019	
B-Waktu	0.8556	1	0.8556	0.2911	0.6062	
AB	0.4886	1	0.4886	0.1663	0.6956	
A^2	38.10	1	38.10	12.96	0.0087	
B^2	16.32	1	16.32	5.55	0.0506	
Residual	20.57	7	2.94			
Lack of Fit	15.60	3	5.20	4.19	0.1001	not significant
Pure Error	4.97	4	1.24			
Cor Total	118.69	5	23.74	8.08	0.0080	significant

The lack of fit statistical test is the average squared ratio of lack of fit with pure error. This test can be used to determine whether there is a significant error or whether it falls within the desired level of significance. The insignificant lack of fit value indicates that the compiled model is good enough because the compiled model must be fit. Based on the data acquired in the ANOVA test, a F-value 4.19 and p-value 0.1001 was produced, which indicated that the lack of fit was not significant relative to pure errors [36]. So, it can be concluded that this model can be used to describe response data for Cr (VI) removal efficiency, and the model used is suitable for predicting the conditions of the coagulation-flocculation process that result in optimum Cr (VI) removal efficiency. Figure 1(a) shows the relationship between the experimental results and the Cr (VI) removal efficiency using the $FeSO_4.7H_2O$ coagulant in the coagulation-flocculation method, demonstrating how accurate the model obtained is by comparing the experimental data to the predicted results of Cr (VI) removal efficiency. According to the data, few sites overlap directly with the diagonal line, while others seem to be close to it. Because the findings collected indicate little difference, it may be inferred that the experimental value and the predicted value have a good fit. The link between the residual probability normal graph and the percent drop in Cr (VI) is depicted in Figure 1(b). Normal probability explains that the points that intersect do not require a change in response and have no problems with normality, or it can simply be interpreted as the difference between the experimental value and the predicted value explained in a linear plot. In addition, the residual normal plot, which shows the probability (%) and residual, can be a determinant of how well the model meets the assumptions of ANOVA and can be used to measure the standard deviation of experimental and predictive data. From Figure 1(b), we can conclude that the experimental and predictive data are very compatible.

3.2 RESPONSE SURFACE ANALYSIS

The data that has been processed with Design Expert 13.0.5.0 software is the most suitable model, namely the quadratic model. The results obtained from data processing are displayed with three-dimensional graphs and contour plots and can be seen in Figure 2. Theoretically, a higher coagulant concentration will increase the efficiency of heavy metal removal [39]. When it reaches its optimum dose, the correct coagulant dose will be efficiently set aside and result in a large reduction. Because the colloidal particles to be neutralized precipitated together with the optimum coagulant concentration and reached equilibrium at concentrations greater than the optimum dose, the resulting concentration will increase the concentration of heavy metals.

Figure 2 shows the effect of coagulant concentration and time factors on Cr (VI) removal efficiency. The red dot in Figure 2 indicates the design point, which is a circle enclosed around the factorial square. According to the graph, the highest percent removal was achieved in this experiment with a coagulant concentration of $FeSO_4.7H_2O$ of 150 mg/L and a stirring time of 60 minutes. Based on the ANOVA model prediction findings in Design Expert 13.0.5.0 software, the ideal coagulant $FeSO_4.7H_2O$ concentration and stirring duration were 165.562 mg/L and 60.527 minutes, respectively, with an efficiency value of 99.724% and a desirability of 0.980. The concentration of the coagulant is critical in achieving optimal circumstances for the coagulation process to attain equilibrium in the creation of flocs, which will eventually precipitate in the form of hydroxide. When the coagulant $FeSO_4.7H_2O$ is added to the coagulation-flocculation process for reducing Cr (VI), it automatically reduces Cr (VI) to Cr (III). Due to the development of Fe (OH) flocs in solution, excessive coagulant concentration in comparison to Cr (VI) levels will automatically disturb the reduction process to Cr (III). Furthermore, the stirring time must be optimized since when it reaches equilibrium and the stirring process is still in progress, the efficiency is reduced due to the floc particles breaking when stirring, which tends to be too long. This is consistent with Yu et al. [40] experiments, which show that extending the settling time does not improve contaminant removal or coagulant efficiency because it reduces the final floc size. Another study conducted by Fitria et al. [23] indicated that the stirring time is adequate for the coagulant to come into touch with the colloids so that the agglomeration process or creation of destabilized floc occurs totally, and when the duration of operations is extended, the floc size will get smaller. Large flocs break down into minute particles that are difficult to settle, thereby decreasing the effectiveness of flocculation coagulation in separating solids. This difficulty is caused because if the flocculation time is high, the grinding speed is greater than the floc formation speed.

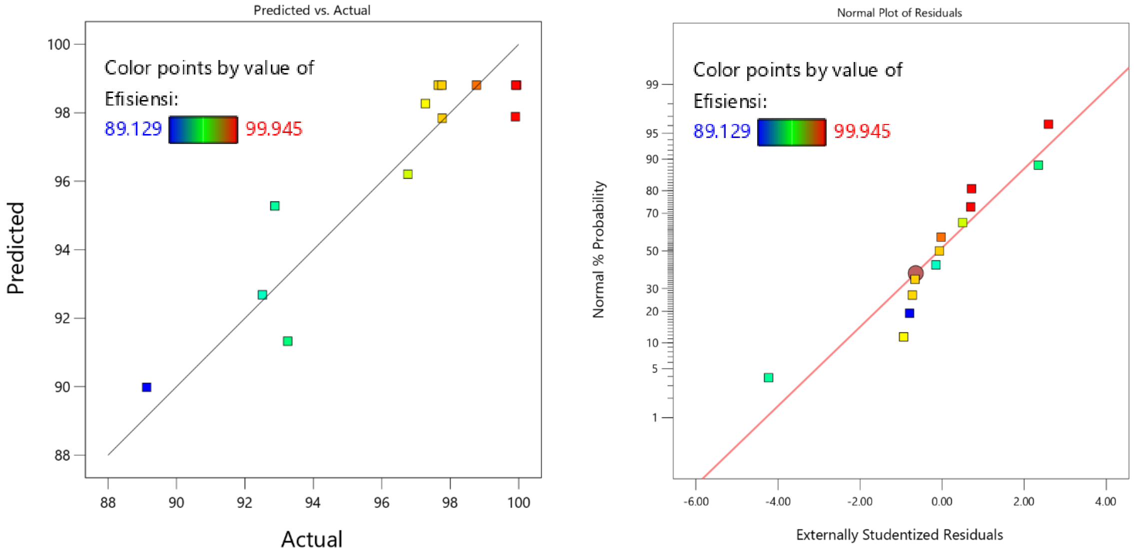

Figure 1. (a) Relationship between predicted and actual value of Cr (VI) removal efficiency using $FeSO_4.7H_2O$ as coagulant, and (b) Relationship between normal plots of residual probabilities for efficiency of Cr (VI) removal.

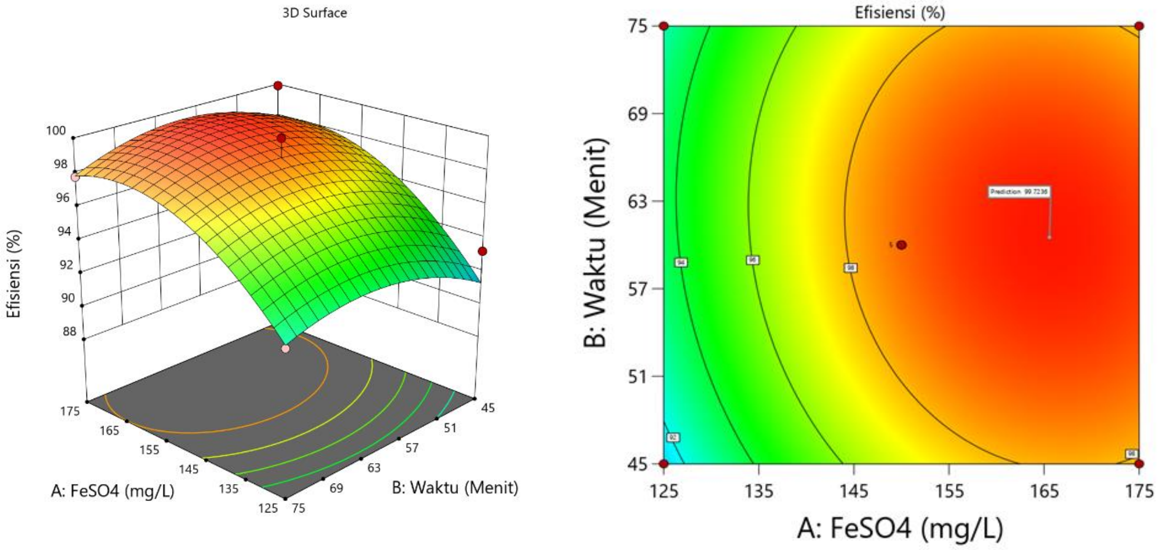

Figure 2. Effect of $FeSO_4.7H_2O$ concentration and settling time to removal efficiency of Cr (VI) (a) 3D plot, (b) contour plot.

4. CONCLUSION

The coagulation-flocculation procedure, which uses the coagulant $FeSO_4.7H_2O$, is an efficient way to remove Cr (VI) from wastewater. The process is described using a second-order equation with two influential parameters, including the concentration of coagulant $FeSO_4.7H_2O$ and stirring time, applying Optimization of Response Surface Methodology using Central Composite Design. Based on the model, the ANOVA test results show a significant number, which is rather high. The model validation findings reveal that the suggested model accords with the experimental results with a relatively high R^2 value. Optimization of RSM showed optimal conditions based on experiments with the variable concentration of coagulant $FeSO_4.7H_2O$ and stirring time, at a coagulant concentration of 150 mg/L and a stirring time of 60 minutes, and the prediction showed that the most optimal conditions were at a coagulant concentration of 165.562 mg/L and a stirring time of 60.527 minutes.

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