



Determination of the Effects of Operating Parameters on the Residual Turbidity of Quartz Suspensions by Design of Experiments

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Abstract

This research represents the results of flocculation of quartz particles in suspensions with an anionic flocculant by applying response surface method (RSM). The experiments were designed and carried out according to the Bohn-Behnken Design (BBD) which is a type of RSM. A BBD with five independent parameters at three levels was applied to jar test studies to investigate the effect of variables examined on quartz flocculation process. Flocculant dosage, rapid mixing time, rapid mixing rate, solid ratio and settling time were tested to evaluate the main and interaction effects of these factors on residual turbidity. An empirical quadratic model with a high correlation coefficient was obtained for the estimation of residual turbidity within the investigated ranges of parameters.

Key words

Quartz suspension, Flocculation, Response surface method, Box-Behnken Design

1. INTRODUCTION

Dewatering is a process identified as a part of solid/liquid separation and is an important process in most mineral processing operations. Generally, dewatering is accomplished by sedimentation or filtration ([1], [2]). Aggregation of fine particles in mineral suspensions can be carried out by applying methods such as coagulation, flocculation or agglomeration methods [3]. Knowledge of detailed information while applying an aggregation method on a special material are necessary to understand the mechanisms and to use less possible amount of reagents during the processes.

Flocculation is usually a necessary pretreatment step in many dewatering streams containing significant quantity of very fine particles. The purpose of flocculation is to form aggregates or flocs from finely dispersed particles with the help of long chain polymers which are referred to as flocculants. Flocculation of suspended particles by polymeric flocculants is a multistep process. A classical coagulation/flocculation process consists of three separate steps: i) Rapid or flash mixing: the suitable chemicals (coagulants/flocculants and if required pH adjusters) are added to the wastewater stream, which is intensively mixed at high speed. ii) Slow mixing (coagulation and flocculation): the wastewater is only moderately stirred in order to form large flocs, which are easily settled out. iii) Sedimentation: the floc formed during flocculation is allowed to settle out and is separated from the effluent stream ([4], [5]).

In a flocculation process applied, finding the optimum flocculation conditions is a very important entity. The flocculation of fine particle suspensions is a complex process and the effectiveness of the process depends not only on the usage of appropriate chemical reagents (coagulants, flocculants, etc.) but also on how they are applied. Flocculation is affected by the complex interactions between a numbers of factors. These factors may

include slurry properties such as particle size and surface charge of particles, solution chemistry, pH and physical variables such as mixing intensity (rate), mixing time and settling time. Each of them determines the flocculation rate and efficiency in term of settling rate, supernatant turbidity and sediment volume ([6], [7]). Therefore, determination of the flocculation behavior of the suspensions is important for an efficient solid/liquid separation. Jar test is one of the most efficient and commonly used methods to determine optimum flocculation conditions.

Classical jar test experiments are usually carried out by systematically changing the level of one factor at a time (OFAT). In OFAT jar test experiments, optimum flocculation conditions are determined by varying a single factor while holding the level of the other factors constant [8]. The level of the factor that results in the best response (e.g. lowest residual turbidity) is then selected and used in subsequent tests which continue in the same manner for other factors [9]. But it is time consuming and does not fully explore the whole experimental space to find the best factors' conditions. Also, it is incapable of identifying the interaction effects resulting from the factors being considered. The classical jar test experiments of OFAT and studying the effect of the variable on the response is a complicated technique, particularly in a multivariate system as in the case of flocculation or if more than one response are of importance. Design of experiments (DOE) is statistical techniques which can be used for optimizing such multivariable systems. For these reasons, DOE has been proposed for the jar tests to overcome the shortcomings and to determine the influences of individual factors and their interactive influences.

The response surface methodology (RSM) which is a combination of experimental, regression analysis and statistical inferences is one of the DOE approach. It is useful for modeling and analyzing problems in which responses of interest are influenced by several factors or variables and in which objective is to optimize the responses. The RSM not only reduces the cost and time, but also provides required information about the interaction effects with minimum number of experiments [10].

There are many scientific works investigating effect of slurry properties such as flocculant dosage, pH on flocculation in the literature ([11]-[21]). However, fewer researches exist about the mixing conditions on flocculation ([4], [21]-[26]). The effect of mixing conditions under constant slurry properties on residual turbidity for the flocculation of quartz sample with anionic flocculant used in this work have been investigated recently by us [25]. Results of this study showed that the effect of rapid mixing rate and time are more important factors compared to slow mixing rate, slow mixing time and settling time. We have also previously examined the effect of slurry properties (flocculant dosage, pH and solid ratio) at constant mixing and settling conditions on residual turbidity for the same material [13]. According to this research, the efficiency of quartz flocculation was dependent to a large extent pH of the suspensions and the excellent results were obtained at alkaline media. However, the results at natural pH were not good compared to acidic or alkaline suspensions. Interaction between solid ratio and flocculant dosages on residual turbidity of suspensions were found to be significant in all pH values tested.

In this study, the effects of five independent variables which include the slurry and mixing conditions of flocculation process namely, flocculant dosage, rapid mixing rate, rapid mixing time, solid ratio and settling time on the flocculation behavior of quartz suspensions were investigated at natural pH of the suspensions. Since numerous numbers of experiments are needed to research the effects of five variables tested, the efficiency of flocculation under tested conditions was determined to examine the main and interaction effects between these variables by the application of five parameter Box-Behnken design (BBD).

2. EXPERIMENTAL

2.1 Materials and Method

A pure quartz sample from Çine region of Turkey was obtained and used in the experiments. The particle size of the sample was less than 20 μm sieve aperture. According to the particle size distribution determined by Malvern Mastersizer 2000, d_{90} , d_{50} and d_{10} diameters were obtained as 15.7 μm , 2.2 μm and 0.6 μm respectively. According to the XRD results all the peaks belonged to quartz and the sample contains more than 95.6 % SiO_2 content indicating that the sample is pure enough. The quartz sample which may causes pollution was chosen as a pollutant material since it is considered as one of the common components present in soils and clays [27].

SPP 508 (supplied from Superkim, Turkey) polymer was used for the flocculation of synthetically prepared fine quartz suspensions. Medium anionic SPP 508 is a high molecular weight ($15\text{-}22 \times 10^6$ g/mol) polyacrylamide with 28% degree of ionization. A solution of polymer (0.01%) was prepared using distilled water. The pH of the solutions was not changed during the experiments and held constant at its natural pH. The neutral pH of samples was determined as 7.95 for all solid ratios by a pH-meter (Orion 5 Star).

A jar test apparatus (Velp Scientifica FC6S) was used in order to determine the effectiveness of flocculant in the experimental conditions tested. It consists of a set of six beakers, which can be stirred simultaneously at

specified speed. The flocculation tests consisted of three stages. First, the flocculant was added to the suspension and a rapid mixing was initiated. The objective is to obtain complete mixing of the flocculant with the suspension to maximize the effectiveness of destabilization of colloidal particles and initiate flocculation. Critical parameters for this stage are the rapid mixing time (duration) and the rapid mixing rate (intensity). Second, the suspension was slowly stirred to increase contact between flocculating particles and to facilitate the development of large flocs. In each experiment, a 10 minute slow mixing at 30 rpm was applied. Finally, mixing was terminated. The flocs are allowed to settle at predetermined settling times and then the turbidity of the supernatants was measured.

The turbidimeter (HF Scientific) was used to measure the residual turbidity. The turbidity is expressed in NTU (Nephelometric turbidity units). The initial turbidity of the samples were determined as 306 NTU for 0.1% solid ratio sample, 2496 NTU for 0.55% solid ratio sample and 4400 NTU for 1.0% solid ratio sample.

2.1 Design of Experiments

The surface response method RSM, using the Box-Behnken experimental design, yielded correlations between the residual turbidity of quartz suspensions and the five independent factors. The RSM involves an empirical model to evaluate the relationship between a set of controllable experimental factors and observed results. Factors considered included the flocculant dosage (mg/l), rapid mixing time (min), rapid mixing rate (rpm), solid ratio (%) and settling time (min). They are represented by *A* to *E*, respectively. The low, middle and high levels of each variable were designated as -1, 0 and +1 respectively, as listed in Table 1.

Table 1. Factors and levels for experimental design using BBD

Variables	Ranges and coded levels		
	-1	0	+1
A: Flocculant dosage (mg/l)	0.02	0.41	0.80
B: Rapid mixing time (min)	1	3	5
C: Rapid mixing rate (rpm)	100	200	300
D: Solid ratio (%)	0.1	0.55	1.0
E: Settling time (min)	5	15	25

The experiments were carried out according to the BBD which is given in Table 2. The independent variables and the mathematical relationship between the response *Y* and these variables can be approximated by a quadratic polynomial equation (1):

$$Y = b_0 + b_1A + b_2A + b_3A + b_4A + b_5A + b_{12}AB + b_{13}AC + b_{14}AD + b_{15}AE + b_{23}BC + b_{24}BD + b_{25}BE + b_{34}CD + b_{35}CE + b_{45}DE + b_{11}A^2 + b_{11}B^2 + b_{33}C^2 + b_{44}D^2 + b_{55}E^2 \tag{1}$$

Where; *Y* is the predicted response variable (turbidity), *b*₀ is the model constant, *b*₁ – *b*₅ linear coefficients, *b*₁₂, *b*₁₃, *b*₁₄, *b*₁₅, *b*₂₃, *b*₂₄, *b*₂₅, *b*₃₄, *b*₃₅, and *b*₄₅ are the cross product coefficients and *b*₁₁, *b*₂₂, *b*₃₃, *b*₄₄ and *b*₅₅ are the quadratic coefficients [28]. Factors and levels and Box-Behnken design of five experimental variables in coded units are given in Table 1 and Table 2 respectively.

Table 2. Box-Behnken design of five experimental variables in coded units

Run	Factors					Run	Factors				
	A	B	C	D	E		A	B	C	D	E
1	-1	-1	0	0	0	24	0	-1	-1	0	0
2	1	-1	0	0	0	25	0	1	-1	0	0
3	-1	1	0	0	0	26	0	-1	1	0	0
4	1	1	0	0	0	27	0	1	1	0	0
5	0	0	-1	-1	0	28	-1	0	0	-1	0
6	0	0	1	-1	0	29	1	0	0	-1	0
7	0	0	-1	1	0	30	-1	0	0	1	0

8	0	0	1	1	0	31	1	0	0	1	0
9	0	-1	0	0	-1	32	0	0	-1	0	-1
10	0	1	0	0	-1	33	0	0	1	0	-1
11	0	-1	0	0	1	34	0	0	-1	0	1
12	0	1	0	0	1	35	0	0	1	0	1
13	-1	0	-1	0	0	36	-1	0	0	0	-1
14	1	0	-1	0	0	37	1	0	0	0	-1
15	-1	0	1	0	0	38	-1	0	0	0	1
16	1	0	1	0	0	39	1	0	0	0	1
17	0	0	0	-1	-1	40	0	-1	0	-1	0
18	0	0	0	1	-1	41	0	1	0	-1	0
19	0	0	0	-1	1	42	0	-1	0	1	0
20	0	0	0	1	1	43	0	1	0	1	0
21	0	0	0	0	0	44	0	0	0	0	0
22	0	0	0	0	0	45	0	0	0	0	0
23	0	0	0	0	0	46	0	0	0	0	0

3. RESULTS AND DISCUSSIONS

3.1 Model Fitting

The residual turbidity values obtained and predicted by the BBD model in each experiment are presented in Table 3. Analysis of the Box-Cox plots showed that residuals could be reduced significantly by a log transformation since the residual turbidity values has broad range of response from 3.3 to 62 NTU.

The Box-Cox plot provides a guideline for selecting the correct power law transformation ($y' = y^{\lambda}$). For this reason, the dependent variable produced after transformation transformation was named as Ln (Turbidity).

Table 3. Response results and predicted turbidities in supernatants

Run	Turbidity		Ln of Turbidity		Run	Turbidity		Ln of Turbidity	
	Obs.	Pre.	Obs.	Pre.		Obs.	Pre.	Obs.	Pre.
1	57	48.13	4.04	3.84	24	19.2	25.79	2.95	3.12
2	38	26.15	3.64	3.34	25	7.5	6.31	2.01	1.86
3	27	32.90	3.3	3.27	26	3.5	10.42	1.25	1.83
4	11	13.93	2.4	2.28	27	3.3	2.44	1.19	1.45
5	14	16.76	2.64	2.86	28	9.3	22.97	2.23	3.06
6	19.8	13.79	2.99	2.54	29	21.7	36.04	3.08	3.81
7	15	20.71	2.71	2.93	30	62	53.82	4.13	4.10
8	7.5	4.44	2.01	1.56	31	7.3	-0.21	1.99	1.86
9	16	19.63	2.77	2.71	32	18.5	13.86	2.92	2.56
10	5	5.66	1.61	1.91	33	3.8	3.30	1.34	1.62
11	15	19.38	2.71	2.75	34	17	12.92	2.83	2.49

12	4.5	5.91	1.5	1.91	35	4.2	4.23	1.44	1.73
13	53	47.39	3.97	3.98	36	34	35.95	3.53	3.41
14	19	19.46	2.94	3.18	37	16.3	14.47	2.79	2.63
15	31.6	30.31	3.45	3.08	38	32.5	34.94	3.48	3.39
16	12.5	17.29	2.53	2.39	39	16.8	15.47	2.82	2.69
17	17.2	15.18	2.84	2.84	40	32	24.55	3.47	3.00
18	7.9	10.68	2.07	2.20	41	16	4.52	2.77	2.07
19	17.2	13.38	2.84	2.68	42	8.9	15.55	2.18	2.44
20	11.5	12.48	2.44	2.42	43	5.5	8.13	1.7	1.73
21	4.6	6.69	1.52	1.78	44	7	4.29	1.95	1.59
22	4.6	6.69	1.53	1.78	45	7.2	4.29	1.97	1.59
23	4.8	6.69	1.57	1.78	46	4.8	4.29	1.57	1.59

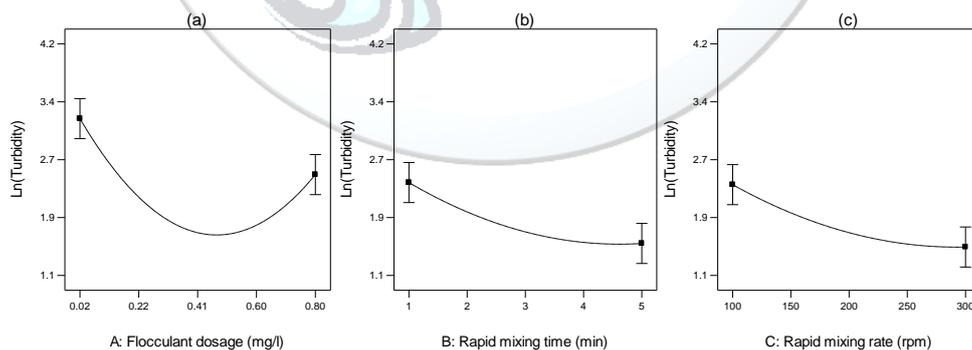
After evaluation of experimental results, a quadratic function for the residual turbidity in terms of coded factors was obtained with a determination coefficient of 0.84 (R^2) and hence correlation coefficient of 0.9165 (R) as:

$$\ln(\text{Turbidity}) = 1.68 - 0.37A - 0.41B - 0.42C - 0.23D + 0.012E - 0.12AB + 0.03AC - 0.75AD + 0.02AE + 0.22BC + 0.05BD - 0.01BE - 0.26CD + 0.04CE + 0.09DE + 1.15A^2 + 0.25B^2 + 0.23C^2 + 0.47D^2 + 0.29E^2 \quad (2)$$

According to this result, the 84 % of the variance can be explained by Eq. 2 and 16% of the variance could not be defined by the model.

3.2 Main Effects of Parameters

The main effect of individual variables of A , B , C , D and E and the perturbation plot which shows the comparison of all factors are plotted in Fig. 2 (a-e) and Fig. 2(f) respectively. In Fig. 2 (a-f), the log transformed residual turbidity is plotted by changing only one factor over its range while the others held constant at their center points. Therefore, both individual and perturbation plots show the main effects of these parameters on residual turbidity at center points of parameters. All individual variables were found to have their own important effect of residual turbidity.



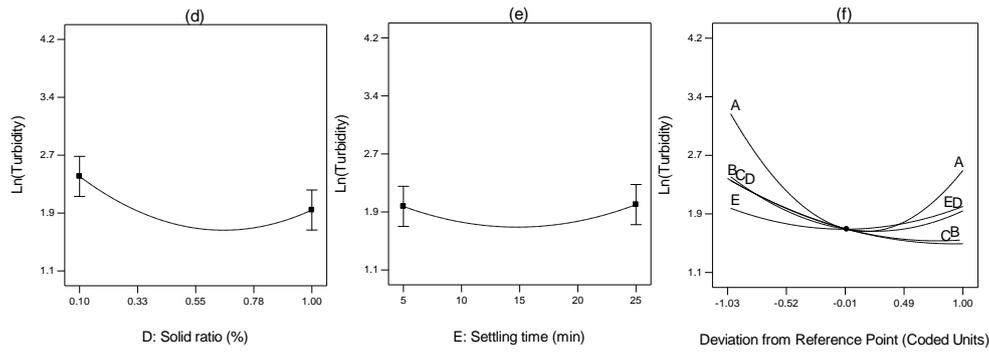


Figure 2. Main effects of flocculation variables (a-e) and perturbation plot for the response (f). For each factor, the remaining factors have held at their middle levels.

It is seen that variables have a curvature effect indicating that the turbidity removal by quartz flocculation within the investigated ranges of variables could be adequately explained by a second order model used in this study. Since the flocculation is considered as a second order rate process, the quadratic model by BBD can adequately describe the flocculation of quartz suspensions within the ranges of variables tested. According to the main effects plots in Fig. 2, the flocculant dosage, A, was the most effective parameter (Fig. 2.a). Initially, the residual turbidity decreases as the increasing of A and thereafter again increases. Altered rapid mixing time (B) and rapid mixing rate (C) affect the residual turbidity. An increase in these operating variables results in an improvement supernatant turbidity as seen in Fig 2 (b-c). While keeping the all parameters at their middle values, solid ratio had a quadratic effect on residual turbidity as shown in Fig. 2(d). Settling time (E) had less a little curvature effect on the response (Fig. 2.e). However, it is not very significant statistically in the investigated range.

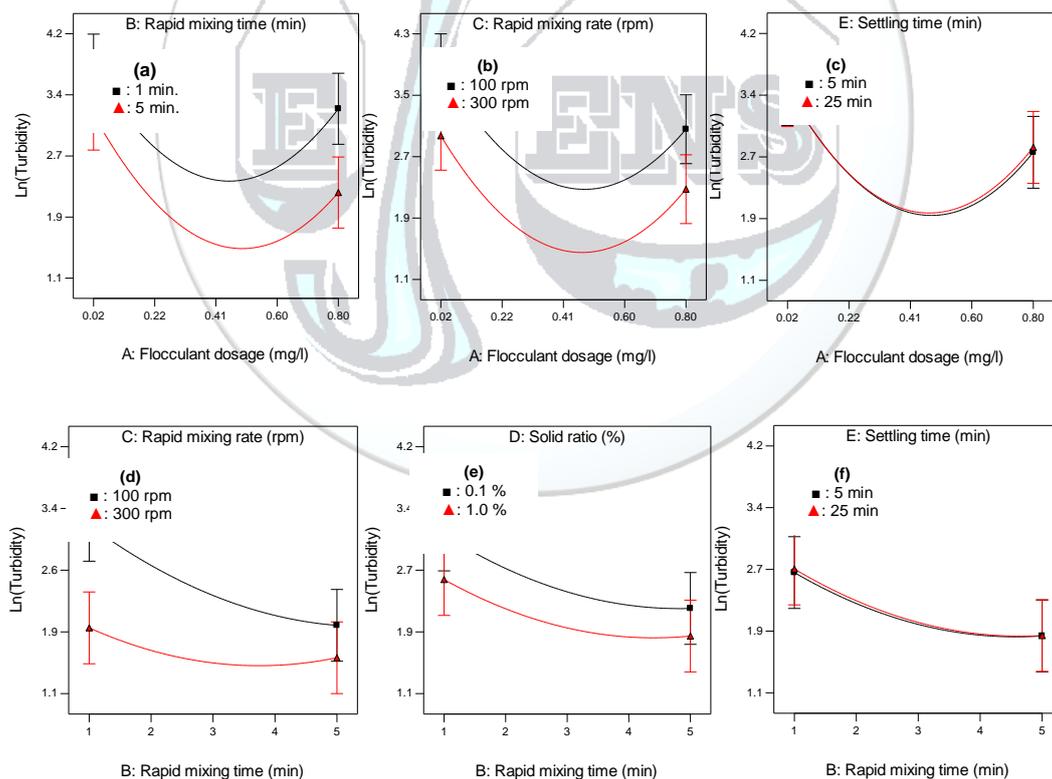
3.3 Interactions Between Factors

Figure 3 (a-i) shows the second order interactions among the factors and curvature effects as predicted by the second order model. The log transformed residual turbidity is plotted by changing only one factor over its range while the others held constant at their center points in Fig. 3 (a-i). It should be noted that the response variable values plotted as a function of one operating parameter over a range of values and the other parameter at two levels indicate the parameter interaction effects. In other words, lines in parallel indicate no parameter interaction effects between the two variables considered. Fig. 3 shows that there are no significant interactions between the variables represented on these plots. The most efficient interaction was found between flocculant dosage (A) and solid ratio (D) and it is given in Fig. 4. However, we can see that altered the process variables from their low values to high values affect the responses significantly (Figs. 3.a, 3.b, 3.d, 3.e and 3.g). On the other hand, changing settling time from 5 min. to 25 min. did not affect the responses considerably since the flocs quickly settle out within the first 5 minute (Figs. 3.c, 3.f, 3.h and 3.i).

In the interaction plots presented in Fig. 3, we can see the effect of rapid mixing rate and time on the residual turbidity of quartz suspensions achieved. In Fig. 3(a, b, d, e, f, g and h) it can be seen clearly that increasing rapid mixing rate from 100 to 300 rpm and increasing the rapid mixing time from 1 to 5 minutes reduces the supernatant turbidities of quartz suspensions. These plots indicate that higher turbidity removal can be achieved by keeping the rapid mixing time and rate at their maximum values when the other variables held constant at their middle values. The first stage of flocculation process is to add the flocculant to the suspension and then a rapid and high intensity mixing is initiated. Reference [7] states that the initial mixing intensity and the mixing time are the most important parameters in the determining the size of the flocs formed and rapid mixing conditions can have major effects on the flocculation process [7]. The flocculants are usually added as fairly concentrated and viscous solutions during the flocculation process. Therefore, intense mixing is needed to achieve rapid and uniform distribution of the polymer molecules throughout the suspension [29]. The objective is to obtain complete mixing of the flocculant with the suspension to maximize the effectiveness of destabilization of suspended particles. The effects of fast stirring rate on flocculation process are well known and some detailed studies have been carried out ([22]-[26]). Reference [7] showed that the poor performance of a flocculation process in the many applications could be attributed to inadequate mixing [6]. If the fast mixing rate is not enough, the flocs are hardly grow and longer flocculation time is required. Insufficient mixing can also

lead to local overdosing and restabilization of some particles, which is responsible for the residual haze in suspensions ([22]-[24]). On the contrary, too high intensity of mixing rate and long mixing time may cause breakup of flocculated flocs and this may cause detrimental effects on flocculation [30]. According to Fig. 3d, at constant solid ratio, flocculant dosage and settling time, more stirring time is required at low stirring rate of 100 rpm to achieve the same turbidity in the supernatant solution. However, at highest stirring rate of 300 rpm, residual turbidity remains almost constant after 3 min fast mixing time.

The effects of fast mixing rate/time and solid ratio on residual turbidity are shown in Fig. 3e and 3g when the other variables held constant at their midpoints. As seen in these plots, there are no clear interactions between rapid mixing rate-solid ratio and rapid mixing time-solid ratio parameters. Increasing rapid mixing time decreases the residual turbidity for all solid ratios but it has a more pronounced effect for the higher solid ratio at constant mixing rate of 200 rpm, flocculant dosage of 0.41 mg/l and settling time of 15 minutes (Fig. 3e). In addition, higher turbidity removal was achieved with increasing rapid mixing time from 1 min. to 5 min. Confirming these findings, it was shown that the effects of mixing conditions are much more apparent for suspensions with higher solid concentrations ([22], [23],[26]). Also, these findings confirmed that flocculation is a second order rate process and flocculation rate depends on the square of particle concentration [29]. These may be due to the higher collision probability which causes adsorption of flocculant molecules onto particle surfaces and flocculation of particles for the higher particle concentrations. It is possible to achieve the same turbidity values at rapid mixing rate of 100 rpm for all solid concentrations but less turbidity are accomplished by increasing the mixing rate for the solid ratio of 1.0% while the residual turbidity values remain almost constant for the solid ratio of 0.1% (Fig. 3g). These results may be attributed to the increase in collision probability between the quartz particles and flocculant at higher solid concentrations [30].



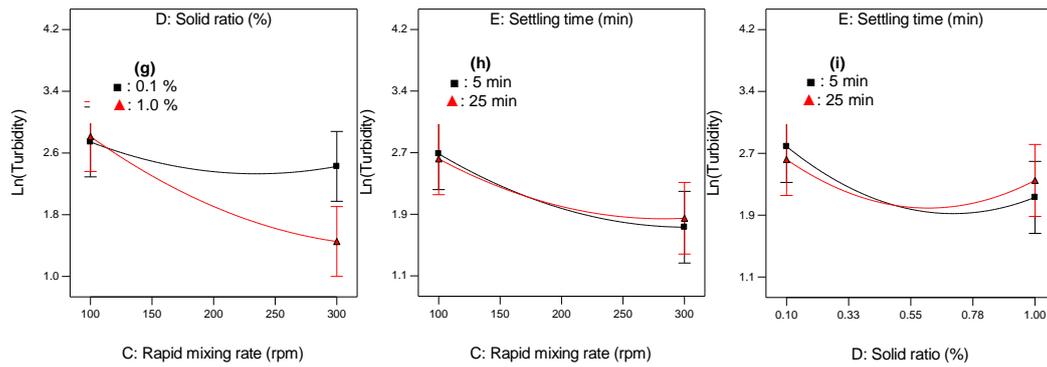


Figure 3. Interaction plots showing effects of flocculation parameters on residual turbidity. For each pair of factors, the remaining factors have held at their midpoint values.

The effects of flocculant dosage and solid ratio on residual turbidity while keeping the other variables at their midpoint levels are shown on the interaction and contour plots in Fig. 4.

As seen from these plots, there is an important interaction between the solid ratio and flocculant dosages. The residual turbidity is decreased with increasing flocculant dosages then increases again after a critical flocculant dosage for the both low and high solid ratio. As seen, less amount of flocculant is sufficient for the solid content of 0.1%. It is well known from the literature that, the optimum polymer dosage generally increases proportionally when particle concentration increases [29]. These critical flocculant dosages indicate the half surface coverage point described by References [2] and [31]. Excess amount of flocculant higher than optimum dosages starts to reverse effect on the residual turbidity for all solid concentrations. In Fig. 3a and 3b, we can see similar effect of flocculant dosage with rapid mixing speed and time and on the supernatant turbidity at constant solid ratio of 0.55% and settling time of 15 minutes. In these figures, residual turbidity decreases with increasing of flocculant dosage up to an optimum dosage and then increase again with over dosage of flocculant.

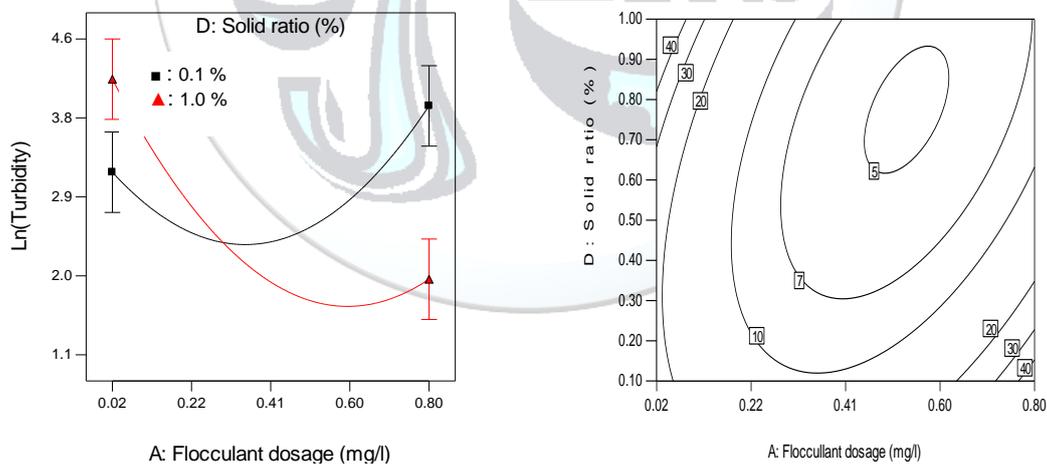


Figure 4. Interaction graph (left) and contour plot (right) of residual turbidity showing effect of solid ratio and flocculant dosage. The other variables are held constant at their midpoint levels.

3.4 Simplified Model for Residual Turbidity

Since some terms of the model in Eq. (2) do not statistically significant at 95% significance level, removal of them did not change the R^2 significantly and a simpler model containing less parameter was obtained for estimating the residual turbidity. The other model terms in Eq. 3 were obtained after removing the insignificant terms (Prob >F more than 0.1) in Eq. 2 for the model improvement by applying backward elimination procedure.. Final second order equation in terms of coded factors for the supernatant turbidity estimation was obtained as:

$$\ln(\text{Turbidity}) = 1.68 - 0.37A - 0.41B - 0.42C - 0.23D + 0.012E - 0.75AD + 1.15A^2 + 0.25B^2 + 0.23C^2 + 0.47D^2 + 0.29E^2 \quad (3)$$

According to the interaction effect analysis, flocculant dosage and solid ratio (AD) on supernatant turbidity were found statistically significant compared to other interactions between variables. The determination coefficient, R^2 , hence correlation coefficient, R , were calculated to be 0.82 and 0.9055 respectively.

4. CONCLUSIONS

Flocculation is defined as a second order rate process. This study confirmed the quadratic effects of investigated parameters on the residual turbidity of quartz suspensions resulted from jar test of flocculation process and showed that the mixing conditions (rapid mixing rate and time) and flocculant dosage had a great influence on the flocculation process. The second order equation obtained from the BBD method can be used to determine the optimum flocculation conditions of quartz suspensions within the investigated ranges of variables.

The second order effect of flocculant dosage (A^2) was found to be the most significant factor to have the largest effect on supernatant turbidity and this was followed by the linear effect of rapid mixing rate (C), linear effect of rapid mixing time (B), the two level interactions between flocculant dosage and solid ratio (AD), the linear effect of flocculant dosage (A), the second order effect of solid ratio (D^2). Moreover the main effect of solid ratio (D), the second order effect of settling time (E^2), the second order of rapid mixing time (B^2) and the second order of rapid mixing time (C^2) were determined to affect the residual turbidity of suspensions. It should be realized that this order of significance is valid strictly within the range of parameter values tested in this study.

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