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To cite this article: Sarah Adil Chasib *et al* 2021 *IOP Conf. Ser.: Earth Environ. Sci.* **722** 012038

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Optimization of composite polymer dosing and pH in wastewater treatment plants

^a Sarah Adil Chasib, ^a Nayyef Mohsin Azeez, ^b Ammar Salman Dawood

^a University of Basrah, college of science, Ecology department

^b University of Basrah, college of engineering, civil engineering department

nayyef.azeez@uobasrah.edu.iq

Abstract

In this paper, a composite Aluminum Chloride – polyacrylamide polymer (indicate $AlCl_3$ -PAM) was prepared by physical mixing. the response surface methodology (RSM) and central composite design (CCD) was utilized for modeling and optimization of the flocculation process of the wastewater, to utilized maximum removal of the (turbidity, total suspended solid and chemical oxygen demand). moreover, the influence of composite polymer dosage and pH on the wastewater flocculation process was discussed. Square models developed for the three responses (Turbidity, TSS and COD) denoted the optimum condition of flocculation process in terms of optimal composite polymer dosage and optimal pH. The optimum composite polymer dosage was (2.5 mg/l) with an optimum pH 7. In these optimal values , the wastewater treatment achieved (84.9%, 97%, and 92.4%) removal of COD, turbidity, and TSS, respectively.

Keywords: Flocculation, wastewater treatment, RSM, composite polymer, $AlCl_3$ -PAM

1. Introduction

Different kinds of industries produce wastewater containing a variety of impurities including small and large suspended and dissolved solids, organic and inorganic particles and metals. this untreated wastewater may discharge to the aquatic environment such as rivers, lakes and streams thus poses a threat to the nature and the population living nearby these water resources. This uncontrolled discharge is widely preented in undeveloped countries. Bringing the contaminants present in the wastewater together to form heavier mass to improve settling and filtration is a challenge because of the surface charge and the very small size of particles [1-2]. Therefore, in the recent years the use coagulation-flocculation processes increased since it considered one of the effective methods in the physicochemical processes used in urban water and wastewater treatments due to its relatively simple design, easy operation, and low energy consumption among removal techniques [3].

Verity of inorganic, organic, composite and hybrid flocculation materials has recently been introduced. With research focused on the synthesis of these inorganic and organic hybrid materials the synthesis and application of inorganic-organic composites materials are now increasingly growing. Hybrid Polymers are known to be the strongest rendering product in contrast with commonly used coagulation and flocculation materials. Therefore the agglomerating strength of flocculating chemicals is improved by stepping



up the proportion of active ingredient and of positive flocculating chemicals as reagent during water and waste water flocculation process. [4-5-6-7].

In order to study the effect of variables and their response, design of experiments was presented to minimize the number of experiments and save time by applying statistical techniques to cover variety of experimental statistics in contrast to traditional multi-factor experiments which considered as time consuming and provide valid results under fixed experimental conditions making the arrival to the same results unreliable if the conditions were to be changed [8]. In addition, the interaction effect of factors to the response is studied by factorial designs [9].

The Response Surface Methodology (RSM) approach is a set of mathematical and statistical techniques used to design experiments and construct models to improve, establish and optimize processes and experimental conditions. RSM is often used to determine the relative value of different variables and to minimize the number of experiments even though complex interactions occur [10-11].

This study is aimed at preparing a composite polymer from inorganic-organic material. The composted polymer then was used for the removal of pollutants from a wastewater. RSM was used to analyze the effect of the composite polymer dose with the initial pH and enhance the flocculation process.

2. Materials and methods

Materials;

Polyacrylamide (PAM) provided by HIMEDIA laboratories Pvt. Ltd. (India). Aluminium Chloride ($AlCl_3$) by Scharlab S.L. (Spain). Hydro Chloric acid (HCl) by SDFCL (India). Sodium hydroxide (NaOH) by VWR prolabo chemicals (EC).

Preparation of composite polymer;

Inorganic material (1%) concentration prepared from dissolving 1g ($AlCl_3$) in 100ml deionized water while organic material (0.1%) concentration prepared by dissolving 0.1g PAM in 100ml deionized water. The mixing procedure was by using magnetic stirrer until complete dissolve of the substance. Then an optimum dosage of composite polymer (PAM + $AlCl_3$) was prepared by mixing 70% inorganic material with 30% organic material using magnetic stirrer for (60-90 minutes) at room temperature to form a homogeneous mixture which represent the composite polymer.

Wastewater sample;

The wastewater samples were collected from the main wastewater treatment plant in university of Basrah, Qarmat Ali campus which located at (30° 33' 21.3" N, 47° 44' 56.7" E). The transport of the sample took 15 minutes. Samples were characterized for the pH, Chemical Oxygen Demand (COD), Turbidity and total suspended solids (TSS). The sample collection and preservation were according to [12]. The measurements of the raw samples were as in (Table1).

Table 1: Measurement of the raw samples.

Source	pH	Turbidity (NTU)	COD (mg/L)	TSS (mg/L)
wastewater	7.7	85.2	173	66

Flocculation process;

A programmable jar research apparatus for the Flocculation experiments (JJ-4A, China) used at room temperature. The initial sample pH was adjusted by using 0.5N HCL and 0.5N NaOH. A 800ml samples transferred to the beakers, AlCl₃-PAM composite polymer was added to each beaker. The aqueous solution was then stirred rapidly by paddle speed of 120 rpm for 1 minute followed by slow mixing at 30 rpm for 15 minutes. Finally, the treated samples were allowed to settle for 45 minutes. The removal efficiency of the pollutants was calculated according to the formula:

$$\text{Removal Efficiency} = \frac{C_i - C_f}{C_i} * 100 \dots \dots \dots (1)$$

Where C_i and C_f the initial and the final concentration of pollutants respectively.

Design of experiment;

Surface Methodology (RSM) is a statistical approach commonly used in experimental design since it is able to optimize processes. Minitab V19 software has been used for statistical data analysis and could be used to optimize the major operating factors (AlCl₃-PAM composite polymer dose and samples pH). The Central Composite Design (CCD) and RSM were applied to determine the effective variables interacting with turbidity flocculation quality, the total suspended solids, and the chemical oxygen demand (COD) removals.

In this analysis, the typical form of CCD that is CCFD consists of 2^k factorial points (k means factors= 2), 2k axial points and 5 at the middle provides an approximation of the experimental variance in error. The composite dose (X₁) and pH (X₂) were the independent variables (factors).

There are three levels: -1 (low), 0 (central) and +1 (high). And the actual values of the coded factor levels have been obtained through preliminary studies, including these codes (Table 2).

Table 2: The three levels of the experimentals factors.

Design Variable (factors)	Symbol	Real value of coded levels		
		-1	0	+1
Composite polymer dose (mg\L)	X ₁	2	2.5	3
Sample pH	X ₂	5	7	9

The second-order empirical polynomial equation (Eq. 2) will be used for proving the relation between the factors (X₁ and X₂) and the responses tested (Y_{COD}, Y_{TSS}, and Y_{Turbidity}). Here Y is the response equation (TSS removal, turbidity removal, and COD removal); β₀ constant represents the coefficient; where β_i is coefficient of linear term; β_{ii} is coefficient of square term;

β_{ij} is coefficient of quadratic term. k is the number of variables independent of each other. X_i and X_j are independent variable coded values.

$$Y = f(x) = \beta + \sum_{i=1}^k \beta_i X_i + \sum_{i=1}^k \beta_{ii} X_i^2 + \sum_{i=1}^{k-1} \sum_{j=i+1}^k \beta_{ij} X_i X_j \dots \dots \dots (2)$$

3.Results and discussion.

Statistical analysis:

Together, there were 13 experiments to test the coefficients for independent variables in the flocculation process (composite polymer dosage and wastewater pH) as demonstrated in (Table 4). There are several models, such as linear, square and 2 way interaction, can be used to find the association of experimental data and for each reaction to achieve regression equation , the square model was depended, according to the experimental data, because the best to represent the correlation between all responses and experimental data , with the lowest F value and P value. As a result, this model was adopted. while, the linear model was not suggested in this study because it had insufficient points to estimate the coefficients of the model. The final square model for each response ($Y_{\text{turbidity}}$, Y_{TSS} , and Y_{COD}) in term of coded factors is shown in equations (Eq. 3, Eq. 4, and Eq. 5):

$$Y_{\text{Turbidity}} = - 437.8 + 98.04 X_2 + 104.7 X_1 - 5.473 X_2^2 - 15.81 X_1^2 - 2.82 X_2 * X_1 \quad (3)$$

$$Y_{\text{TSS}} = - 661 + 108.0 X_2 + 306 X_1 - 5.92 X_2^2 - 48.8 X_1^2 - 9.85 X_2 * X_1 \quad (4)$$

$$Y_{\text{COD}} = 16.9 + 5.97 X_2 + 37.38 X_1 - 0.391 X_2^2 - 7.05 X_1^2 - 0.289 X_2 * X_1 \quad (5)$$

Where ($Y_{\text{turbidity}}$, Y_{TSS} , and Y_{COD}) are the responses (dependent variables) of turbidity, COD and TSS removal efficiency, respectively. X_1 and X_2 are the coded factors (independent variables) of wastewater pH respectively and composite polymer dose.

Table 3: Experimental design and responses

Run	Experimental design		Responses (results)		
	X_1 Composite polymer dosage (mg/L)	X_2 pH	Turbidity Removal %	COD Removal %	TSS Removal %
1	2.5	7	88.0869	82.0809	81.8182
2	3.2	7	93.7207	80.3468	78.7879
3	2.5	4.1	6.1033	82.0809	33.3333
4	1.7	7	78.5798	79.7688	42.4242
5	3.0	9	93.2512	80.3468	33.3333
6	2.5	7	94.0141	84.9711	91.9697
7	2.5	7	94.0141	84.9711	89.3939
8	2.5	7	96.3615	83.8150	91.5152
9	2.5	9.8	94.1315	79.7688	39.3939
10	3.0	5	43.4859	80.9249	51.5152
11	2.5	7	97.0070	83.8150	92.4242
12	2.0	5	34.6831	79.7688	69.6970
13	2.0	9	95.7160	80.3468	90.9091

Analysis of variance (ANOVA);

The square model results for each response were evaluated by ANOVA to determine the "goodness of fit." In equations (Eq.3, Eq. 4, Eq. 5) some values were non-significant statistically due to its low F-value. Thus, it's important to disregard these values from the response equations. The P-value are more significant regarding each response equation for (Turbidity, COD and TSS removals) models and have the probability of alpha value 0.05 confidence level except in the interaction terms in each COD and TSS are not significant due to being 0.1 as shown in (Table 4). R- squared are used for predict the response in the model, R-squared near one provide more exact prediction since it measures how much inconsistency the values of an observed response [13]. The value of R- square for (TSS, Turbidity and COD removals) have been 0.9919, 0.7089, and 0.8337 respectively. Based on these results, there are high dependence and correlation between the predicted and observed values of the response for both Turbidity and COD, yet it's not high for the TSS. The adjusted R-sq. of (Turbidity, TSS, and COD removals) have been 0.9857, 0.5009, and 0.7149 respectively. Turbidity and COD adj. R- sq. value was close to R- sq. thus, it is satisfactory prediction [14], as for TSS the adj. R-sq. value shows a poor prediction.

Table 4: Flocculation model for pollutant from wastewater using (AlCl₃-PAM) composite polymer.

Source	Turbidity removal		TSS removal		COD removal	
	F-value	P-value	F-value	P-value	F-value	P-value
Linear	118.87	< 0.001	6.37	0.027	11.26	0.006
Square	134.51	< 0.001	7.76	0.017	16.53	0.002
2WI	2.52	0.157	1.30	0.291	0.29	0.605

Based on the ANOVA analysis shown in (Table 5). The non-significant terms of the equations (Eq. 3, 4, 5) were eliminated. Therefore, new response equations were obtained as shown in (Eq. 6, 7, 8).

Table 5: ANOVA table for square model of turbidity, TSS and COD removal

Source	Turbidity removal		TSS removal		COD removal	
	F-Value	P-value	F-Value	P-value	F-Value	P-value
Model	166.81	< 0.001	3.41	0.07	7.02	0.012
X ₁	230.3	< 0.001	11.82	0.01	9.46	0.018
X ₂	14.76	< 0.001	5.32	0.055	20.86	0.003
X ₁ ²	268.64	< 0.001	13.31	0.008	15.18	0.006
X ₂ ²	10.43	0.014	4.2	0.079	23.01	0.002
X ₁ X ₂	2.52	0.0157	1.3	0.291	0.29	0.605

$$Y_{\text{Turbidity}} = -437.8 + 98.04 X_2 + 104.7 X_1 - 5.473 X_2^2 - 15.81 X_1^2 \quad (6)$$

$$Y_{\text{TSS}} = -661 + 108.0 X_2 + 306 X_1 - 5.92 X_2^2 - 48.8 X_1^2 \quad (7)$$

$$Y_{\text{COD}} = 16.9 + 5.97 X_2 + 37.38 X_1 - 0.391 X_2^2 - 7.05 X_1^2 \quad (8)$$

Flocculation process:

The contour and 3D surface plots for each model display each experimental variable response in order to define the main interactions between the variables used in these plots. For turbidity removal, (figure 1) shows the optimum polymer dose was 2.5 (mg/L) were the removal of turbidity reached 97% at pH value of 7. This removal was due to ionization of AlCl₃-PAM thus Al⁺³ becomes ionized therefore residual charge were neutralized on particulates and expand the bridge chain. In pH neutral the hydrogen ions (H⁺) and hydroxyl ions (OH⁻) are equal. Thus, the composite polymer (AlCl₃-PAM) can effectively reduce the turbidity.

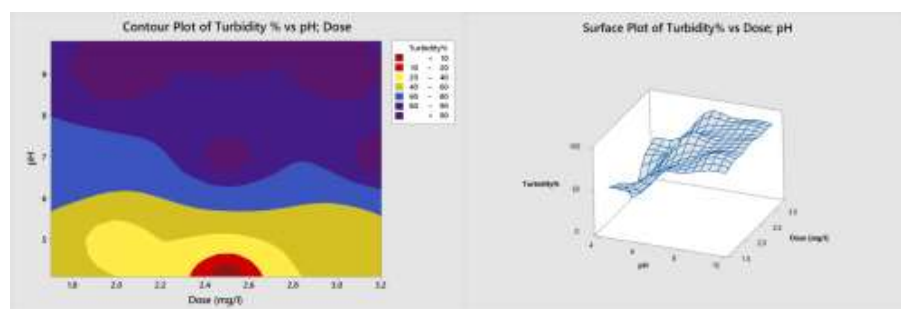


Figure 1: Contour plot and 3D surface plot for turbidity removal.

Contour and 3D surface plots for COD removal (figure 2) present the optimum removal percentage of 84.9% at polymer dose of 2.5 (mg/L) and pH 7. The COD are removed due to the presence of the Al(OH)₃-PAM complex which formed by the aluminium hydroxide precipitate [15].

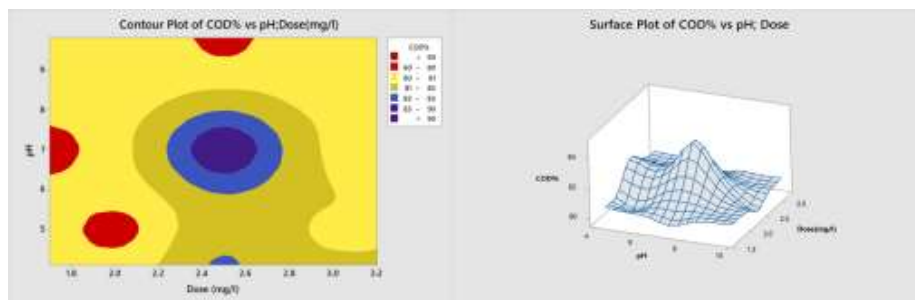


Figure 2: Contour plot and 3D surface plot for COD removal.

Contour and 3D surface plots in (figure 3) show an optimum removal for TSS of 92.4% at a polymer dose 2.5 (mg/L) and pH of 7. The removal of total suspended solids is similar to the turbidity removal because of the effect of the polymer on the particulates and suspended solids[16].

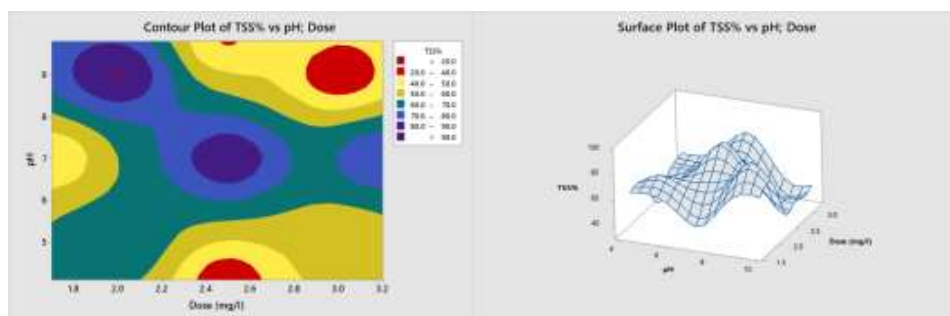


Figure (3): Contour plot and 3D surface plot for TSS removal.

Conclusion

In This study a composite polymer (PAM- AlCl_3) was prepared and utilized in wastewater treatment. RSM and CCD were utilized to improve and optimize the flocculation process of the wastewater. Analysis of variance (ANOVA) was performed to check the results and study the effect of the pH and composite polymer wastewater dose on optimal operating conditions. Thus, we concluded that the optimum dosage of the composite polymer was (2.5 mg/l) and optimum pH of (7) achieving (97%, 92.4%, and 84.9%) Turbidity, TSS, and COD removal respectively.

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