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Polymer Induced Flocculation for Treatment of a Tile Factory Wastewater using Polyacrylamide (PAM): Optimization by Response Surface Methodological Analysis

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Abstract
Flocculation is an important industrial process for solid-liquid separation during the primary purification of wastewater. Effects of the solution pH and flocculants dosage to examine the performance of five commercially available cationic & anionic polyacrylamides (Chemfloc 3876, Chemfloc 1510C, Chemfloc 530A, AN913 and AN913 SH), with different molecular weights and different charge densities to treating the tile factories wastewater were investigated. To minimize turbidity and total suspended solids (TSS), the experiments were carried out using jar tests, and response surface methodology (RSM) was applied to optimize this process. The optimal conditions for flocculent dosage and pH were determined 20 mg/l and 3-5.25, respectively, where 99.6% of TSS removal and final turbidity of 18 NTU could be obtained for Chemfloc 1510C. This paper shows that higher molecular weight polymers improved initial aggregation but the effect of cationic charge was more important for a stable flocculation. The results suggest that single-polymer system can be used alone in the coagulation-flocculation process due to the efficiency of the polyacrylamide.

Keywords: Flocculation, Polyacrylamide, Tile Industry Wastewater, Turbidity, RSM.

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1. Introduction

Depending on rapid developments in tile processing application due to the increasing demands for tile in civil engineering applications, the number of the tile processing plants and the workshops and eventually the trading volume has increased in recent years. As a result of this increasing trading volume, the wastewater discharged into water bodies after processing of tile blocks has also increased (Solak et al., 2009). To the knowledge of author, there are only a few studies about the flocculation of inorganic industries wastewater. Ersoy (2005) studied the effect of suspension pH and polymer charge density on flocculation of NSS (natural stone suspension) in terms of settling rate and turbidity. The main

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objective of this work was to optimize the flocculation process and investigate the flocculation efficiencies of various types of PAM flocculent and interactive effects of experimental factors, including pH and flocculent dosage.

In this process, many factors can influence its efficiency, such as the type and dosage of coagulant/flocculent (Amudaa & Amoo, 2007; Lee et al., 2009), pH (Song et al., 2004; Zhao et al., 2011), mixing speed and time (Chong et al., 2009; Merzouk et al., 2011; Ugurlu et al., 2008), temperature and retention time [Kushwaha et al., 2010; Duan & Gregory, 2003) etc. The optimization of these factors many significantly increase the process efficiency. In conventional multifactor experiments, the response surface methodology (RSM) has been proposed to determine the influences of individual factors and their interactive influences. The RSM is a statistical technique for designing experiments, building models, evaluating the effects of several factors, and searching optimum conditions for desirable response and reducing number of experiments. In this study, the RSM was employed for the optimal experimental design of flocculation process (Wang et al., 2007). RSM uses an experimental design such as the central composite design (CCD) to fit a model by least squares technique (Ahmad et al., 2007). Adequacy of the proposed model is then revealed using the diagnostic checking tests provided by analysis of variance (ANOVA). The present investigation aims at optimization of flocculent dosage and pH to achieve highest removal of total suspended solids (TSS) and lowest final turbidity from tile wastewater using cationic and anionic polyacrylamide. The experiments were carried out by jar test which is usually employed to evaluate the treatment process efficiency.

Table 1. Chemical characteristics of the wastewater used.

Parameters	Values
TSS (mg/L)	8800
Turbidity (NTU)	4000
Alkalinity(mg $CaCo_3$/L)	1400
pH (average)	7.3
Color	Brown

2. Methodology

The tile wastewater effluents utilized for this study were taken from- chini kord -factory, nearby Kermanshah, Iran. Wastewater (400m3/h) is discharged into sedimentation pools. The primary TSS content of raw wastewater 22800 mg/l was obtained. But the raw wastewater sample is allowed to settle for 30 minute and the supernatant was taken with TSS content of 8800 mg/l. And this sample is ready to experiment. The wastewater samples were characterized and the analyses are given in Table 1. These parameters were measured based on the Standard Methods for the Examination of Water and Wastewater (APHA).

Various cationic polyacrylamides (C-PAM) and anionic polyacrylamides (A-PAM) of commercial grade in wide range of molecular weight and charge density were used. The properties of the PAM used are shown in table 2. Distilled water was used to prepare all the PAM feedstock solution of 2000 mg/l.

Table 2. The properties of the polyacrylamide used.

Polyelectrolytes	Molecular weight	Charge density	Charge
Chemfloc 3876	Medium	Cationic	High
Chemfloc 1510	Medium	Cationic	Medium
Chemfloc AN 530	High	Anionic	High
AN 913	High	Anionic	Low
AN 913SH	Very high	Anionic	Low

The experiments were carried out in at laboratory bench scale using a jar test apparatus (SK/ 2008), using 240 ml wastewater samples with the C-PAM and A-PAM dosage of 10, 15, 20, 25, 30 mg/l, and keeping other variables constant. The selected polyelectrolyte dosage was added to 240 ml of wastewater and it was stirred for a selected mixing rate and mixing time, at selected pH value. The flocs formed were allowed to settle, and after settling, the turbidity and TSS of the supernatant were determined.

The experiments were repeated tow times to get the average value. For the other variables this trend carried out. The parameters were determined according to the APHA method. A compare the differences of the trend of each PAM on the removal of TSS and reduction of turbidity was carried out using the central composite design at response surface methodology (RSM) available in the design-expert software. The pH value of 240 ml wastewater sample was adjusted to pH in the range of 3-12, respectively, by using 1N HCl or 1N NaOH.

A pH meter model 3510 (from JENWAY) was used to measure the solution pH. Turbidity was measured by turbidity meter (model 2100 Turbidimeter HACH Company). The TSS concentration was determined by the filter was weighed after drying in the oven at 115º C for 30 min. The RSM used in the present study was a CCD involving tow numerical factors; flocculent dosage and pH, and categorical factor; types of flocculants. The ranges and the levels of the variables investigated in the study and the responses of these factors are given in table 3 and 4, respectively.

Table 3. Experimental range and levels of the indpendent variables.

Factor	Name	Type	Low actual	High Actual	Low coded	High coded	Mean
A	flocculent dosage, (mg/L)	Numeric	10	30	-1	+1	20
B	pH	Numeric	3	12	-1	+1	7.5
E	Type of coagulant	Categorical	Level of E	Level 5 of E	-	-	-

Table 4. Responses of the factors.

Respons	Name	Units	Analysis	Min	Max	Mea	Ratio	Trans.	Model
Y1	Final Turbidity	NTU	Polynomial	18.00	1250	400.29	69.44	Natural log	RQuadratic
Y2	TSS removal	%	Polynomial	76.00	99.60	91.35	1.31	None	RQuadratic

The quadratic equation model (Pavanelli & Bigi, 2005) for predicting the optimal point can be expressed according to Eq. (1):

$$R = \beta_0 + \sum \beta_i X_i + \sum \beta_{ij} X_i X_j + \sum \beta_{ii} X_i^2 + \varepsilon \quad (1)$$

Where R is the response,β_0 is the constant term,β_i represents the coefficients of the linear parameters, β_{ii} represents the coefficients of the quadratic parameter, β_{Ij} represents the coefficients of the interaction parameters X_i and X_j and i < j , X_i and X_j represents the variables and ε is the random error or noise to the response. The Design Expert Software (version7.0.0, Trial) was used for regression. Analysis of variances (ANOVA) was used for graphical analyses of the data to obtain the interaction between the process variables and the responses. The quality of the fit polynomial model was expressed by the coefficient determination R^2, and its statistical significance was checked by the Fisher F-test in the same program. Model terms were selected or rejected based on the *P* value (probability) with 95% confidence level. Three-dimensional plot and their respective contour plots were obtained based on the effects of the levels of four factors (flocculent dosage, pH, mixing time and mixing rate). From these three-dimensional plots, the simultaneous interaction of four factors on the responses was studied.
The optimum region was also identified based on the main parameters in the overlay plot.

3. Results

Development of mathematical equations where each response variable R is assessed as a function of flocculant dosage (A), pH (B), and type of flocculent (E) and calculated as the sum of constant, first-order effects (terms in A, B, E), one interaction effect (such as AB, BE etc.) and second order effect (A^2 , B^2) according to Eq. (1). The results obtained are then analyzed by ANOVA to assess the "goodness of fit". Only terms found statistically significant are included in the model. Values of "Probe > F" less than 0.0500 indicate model terms are significant. The quadratic model is well fitted to the observed data and the following empirical models in terms of coded values are obtained for the turbidity and TSS removal.

3.1 Turbidity

Y1 = +6.43 +0.22A + 0.37B – 0.091AB + 1.43A² – 2.32B² - 0.97E[1] - 1.52E[2] + 1.21E[3] + 0.26E[4] +1.27E[5]

3.2 TSS removal

Y2 = +84.34 -1.34A – 1.07B – 7.95A² + 14.15B² + 7.49E [1] + 8.25E[2] -11.04E[3] +4.40E[4] – 10.9E[5]

Where: E[1] = Chemfloc 3876, E[2] = Chemfloc 1510C, E[3] = Chemfloc 530A, E[4] = AN 913 and E[5] = AN 913 SH. Table 4 represent the statistical parameters obtained from the ANOVA for the reduced models of the responses.

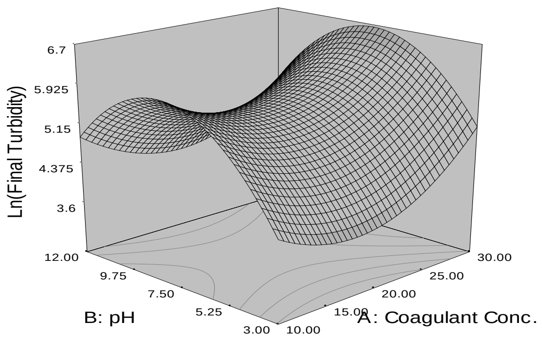
Table 5. Statistical parameters obtained from the ANOVA for the reduced models.

Variable	Turbidity	TSS removal
Significant terms	A, B, AB, A², B², E	A, B, A², B², E
R²	0.9768	0.9720
R² adjusted	0.9738	0.9684
Prob > F	<0.0001	<0.0001
Adequate precision	66.012	55.614
Standard deviation, S.D.	0.18	1.25
Coefficient of variance, CV	3.17	1.37

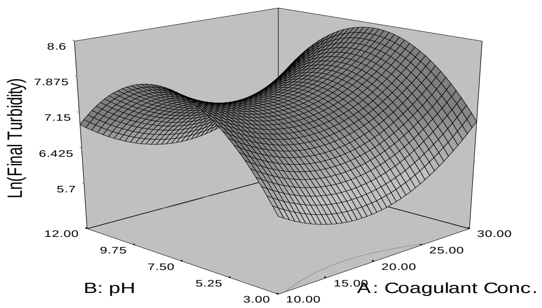
3.3 Turbidity

Turbidity, a measure of the light-transmitting properties of water, is used to indicate the clarity of treated wastewater with respect to colloidal and residual particulate matter. Turbidity in water is caused by the presence of suspended particles that reduce the clarity of the water. Turbidity is one of the indicators that show flocculation efficiency.

From the ANOVA results, final turbidity was function of A, B and E; Figure 1 shows the response surface plots for turbidity values of the supernatant liquid as a function of pH and flocculation dosage in the presence of chemfloc 1510 (a) and AN913 (b). These trends are similar to other PAM as flocculent.



(a)



(b)

Figure1. Design-expert plot. Response surface plots for turbidity, chemfloc 1510 (a) and AN913SH (b).

3.4 pH

The initial pH of wastewater is a key parameter in the coagulation & flocculation process. Only the use of the optimum pH display maximum pollutant removal. In order to determine the optimum pH, various concentrations (10 - 30 mg/l) of each PAM's was added to each sample at different pH values, adjusted using HCl 1N & NaOH 1N. The turbidity decrease with a decrease and increase of pH values from the natural pH value of solution (pH =7.3), and it reach to 18 NTU for chemfloc 1510, 67 NTU for chemfloc 3876, 380 NTU for AN530, 220 NTU for AN913 and 400NTU for AN913. This may be attributed to destabilization effect of H^+ at low pH and destabilization effect of OH^- ions at high pH on the negatively charged inorganic particles. Since, in addition to inorganic ions such as Ca^+, CO_3^- , Na+ and HCO_3^- and etc. H^+ and OH^- ions can be considered as potential determining ions for this type of inorganic wastewater. Therefore at low and high suspension pH, the inorganic particles of colloidal sized may be coagulated prior to flocculation and thus the turbidity in the supernatant liquid decreased (Gregory, 1989).

From this figure it is clearly seen that acidic pH (3 - 5.25) gave the best performance for all the polymers, while pH 7.5 (natural pH of solution) gave the maximum turbidity by all the polymers. A similar case is also reported by B.ersoy earlier on flocculation of NSS (natural stone suspension) by PAM [2]. Unlike this paper Ersoy (2005) determined that the anionic polymer gives the lowest turbidity for NSS. At all range of pH values, cationic 1510 polymer gave the lowest turbidity (the highest clarity), while by 530, 913 & 913SH, the anionic polymers, the worst (higher turbidity) results have been obtained. This is attributed to negatively charged density of these polymers and the repulsive force of solution negative ions. The efficiency of the C-PAM in the reduction of turbidity is impressive, even at alkalinity range.

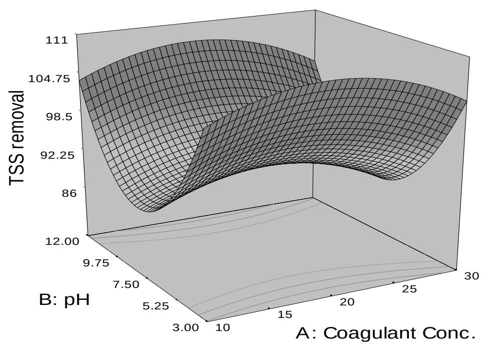
3.5 Concentration

From Fig. 2 (a) and (b) it is clearly observed that flocculent concentration has the subtle effect at the turbidity decreasing at all pH range (particularly in natural pH of suspension: 7.3.); The turbidity decrease with an increase of polymer dosage to a certain dosage, further increase in the dosage leads to an increase in turbidity again owing to the dispersion of flocculated colloidal particles. Dispersion of colloidal inorganic particles at high polymer dosage results from the excess polymer adsorption at which the polymer bridging mechanism is prevented. Because there is insufficient free particle surface for bridging contacts to occur and the adsorbed layers may also cause steric repulsion [19]. The optimal dosages of the flocculants are based on the reduction of turbidity; The dosage beyond not only there is no significant enhancement with further addition of polyacrylamide but also increase turbidity values for all flocculants, that is defined as the optimum dosage. The optimum dosages of each of polymers are 20 mg/l.

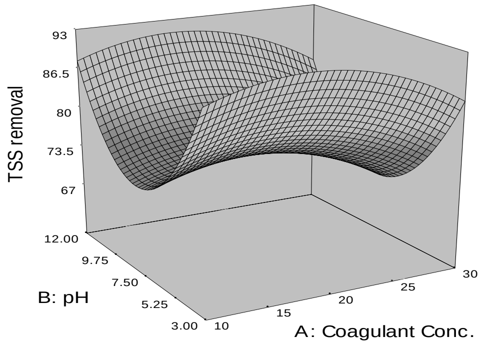
At all dosages, cationic 1510 polymer gave the lowest turbidity (the highest clarity) while by 530 & 913SH two anionic polymers, the worst (higher turbidity) results have been obtained. This is attributed to inefficiency of tow anionic polymers (530 & 913SH) to induce polymer bridging mechanism. It can be seen clearly from Fig .3-1that the A-PAM are not as effective as C-PAM in the reduction of turbidity (except to AN913). The highest reduction of turbidity is only 84% recorded by the anionic PAM 530AN with high MW and high charge density at the dosage of 20mg/l. Barany and Szepesszenengyorgyi (2004) describe that for the optimum aggregation of more concentrated suspension, a lower amount of polymer is needed, and the addition of this amount results in a high degree of flocculation. This result suggested that the optimum values of pH and concentration, for all flocculants in terms of turbidity, are 3 and 20 mg/l, respectively. Chemfloc 1510 cationic is the highest efficient among the cationic & anionic PAM studied.

3.6TSS removal

The concept behind turbidity is that when a light beam passes through a wastewater sample, it is subjected to scatter, transmission and adsorption because of the presence of particles in suspension (Pavanelli & Bigi, 2005). This is the reason why both the results of TSS and turbidity removal were normally similar in trend. Generally the total suspended solids removal increase with increasing dosage of PAM. The addition of PAM will neutralize and create a collision condition between particles, thus forming small flocks. Apart from that, the incorporation of PAM will increase flock size by forming a bridge between them. The total suspended solid will increase when the flocks formed are many and bigger (Yan et al., 2004). Total suspended solids concentrations were calculated based on the difference in weight prior to and following drying and results recorded and reported in mg/l TSS. Slurry solids dry weight was determined by heating samples to dryness and calculating solids dry weight basis in mg/l by comparing initial to final weights. From the ANOVA results, TSS removal was function of A, B and E; these trends are similar to other PAM as flocculent. Figure 2 shows the response surface plots for TSS removal values of the supernatant liquid as a function of pH and flocculation dosage in the presence of chemfloc 1510 (a) and AN913 (b).



(a)



(b)

Figure 2. Design-expert plot. Response surface plots for TSS removal, chemfloc 1510 (a) and AN530 (b).

3.7 pH

The percentage TSS removal of C-PAM & A-PAM at various pH of solution are shown in figure 2 .The removal trends of the TSS are similar to those of the turbidity removal for both of C-PAM and A-PAM. It clearly seen that pH 3 & 12 gave the best performance for all the polymers, such turbidity this may be attributed to destabilization effect of H^+ and OH^- , at low and high pH , respectively. In Fig. 3.2, the optimum pH of the C-PAM and A-PAM in the removal of TSS is 3 with 99.6 % and 96.2 %, respectively, for chemfloc 1510 and AN913SH. S.S Wong et al (2006) who studied the purifying of pulp and paper wastewater by polyacrylamide reported that C-PAM are more effective than A-PAM. Thought the A-PAM exhibit encouraging results in terms of TSS removal, but the C-PAM show much better removal efficiency than A-PAM in the TSS removal. The removal efficiency of TSS with C-PAM and A-PAM, at optimum conditions in terms of pH and flocculent dosage are more than 98% and 96%, respectively.

3.8 Concentration

Similar to the result obtained for the removal of turbidity, the TSS removal increases with an increase of polymer dosage to a certain dosage, further increase in the dosage leads to a decrease in TSS removal again owing to the dispersion of flocculated colloidal particles, Ersoy (2005) in line with the results of this study. The percentage TSS removal of C-PAM and that of A-PAM at optimum dosage 20mg/l and optimum pH value 3 are: chemfloc 3876 with 98% removal, chemfloc 1510 with 99.6% removal, AN530 with 82% removal, AN913 with 96.96% removal and AN913SH with 84% removal. Based on the reduction and removal efficiencies of the turbidity and TSS by the PAM studied, the flocculation performances of the PAM are influence by its molecular weight and charge density as can be seen from Figs. 3-1, 3-2. In the case of C-PAM, the C-PAM with medium molecular weight and positive charge density performed better than high molecular weight A-PAM. Medium positive charge density (Chemfloc 1510) is the most appropriate charge density for the C-PAM to obtain better turbidity reduction and TSS removal. The molecular weights of the A-PAM have no significant effect on the turbidity reduction and TSS removal. From this figure A-PAM with high molecular weight & low charge density (AN 913) shows the best reduction and removal efficiency in turbidity and TSS.

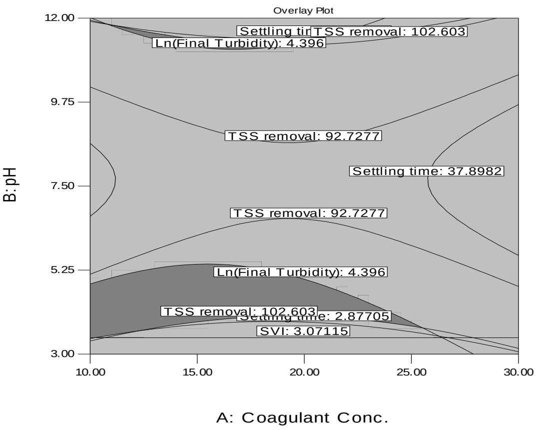


Figure 3. Design-expert plot. Overlay plot for optimal region. Chemfloc 1510.

3.9 Optimization

The optimum condition can be visualized graphically by superimposing the contours for various response surfaces in an overlay plot. By defining the limits of the TSS removal, final turbidity, SVI and settling time desired, the shaded portion of the overlay plot, as shown in Fig. 5, for chemfloc 1510, defines the permissible values of the dependent variables. The optimum region is made by considering TSS removal, turbidity, SVI and settling time in range of the values mentioned in the overlay plot. Based on the overlay plot, the optimum conditions for flocculent dosage and pH are 15-20 mg L^{-1} and 3-5.25, respectively. A confirmation of the results applying the flocculent dosage and pH for those fall in the optimum region is accomplished by repeating two additional experiments. Overlay plot obtained for chemfloc 3876 is similar, while there was no optimal region for AN530, AN913 and AN913 at specified range.

4. Conclusion

In this paper reduction of turbidity and removal efficiency of TSS have been studied using C-PAM and A-PAM as flocculants in treating tile wastewater. Response surface methodology using CCD was applied to determine the optimum operating conditions for maximum TSS removal and lowest turbidity. The flocculent dosage and pH are both significant terms to yield higher removal of TSS and minimum turbidity. The polymer charge and the suspension pH play a crucial role on the flocculation. The results show that C-PAM are more effective than A-PAM. Chemfloc 1510 with medium molecular weight and medium charge density is the best flocculent with highest flocculation efficiency for the treatment of tile wastewater. By applying RSM, the optimum region for the flocculation process operation is located. The optimum conditions for flocculent dosage and pH are 15-20 mg/l and 3-5.25, respectively; where 18 NTU of turbidity reduction and 99.6% of TSS removal can be obtained, for chemfloc1510. Based on cost evaluation, the use of PAM is economically feasible to treat the tile wastewater. This result suggests that single-polymer system can be used alone (without combination with inorganic coagulant) in the coagulation-flocculation process since the efficiency of the PAM is remarkable.

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