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Optimization of coagulation–flocculation process for turbidity removal using response surface methodology: a study in Ilam water treatment plant, Iran

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ABSTRACT

Coagulation–flocculation is an important process in removing suspended and colloidal matter from water in treatment plants. In this batch study, response surface methodology (RSM) was used to investigate the interactive influences between selected operating parameters of coagulant and coagulant aid dosage as well as pH for an optimized condition of turbidity removal from Ilam wastewater treatment plant based on data from jar tests. In this study, two common coagulants including aluminum sulfate and ferric chloride were used. LT25 and LT27 were applied as coagulant aids. Aluminum sulfate yielded highest turbidity removal at a dosage of 5 mg L^{-1} at pH 7. For ferric chloride, the maximum efficiency was observed at dosage 5 mg L^{-1} at pH 9. Application of aluminum sulfate in the presence of LT27 showed the highest turbidity removal among the studied reagents, and the efficiency increased with the increase in dosage. Based on the results of the present study, aluminum sulfate generally yielded a higher turbidity removal. But, ferric chloride formed larger flocs. Moreover, LT27 had a higher efficiency compared with LT25. Finally, the RSM was indicated as an appropriate tool for the optimization of the coagulation–flocculation process by confirmation experiments.

Keywords: Water treatment; Turbidity; Coagulation; Aluminum sulfate; Ferric chloride

1. Introduction

In recent decades, water pollution has been remarkably increased due to human activities and also natural processes [1–3]. Nowadays, due to rapid population growth, urbanization, and industrial development, the existing demand for clean water continues to increase [4–8]. In order to reduce the final water costs, it is necessary to optimize the main operating factors including electricity, chemicals, etc., in water treatment plants [9]. Based on raw water characteristics, many techniques have been employed to make water safe and healthy to the consumer [10–13].

One of the major obstacles with purification of surface water is the large periodical changes in turbidity [14]. Many studies have utilized the coagulation process to treat highly

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turbid waters [15–17]. Coagulation is one of the key processes for effective removal of impurities including colloids, microbes, natural organic matter, and so on in surface water in water treatment plants [18,19]. Most of these small particles have negative charges in their structures and cause the particles to repel each other and keep them in suspension for a long time. The coagulation process reduces this charge and increases the van der Waals's force of attraction among the particles [20]. By far, the most widely used coagulants applied in water treatment plants of Iran as well as many countries are aluminum sulfate, ferric sulfate, ferric chloride, and polyaluminum chloride [21]. These agents have been applied with aluminum sulfate, bentonite, sodium silicate, and polyelectrolytes as coagulant aids which promote the removal of colloidal matters from water [22]. Polymers have been widely utilized in many treatment processes. Polymeric agents are commercially available as anionic, cationic, and nonionic types in different charge forms [23]. Polymeric anionic coagulant aids such as LT25 and LT27 (copolymer of sodium acrylate and acrylamide) are sometimes used for improving the turbidity removal of waters [24].

Many parameters such as raw water quality, coagulant type, solution pH, and coagulant dosage have been identified to affect the coagulation efficiency [25]. The optimization of these parameters may considerably enhance the process efficiency [26–29]. The major limitation is to determine the optimal coagulant dosage by considering raw water characteristic variations. Use of excessive amounts of a coagulant increases the purification costs and consequently public health issues, while lower amounts fail to meet the water quality standards and less effectiveness of the water treatment plant [30]. Therefore, it is necessary to utilize the optimum conditions when performing coagulation–flocculation so that wastage of the associated chemicals may be reduced [31,32].

In many conventional multi-variable tests for the coagulation–flocculation process, optimization is generally performed by changing single variable while keeping all other variables constant at a specific set of conditions [33,34]. This method wastes time, energy, and chemicals, and it is also unable to predict the real optimum conditions because of neglecting the relationships among factors [35]. These problems of the traditional method can be prevented by using the response surface methodology (RSM) that comprises statistical design of experiments in which all variables are considered together by generating a set of runs [36]. In literature, RSM has been used in various types of optimization processes in the field of water treatment [37–40].

Diagnostic experiments using analysis of variance (ANOVA) then evaluate the validity and fitness of the proposed model.

The main aims of the present study were to determine the optimum coagulation–flocculation conditions for two common coagulants including aluminum sulfate and ferric chloride by using jar testing and response surface methodology for influent water to Ilam's water treatment plant. The experiments were conducted by jar test which is commonly employed to investigate the treatment process efficiency. By doing jar tests, applied coagulant, coagulant dosage, and pH are determined. The Box–Behnken design was applied to find the effect of the main operating variables including coagulant dosage, pH, and coagulant aid dosage on the removal of turbidity by using coagulants such as aluminum sulfate and ferric chloride and LT25 and LT27 as two anionic polymers.

2. Materials and methods

This batch scale study was conducted in order to determine the optimum conditions of two common coagulants, namely, aluminum sulfate (alum) and ferric chloride, using response surface methodology. Two polymeric anionic coagulant aids such as LT25 and LT27 also were used to improve the coagulation–flocculation process. All these chemicals were purchased from Sigma Aldrich Company (Germany).

In this study, jar experiments were conducted on water samples collected from the influent of Ilam's water treatment plant. The treatment plant is supplying drinking water for a population of 213,579 individuals of Ilam city located at 33°38′15″N 46°25′22″E in the west of Iran. The Ilam dam supplies the water of Ilam's water treatment plant. The turbidity and pH of the obtained samples were in the range of 1.5–8.04 NTU and 8–8.3, respectively. The quality of surface water in Ilam dam is changed during rainy period, which necessitates the use of exact amounts of coagulants.

Jar tests were performed using a variable speed Jar tester (HACH, USA) equipped with six stainless steel flat paddle mixers. The experiments were conducted at room temperature (20°C). After the addition of appropriate dosage of each coagulant, the solution pH was adjusted according to experimental range suggested in Table 1 using either 0.1 M H_2SO_4 or 0.1 M NaOH. Thereafter, the jars followed 1 min of rapid mixing at 120 rpm and 30 min of slow mixing at 20 rpm. After settling for 20 min, about 15 mL of the liquid was withdrawn using a pipette from 3 cm height below the water surface level in each jar for measurement of final turbidity [41]. Turbidity measurement was analyzed by turbidity meter (model HACH-P2100). The performance of these experiments was examined by measuring turbidity removal percentages.

2.1. Preparation of coagulants and coagulant aids

Initially, a 1% stock solution of coagulants was used for the preparation of desired concentration of each coagulant. Accordingly, 1 g of aluminum sulfate powder was dissolved into 100 mL of distilled water. For preparation of stock of ferric chloride, 2.5 mL of 40% ferric chloride solution was diluted to 100 by distilled water. For coagulant aids, 0.1 g

Table 1
Actual and coded values of input variables of the Box–Behnken design

Coagulant	Factor	Code	Variable level		
			–1	0	+1
Aluminum sulfate	Coagulant dosage (mg L^{-1})	X_1	3	6.5	10
	pH	X_2	6	7	8
	Coagulant aid (mg L^{-1})	X_3	0.1	0.2	0.3
Ferric chloride	Coagulant dosage (mg L^{-1})	X_1	0.3	0.5	0.7
	pH	X_2	5	7	9
	Coagulant aid (mg L^{-1})	X_3	0.1	0.2	0.3

of coagulant aids were diluted to 100 mL and then stirred at 500 rpm.

2.2. Experimental design and data analysis

The Design-Expert software version 10 was applied to design the experiments within the ranges of parameters to decline the number of required experiments. The interactive influences of the independent parameters on the response ones were illustrated by two and three dimensional contour plots. A Box–Behnken design (BBD) in RSM was utilized to study the influences of the main process variables on the coagulation–flocculation efficiency. The effects of factors on the response were found by employing the quadratic BBD with four factors, three levels, and five replications at the design center. In the current work, coagulant dosage (X_1), pH (X_2), and coagulant aid (X_3) were chosen as three independent variables in the coagulation–flocculation process. Three levels of low (–1), high (+1), and medium (0) were selected for the purpose of this study.

In order to develop the regression equation, the interaction between the coded and actual amounts is described based on Eq. (1) [42–44].

$$Z_i = \frac{X_i - X_0}{\Delta X} \tag{1}$$

where Z_i is the coded amount of ith variable, X_i is the original amount of the ith variable, X_0 stands for the un-coded amount of the ith input variable at the center point, and ΔX is the amount of step change.

Range and coded levels of the selected input variables for model study are summarized in Table 1. The three factor designed experiments were carried out with five replications at the design center to evaluate the pure error. The output response is the coagulation–flocculation efficiency and totally 17 experiments were conducted for each coagulant. All chemicals used for the analytical determinations were of analytical grade. Normally, the relationship between the responses (% turbidity removal or Y) and the independent variables (coagulant dosage, pH, and coagulant aid dosage) in coagulation–flocculation process cannot be appropriately described by a first order model. Therefore, a general form of second-order polynomial quadratic model was applied to study the influences of different selected variables on the responses according to Eq. (2) [36]:

$$Y = \beta_0 + \beta_1 x_1 + \beta_2 x_2 + \beta_3 x_3 + \beta_{12} x_1 x_2 + \beta_{13} x_1 x_3 + \beta_{23} x_2 x_3 + \beta_{11} x_1^2 + 1 + \beta_{22} x^2 + \beta_{33} x^3 \tag{2}$$

where Y is the predicted response, β is the regression coefficient, β_0 is the intercept, β_1, β_2, β_3 are the linear coefficients, β_{12}, β_{13}, β_{23} are interaction coefficients, and β_{11}, β_{22}, β_{33} are quadratic coefficients [45–47].

3. Results and discussion

The objective of our research is to determine the optimal conditions, namely, optimal dosages of coagulants and coagulant aids as well as pH, leading to the highest removal of turbidity. Three-factor and three-level Box–Behnken design and experimental results are given in Table 2. Optimal dosages are considered as the amount above where there is no enhancement of removal percentage even if we add more coagulant [46]. Accordingly, the effect of key parameters in coagulation process such as coagulant and coagulant aid dosages and pH was investigated.

The effect of pH and coagulant dosage on turbidity removal by aluminum sulfate is studied in the range of 6–8 and 3–10 mg L^{-1}, respectively (Table 1). The dosage range of coagulants and coagulants aids in this study was selected based on the studies in literature [48–50]. The effect of the applied aluminum sulfate dosage and pH on turbidity removal is illustrated in Fig. 1. As shown in the figure, the highest removal was observed at dosage of 5 mg L^{-1} and pH 7.

Fig. 2 represents the effect of the applied ferric chloride dosage and pH on turbidity removal. Ferric chloride dosages of 0.5, 0.7, and 1 mg L^{-1} and pH of 5, 7, and 9 were studied and the highest removal was obtained at dosage of 1 mg L^{-1} and pH 9.

Figs. 3(a) and (b) depict two and three dimensional contour plots showing the combined effects of aluminum sulfate and pH, and aluminum sulfate and LT27 dosages on turbidity removal. As can be seen in Fig. 3(a), the highest

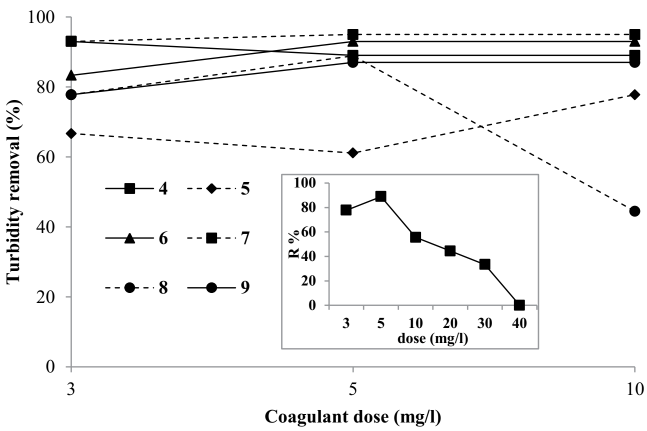

Fig. 1. Effect of aluminum sulfate dosage and pH on turbidity removal.

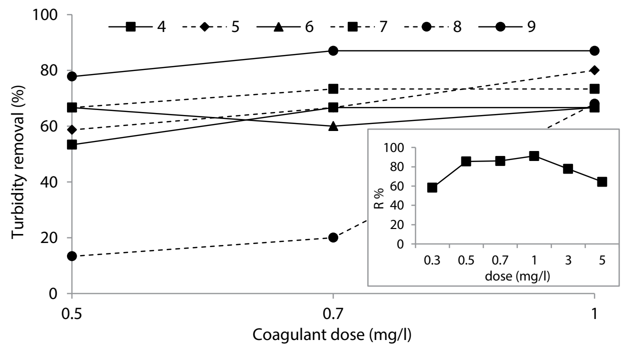

Fig. 2. Effect of the applied ferric chloride dosage and pH on turbidity removal.

Table 2
Three-factor and three-level Box–Behnken design and experimental results

No	AlumL+25			Removal %	Alum+ L27			Removal %	Ferric+ L25			Removal %	Ferric+ L27			Removal %
	X_1	X_2	X_3		X_1	X_2	X_3		X_1	X_2	X_3		X_1	X_2	X_3	
1	10	7	0.1	80.64	10	7	0.1	92.12	0.5	7	0.2	78.31	0.5	7	0.2	89.49
2	6.5	6	0.3	99.81	6.5	6	0.3	92.92	0.5	9	0.1	67.91	0.5	9	0.1	93.43
3	3	7	0.3	72.1	3	7	0.3	87.45	0.7	9	0.2	81.59	0.7	9	0.2	98.23
4	6.5	7	0.2	90.65	6.5	7	0.2	90.98	0.5	7	0.2	79.51	0.5	7	0.2	90.93
5	6.5	8	0.3	77.43	6.5	8	0.3	95.34	0.7	7	0.1	82.79	0.7	7	0.1	91.73
6	10	8	0.2	92.81	10	8	0.2	97.65	0.5	9	0.3	65.43	0.5	9	0.3	90.52
7	6.5	7	0.2	84.4	6.5	7	0.2	90.43	0.5	7	0.2	78.42	0.5	7	0.2	91.58
8	10	6	0.2	98.7	10	6	0.2	93.49	0.5	7	0.2	77.91	0.5	7	0.2	89.05
9	6.5	8	0.1	93.76	6.5	8	0.1	90.65	0.3	5	0.2	68.88	0.3	5	0.2	79.86
10	3	8	0.2	88.21	3	8	0.2	86.39	0.5	5	0.3	87.31	0.5	5	0.3	80.74
11	6.5	6	0.1	95.1	6.5	6	0.1	86.56	0.3	7	0.1	55.1	0.3	7	0.1	81.65
12	6.5	7	0.2	85.29	6.5	7	0.2	90.79	0.3	9	0.2	61.82	0.3	9	0.2	89.47
13	10	7	0.3	92.87	10	7	0.3	98.23	0.7	7	0.3	88.59	0.7	7	0.3	95.92
14	3	7	0.1	86.35	3	7	0.1	82.83	0.3	7	0.3	69.4	0.3	7	0.3	77.72
15	6.5	7	0.2	94.06	6.5	7	0.2	90.58	0.5	7	0.2	77.3	0.5	7	0.2	92.11
16	6.5	7	0.2	95.25	6.5	7	0.2	90.5	0.5	5	0.1	70.11	0.5	5	0.1	82.52
17	3	6	0.2	94.32	3	6	0.2	83.91	0.7	5	0.2	97.79	0.7	5	0.2	89.43

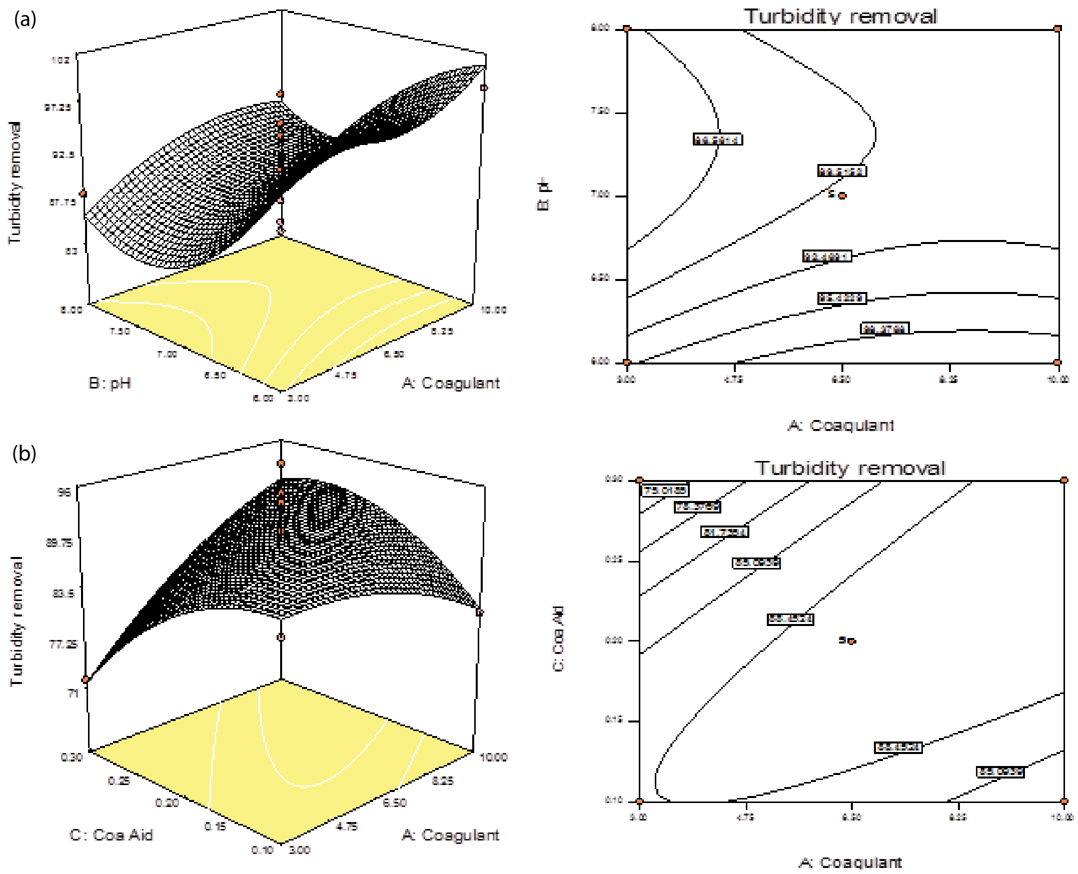

Fig. 3. Two and three dimensional contour plots showing the combined effects of (a) aluminum sulfate dosage and pH, and (b) aluminum sulfate and LT25 dosages on turbidity removal.

turbidity removal was seen at dosage range of 3–4.75 mg L^{-1} and neutral pH of 7–7.5 for aluminum sulfate. Based on Fig. 3(b), turbidity removal decreases with coagulant dosage increase. But by increasing dosage of coagulant aid LT25, the turbidity removal decreases.

Fig. 4(a) illustrates that by increasing aluminum sulfate dosages from 3 to 10 mg L^{-1}, the turbidity removal was increased. But in Fig. 4(b) a dosage range of aluminum sulfate of 8.25–10 mg L^{-1} and LT27 dosage of 0.1–0.15 mg L^{-1} has the highest turbidity removal.

Fig. 5 shows the combined effects of (a) ferric chloride dosage and pH, and (b) ferric chloride and LT25 dosages on turbidity removal. As mentioned above, for a given coagulant dosage, the acidic condition was appropriate for the removal of turbidity. Fig. 5(a) shows that the highest turbidity removal was observed at acidic pH values and coagulant dosage of 0.7 mg L^{-1} with the ferric chloride used as the coagulant. This is mainly due to the electrostatic attraction between negative charge of LT25 and positive charge of ferric chloride. Bazrafshan et al. [51] found aluminum sulfate had a higher turbidity removal at pH range 5.5–7.5 compared with ferric chloride and polyaluminum chloride.

Figs. 6(a) and (b) illustrate two- and three-dimensional contour plots showing the combined effects of ferric chloride dosage and pH, and ferric chloride and LT27 dosages on turbidity removal, respectively. As clearly shown in the figure, the turbidity removal efficiencies increase by decreasing pH as well as increasing coagulant dosages. At higher coagulant dosages, more colloids can be removed by more numbers of Fe^{3+} ions.

Based on Fig. 6(a), the maximum removal was seen for pH range of 5–6 and ferric chloride dosage of 0.6–0.7 mg L^{-1}. Fig. 6(b) illustrates that turbidity removal increased with coagulant aid LT27 dosage increase until a specified amount of dosage and beyond that it decreased. Osouleddini and Abdollahzadeh et al. [52] reported that ferric chloride decreased the solution pH but LT25, a coagulant aid, had no effect on solution pH. Therefore lower dosages of ferric chloride also reduce the amounts of coagulant aids used. LT25 enhances the size of formed flocs and thus increases the settling velocity.

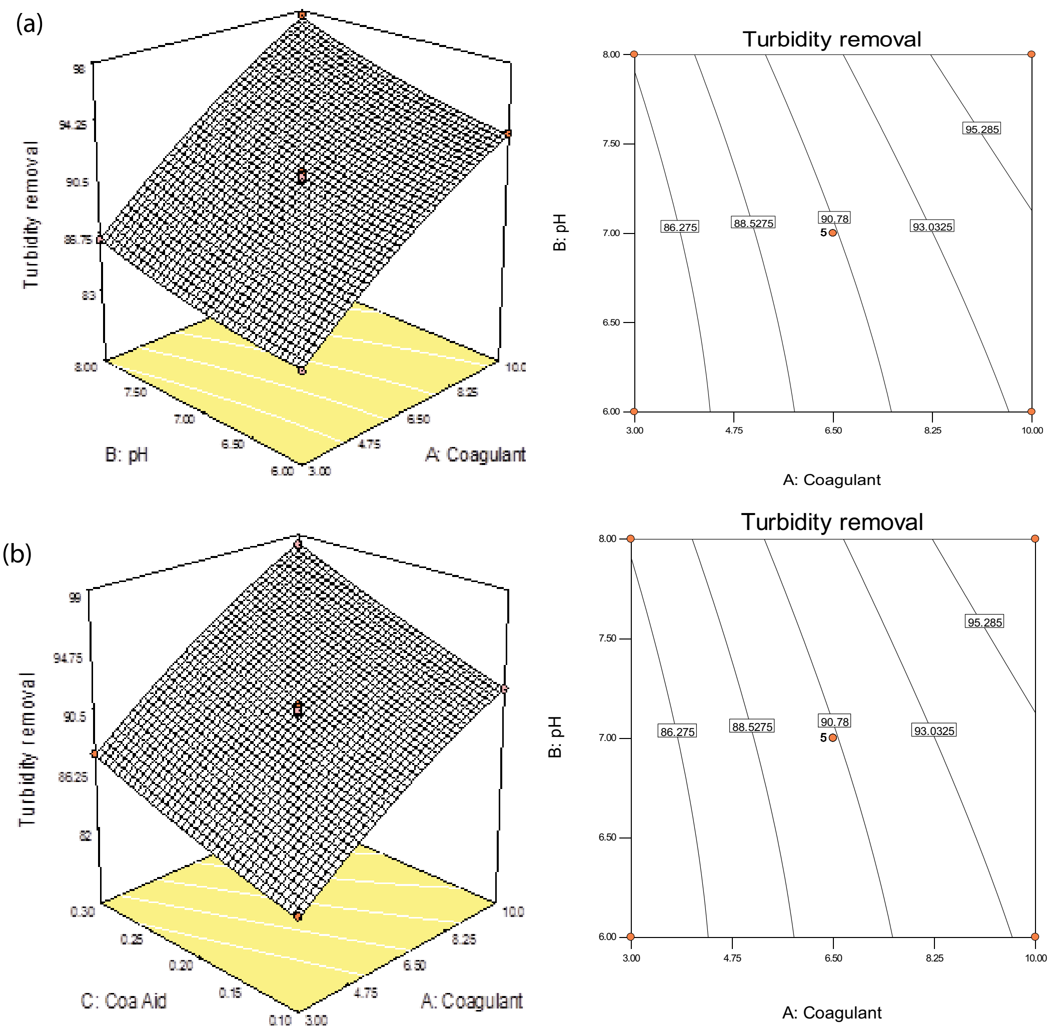

Fig. 4. Two and three dimensional contour plots showing the combined effects of (a) aluminum sulfate dosage and pH, and (b) aluminum sulfate and LT27 dosage on turbidity removal.

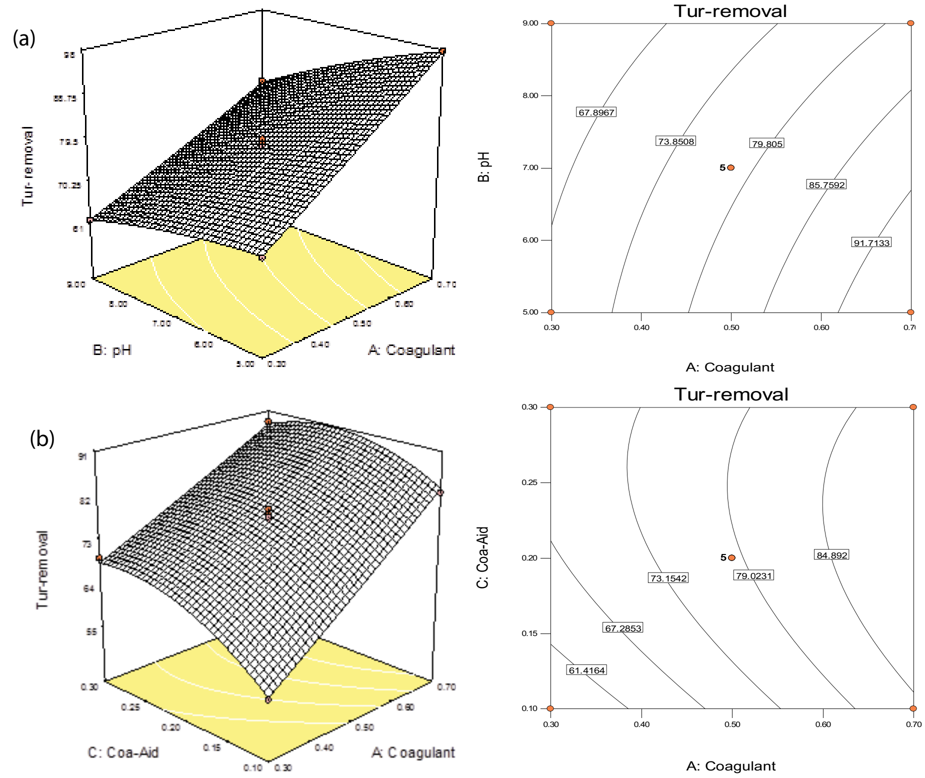

Fig. 5. Two and three dimensional contour plots showing the combined effects of (a) ferric chloride dosage and pH, and (b) ferric chloride and LT25 dosages on turbidity removal.

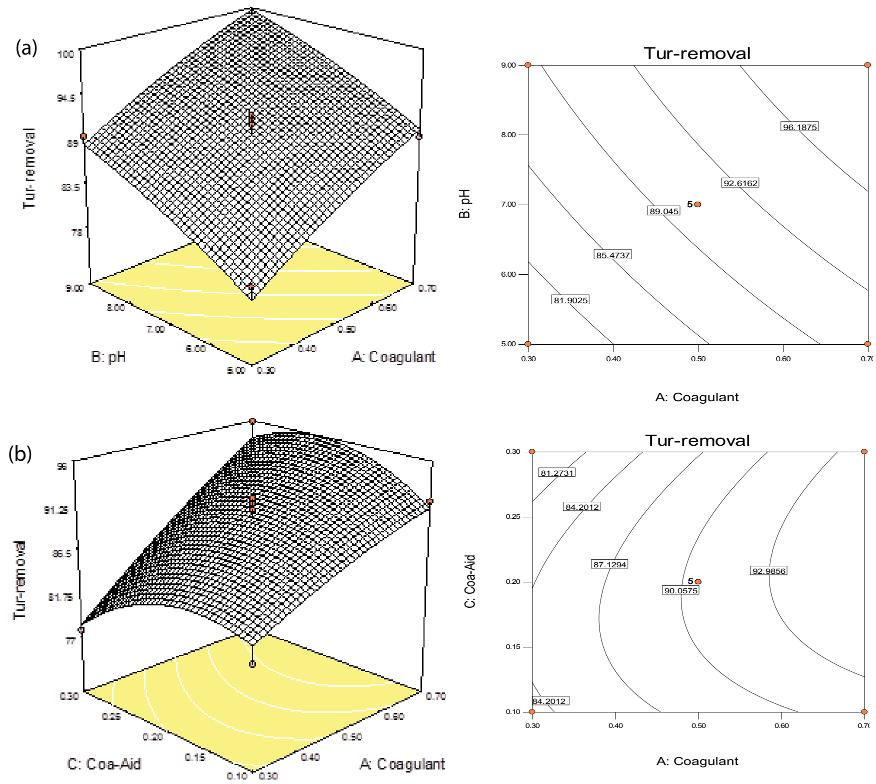

Fig. 6. Two- and three-dimensional contour plots showing the combined effects of (a) ferric chloride dosage and pH, and (b) ferric chloride and LT27 dosages on turbidity removal.

Table 3
ANOVA results for the four responses

Chemical	Final equation in terms of coded factors	p-value	R^2	Adjusted R^2	Lack of fit
Alum+LT25	$Y = +89.93 + 3.01X_1 - 4.46X_2 - 1.70X_3 + 0.055X_1X_2 + 6.62X_1X_3 - 5.26X_2X_3 - 2.48X_1^2 + 6.06X_2^2 - 4.46X_3^2$	0.0269	0.85	0.67	0.72
Alum+LT27	$Y = +90.66 + 5.11X_1 + 1.64X_2 + 2.72X_3 + 0.42X_1X_2 + 0.37X_1X_3 - 0.42X_2X_3 - 0.75X_1^2 + 0.46X_2^2 + 0.25X_3^2$	0.0001	0.99	0.99	0.65
Ferric chloride+ LT25	$Y = +78.29 + 11.95X_1 - 5.92X_2 + 4.35X_3 - 2.29X_1X_2 - 2.13X_1X_3 - 4.92X_2X_3 + 0.25X_1^2 + 0.46 - 1.03X_2^2 - 4.57X_3^2$	0.0001	0.99	0.99	0.24
Ferric chloride+LT27	$Y = +90.63 + 5.83X_1 + 4.89X_2 - 0.55X_3 - 0.20X_1X_2 + 2.03X_1X_3 - 0.72X_2X_3 - 0.72X_1^2 - 0.67X_2^2 - 3.16X_3^2$	0.0005	0.95	0.90	0.15

In this study, ANOVA was applied for graphical analyses of the data to express the interaction between the input variables and the responses. The quality and statistical significance of the fit polynomial model was explained by the p-value, coefficient of determination R^2, adjusted R^2, and lack of fit [53]. Table 3 summarizes ANOVA results for alum+LT25, alum+LT27, ferric chloride+LT25, and ferric chloride+ LT27. The results for the responses yielded by ANOVA to evaluate the goodness of fit. The p-values < 0.05 (Table 3) illustrates that the second-order polynomial model fitted the experimental findings well for all the studied reagents. The p-values for alum+LT25, alum+LT27, ferric chloride+LT25, and ferric chloride+LT27 were 0.0269, 0.0001, 0.0001, and 0.0005, respectively.

A high value of R^2 is acceptable and a reasonable agreement with adjusted R^2 is important [54]. The high values of coefficient of determination for alum+LT25 ($R^2 = 0.85$), alum+LT27 ($R^2 = 0.99$), ferric chloride+LT25 ($R^2 = 0.99$), and ferric chloride+LT27 ($R^2 = 0.95$) and the low p-values (<0.05) suggest a good agreement between the experimental data and predicted values obtained from the equations. The other evaluation of the model is the lack of fitness test, which explains the residual with the pure error obtained from the replicated design points [55]. As can be seen in Table 3, lack of fitness values for alum+LT25, alum+LT27, ferric chloride+LT25 and ferric chloride+LT27 were 0.72, 0.65, 0.24, and 0.15, respectively, which were all insignificant.

4. Conclusions

Improving the final treated water characteristics and reducing various operating issues concerning the challenging task of controlling coagulant dosing rate at many water treatment plans is of great importance. Jar test is commonly used worldwide for determination of operating condition for coagulation–flocculation process. In this study, a coagulation–flocculation process with aluminum sulfate and ferric chloride as the coagulant and LT25 and LT27 as polymers were employed to optimize jar test results obtained in order to improve the final water quality of treatment plant of Ilam city by use of BBD in RSM. Three important parameters in coagulation–flocculation process including used coagulant, coagulant aid dosage, and pH were applied for the purpose of optimization in this work.

The results of ANOVA clearly showed that aluminum sulfate in the presence of coagulant aid LT27 had the highest effect on turbidity removal.

Based on the results in the present work, response surface methodology can be successfully applied for modeling and optimizing the best operating conditions used in the practice and it is the cost-effective approach of obtaining the maximum amount of information in a short period of time and with the least number of experiments.

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