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Optimizing the Settling Parameters of the Final Tailing of Zonouz Kaolin Processing Plant using RSM

Seyyed Mohsen Zamzami, and Javad Vazifeh Mehrabani*

Department of Mineral Processing, Faculty of Mining Engineering, Sahand University of Technology, Sahand New Town, Tabriz, Iran

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Abstract

In this research, solid phase settling process from the liquid phase were optimized simultaneously on the different responses, using the response surface methodology (RSM). The effect of solid percentage, flocculant dosage, temperature, and pulp pH were evaluated on the responses of solid settling velocity, water turbidity, viscosity and density of settled pulp. The results showed that by increasing the flocculant dosage from 0.5 to 3.5 g/ton, settled pulp viscosity decreases from 49.05 cSt to 17.54 cSt. The higher values of pulp pH as well as low amount of solid percentage resulted in high water turbidity, which shows the lack of contact between flocs and suspended particles. The results indicated that the pulp solid percentage and the flocculants dosage are the most significant parameters on the responses. Optimum test conditions were obtained in industrial mode by using 5 g/t flocculant, solid percentage 23.96%, pH=7.5 temperature of the pulp 21.5°C in which condition, settling rate, pulp viscosity, pulp density and water turbidity were predicted to be 13.23 cm/min, 5.1 cSt, 1.61 g/cm3 and 15.7 NTU respectively. Repetition test in the model predicted optimum condition was carried out and verified the predicted optimized condition.

1. Introduction

In most mineral separation processes, a significant amount of water is used, and the final concentrate must be separated from the pulp, where the ratio of solids to water may be high [1]. Most of the processes of separation and beneficiation of mineral materials (flotation, magnetic methods, leaching) are carried out in water environments [2]. The proper separation of water from the solid phase and its return from the tailings of the processing plant has a significant effect on water consumption and the economic efficiency of the process, as well as from an environmental point of view [3]. Separation of water from tailing is an essential step in the processing of minerals, which not only recycles the water but also reduces the risk of environmental pollution [4]. In the conditions where the density difference between solid and liquid is high, the settling method in tanks such as washer, clarifier and thickener have the best efficiency [5,6]. In the sedimentation process, various parameters can be investigated and studied. The effect of particle properties, size distribution, specific surface area, density, pulp temperature, surface structure, substrate porosity, and particle sphericity, as well as pulp properties such as solid concentration, pH, zeta potential, and porosity of flocs on sedimentation and dewatering performance in water environments are reviewed by researchers [7-11]. Studies have shown that lowering the temperature increases the viscosity of water, which increases the settling time, which is immediately reflected in the settling rate [12]. Clay particles in water can lead to hydration, selective absorption, and dissolution. These processes lead to a negative charge on the surface of the particles, the creation of an electric double layer and a hydration layer, which in turn creates an electrostatic repulsion effect and potential

Corresponding author: mehrabani@sut.ac.ir (J. Vazifeh Mehrabani)

resistance and prevents the process of sedimentation and filtration in wastewater [13,14]. The interfacial characteristics of clay minerals in aqueous suspension are complex, affected by water, pH and ion composition of the suspension. Hydration or swelling characteristic of clay-water, variable charges on the surface of clay particles in different pH and complicated ion composition have higher requirements for flocculants [15]. Extensive studies indicate that flocculation can significantly affect the settling rate of flocs [16,17]. The results show that the sedimentation speed and the density of flocs are affected by the dosage of coagulant. The rate of flocs settling increases with the increase of the flocs dosage and leads to an increase in the downstream concentration. Meanwhile, increasing the flocculant dosage leads to the formation of looser flocculent structure with enhanced particle-particle interaction. Two opposing effects caused differences in downstream concentration change as a function of flocculant dosage for unsorted and fine tailings [18]. Alex et al. concluded that in the high consumption flocculant dosage, the size of particles and bridging of particles increases, which causes the production of strong particles and the fragility of the floc decreases [19]. In a study, Oroj et al. reported that the high molecular weight flocculants form bridges between particles through hydrogen or chemical bonds [7]. In terms of polymeric flocculants, polyethylene oxides outperform polyacrylamide by lowering the turbidity of the suspension after flocculation sedimentation, because of the stronger affinity to quartz by the hydrogen bond between the ether oxygen and silanol group [20]. Parsafer et al. observed that the size, shape, and density of the flocs change with the increase in the flocculant dosage. The size of the flocs increases with the amount of flocculant, and on the contrary, their density decreases [21]. Besra observed that the settling velocity for low solid percentages is higher than pulp with high solid percentages. In pulps with high density, flocs are formed in the form of a network, which relatively reduces the sedimentation rate [22,23]. Recently statistical optimization and modelling techniques including RMS are used for modelling process parameters in mineral processing [24-28]. A study was carried out by Zamanikherad et al. regarding the efficient design of primary sedimentation tanks using a response surface [29]. Ebru Taş et al. investigated the application of biopolymer in the turbidity removal and sedimentation behavior of travertine processing wastewater sludge using RSM [30].

Zonouz kaolin processing plant is located in Iran - East Azarbaijan province, north of Marand city, which is the largest kaolin mine in the Middle East. The final tailings of Zonouz kaolin processing plant are directly discarded to the tailings dam. These tailings generally consist of considerable amount of water and solid phases of quartz, kaolinite, calcite, and iron oxides. Investigating the sedimentation behavior of the tailing can be important in the dewatering thickener. In the previous research work by the authors, the settling conditions of waste pulp solid particles were evaluated [31]. In this research, a simultaneous optimization technique was used for optimization of multiple responses of solid settling in the tailing pulp, in which the best sedimentation conditions are achieved along with the highest transparency of the water in the clear part. Since the quality of water returned to the processing circuit is important, it is very important to check the factors simultaneously in order to achieve a successful sedimentation process.

2. Materials and methods

2.1. Sampling and phasing of the tailings sample

A representative tailing sample was collected from the tailing stream of Zonouz kaolin plant (Figure 1). The sample was collected from the output of the final tailings transfer pumps to the tailings dam. It was collected using a 15-liter bucket in 2 consecutive days and during an 8-hour shift every two hours, which was used after dewatering. The weight of collected samples from the plant feed was more than 50 kg.

The results of particle size analysis showed that the D_{80} particle size is around 70 microns. In addition, particle size analysis indicated that 46% of the particles are smaller than 38 microns, 21% are smaller than 10 microns, and 9% of the particles are smaller than 5 microns. XRD analysis showed that quartz and kaolinite are the main phases in the tailing sample. Calcite and iron oxide mineral were detected as minor phases [31]. The chemical analysis of the waste sample showed that the tailing consists of 82.45% SiO_2, 11.26% Al_2O_3, 0.35% Fe_2O_3 and 0.23% CaO. Also, the percentage of volatile substances was measured at 5.71%. The image analysis of kaolinite showed that the structure of kaolinite is lamellar, and the zeta potential measurement using a zeta-meter showed that the isoelectric point of the tailings sample is 2.4 [31].

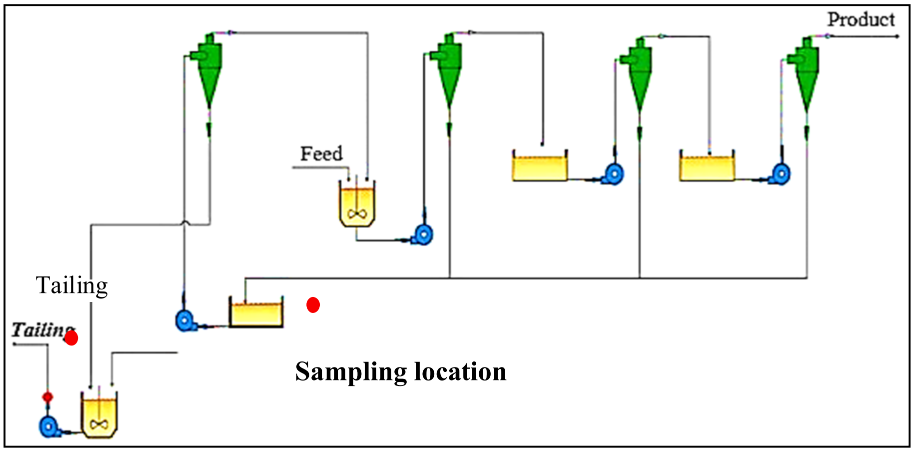

Figure 1. Sampling location of the tailings of the kaolin processing circuit [31].

2.2. Reagents

Sulfuric acid and sodium hydroxide (Merck) were used to adjust the pH of the pulp, an anionic flocculant type from Besflak Company with a molecular weight of 8 to 24 million was applied for sedimentation experiments.

2.3. Equipment

2.3.1. Drop viscometer

In order to measure the viscosity, the pulp is poured into a cylindrical tank with a conical end. The pulp comes out from the hole at the end of the cone, which is 4 mm in diameter, and the duration of pulp discharge from the tank is measured, recorded in seconds and converted into centistokes [31].

2.3.2. Turbidity meter

A turbidity meter is used to determine the amount of water turbidity. Turbidity unit (NTU) is a relative measurement. The device used in this study is Turb 550 WTW made in Germany. In this device, however, the clearer the water is, its turbidity number is close to zero. The turbidity value of distilled water measured with this device is 0.1 NTU. In this study, water transparency is also proposed as a response, and water transparency is based on the fact that it must have a desirable quality.

2.4. Calculation of settling rate

To calculate the velocity at different settling times, the height of the mud line was recorded, and the mud line graph was drawn at different times. At a point in the Graph where the settling speed was out of linear mode, it was selected as the desired point to calculate the speed. The initial speed was obtained by dividing the height difference (A in Figure 2) by the time (B in Figure 2) it took the mud line to reach the desired point which is shown in Figure 2 [31].

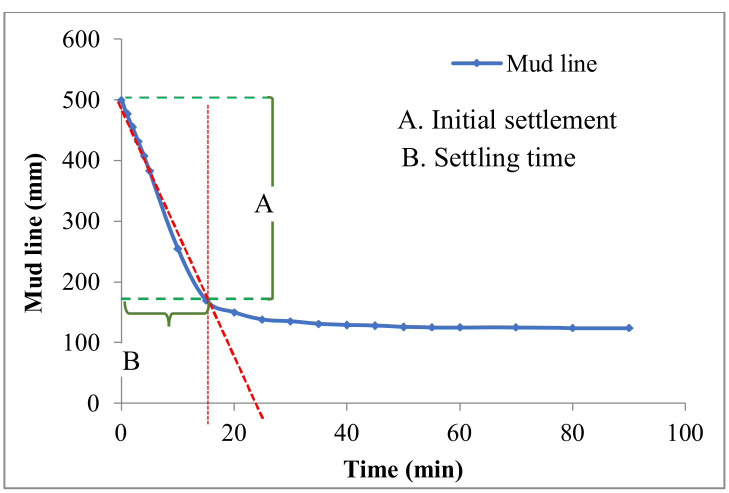

Figure 2. Calculation diagram of the settling speed of pulp solid particles [31].

2.5. Sedimentation tests and work methods

Knowing the effective parameters in the sedimentation process will be a critical step to optimize the sedimentation conditions. In order to identify the mutual effects between the factors and find the optimal state of the experiments using Modde software, the experiments have been carried out in a designed way. As initial parameters had been determined, only the effective parameters that were effective on the sedimentation speed, water turbidity, pulp density, and viscosity in the sedimentation process were used in the supplementary experiments [31]. The design used in this study was the CCC (Circumscribed Central Composite) method.

2.6. Central composite design

Response surface methodology (RSM) is a collection of statistical and mathematical techniques useful for the modelling and analysis of problems in which a response of interest is influenced by several variables and the objective is to optimize this response [32]. In most RSM problems, the form of the relationship between dependent and the set of independent variables is unknown. Thus, the first step in RSM is to find a suitable approximation for a functional relationship between dependent and independent variables. Usually, a low order polynomial in some region of the independent variables is employed. If there is a curvature in the system, then a polynomial of higher degree is used. Second-order models are widely used in RMS as they have several advantages. They are very flexible and can take on a wide variety of functional forms so they will work well as an approximation to the true response surface. Moreover, it is easy to estimate the parameters in a second-order model using the method of least squares. Circumscribed Central Composite (CCC) is used extensively in building the second-order response surface models. It is one of the most important experimental designs used in the process optimization studies. Central composition designs, like Box Bunker and Doehlert, are among the main level response methods used in experimental design. The most popular RMS is the central composite design [32-35]. Using the design of experiments based on RSM, optimal settling conditions with satisfactory performance can be obtained with a minimum number of experiments without the need for experimental study of all possible combinations. Furthermore, the input levels of the different variables for a particular level of response can also be determined. In order to determine a critical point (maximum, minimum, or saddle), it is necessary for the polynomial function to contain quadratic terms according to the following equation:

$$Y = \beta_0 + \sum_{i=1}^{k} \beta_i x_i \sum_{<i<j}^{k} \beta_{ii} x_i^{\,2} + \sum_{1<i<j}^{k} \beta_{ij} x_i x_j + \varepsilon \qquad (1)$$

where k, β_0, β_i, x_i, β_{ii}, β_{ij} and ε represent number of variables, constant term, coefficients of the linear parameters, variables, coefficients of the quadratic parameter, coefficients of the interaction parameters and residual associated to the experiments, respectively [35]. The selected control factors, including pH, solid percent flocculant dosage and temperature and their levels are shown in Table 1.

Table 1. The level of variables in the CCC

	Factor name			
	Flocculant dosage (g/t)	(%)Solid percentage	pH	Temperature (C°)
Code	D	C	B	A
High level	5	25	9	30
Low level	2	10	4	15
Central range	3.5	17.5	6.5	22.5
- α	0.5	2.5	1.5	7.5
+α	6.5	32.5	11.5	37.5

3. Results and discussion

3.1. Construction of model equations

Thirty settling experiments were designed using central composite design methodology. The experimental conditions and their responses are shown in the Table 2. The results entered in "Modde" software and appropriate models were selected from several models and fitted with the results. Four models were fitted for settling rate, water turbidity, density and viscosity of settled paste. Linear and quadratic models found to be adequate for the prediction of the response variables are given by the following equations:

$$\text{Pulp viscosity} = 11.93 - 0.22B - 0.14C - 0.1D + 0.5CD + 0.92D^2 \quad (2)$$

$$\text{Water turbidity} = 1.69 - 0.04A - 0.1B - 0.19C - 0.12D - 0.25BC - 0.14A^2 \quad (3)$$

$$\text{Pulp density} = 1.61 + 0.003A - 0.008B + 0.02C - 0.004D + 0.01BC - 0.01BD - 0.02A^2 - 0.01B^2 \quad (4)$$

$$\text{Settling Rate} = 7.55 + 0.3A + 1.1B - 3.2C - 0.1D \quad (5)$$

In these models all variables are in coded values and A is Pulp temperature, B is pH, C is solid percentage, D is flocculant dosage and BC, BD, CD are interaction of main parameters.

Table 2. Test conditions table for CCC design

Test number	Temperature (C°)	pH	Solid percentage (%)	Flocculant dosage (g/ton)	Viscosity (cSt)	Water turbidity (NTU)	Density (g/cm^3)	Settling rate (cm/min)
N1	15	4	10	2	39.92	75	1.572	9.252
N2	30	4	10	2	37.89	67	1.538	10.28
N3	15	9	10	2	33.08	135	1.53	15.81
N4	30	9	10	2	14.2	114	1.539	13.7
N5	15	4	25	2	33.77	83	1.571	1.101
N6	30	4	25	2	25.17	110	1.606	2.827
N7	15	9	25	2	14.91	57	1.627	5.654
N8	30	9	25	2	16.43	15	1.604	4.112
N9	15	4	10	5	25.17	26	1.607	10.023
N10	30	4	10	5	23.68	15	1.576	10.28
N11	15	9	10	5	22.93	88	1.515	10.28
N12	30	9	10	5	21.41	80	1.465	16.21
N13	15	4	25	5	29.46	85	1.606	2.57
N14	30	4	25	5	29.97	15	1.637	4.12
N15	15	9	25	5	25.09	4.2	1.57	3.94
N16	30	9	25	5	19.88	3	1.601	5.84
N17	7.5	6.5	17.5	3.5	16	4.6	1.48	7.71
N18	37.5	6.5	17.5	3.5	16.74	9.8	1.521	7.34
N19	22.5	1.5	17.5	3.5	8.77	145	1.563	4.626
N20	22.5	11.5	17.5	3.5	14.26	28	1.561	7.71
N21	22.5	6.5	2.5	3.5	11.88	117	1.585	12.33
N22	22.5	6.5	32.5	3.5	22.93	15	1.631	2.313
N23	22.5	6.5	17.5	0.5	42.9	31	1.64	7.196
N24	22.5	6.5	17.5	6.5	49.11	85	1.58	5.825
N25	22.5	6.5	17.5	3.5	11.85	45	1.61	7.99
N26	22.5	6.5	17.5	3.5	12.22	42	1.622	7.19
N27	22.5	6.5	17.5	3.5	12.37	34	1.624	7.22
N28	22.5	6.5	17.5	3.5	11.1	31	1.592	7.71
N29	22.5	6.5	17.5	3.5	12.8	19	1.628	7.71
N30	22.5	6.5	17.5	3.5	12.1	35	1.604	7.57

The results of Table 3 show that the p-value for the regression model of all four responses is less than 0.05, which indicates that the model is satisfactory at the engineering confidence level of 95%. It should be noted that temperature (A) is not significant factor in the selected range for most responses, however, its interaction or (A^2) were significant. The accuracy and variability of the models could be evaluated by the coefficient of determination R^2. The R^2 value is always between 0 and 1.

The closer the R^2 value is to 1, the better the model predicts the response. By examining R^2 in Table 3, it can be seen that the answers contributed well to the regression model, in other words, the raw data is compatible with the regression model. The results of Q^2 show that the model has a higher degree of prediction. The value of the adjusted determination coefficient adj. R^2 suggested that there are excellent correlations between the independent variables.

Table 3. Analysis of variance table

Source	Sum of Squares	df	Mean Square	F-Value	p-value
Pulp Viscosity					
Model	44.869	14	3.205	3.163	0.017
B	2.857	1	2.857	2.819	0.114
C	1.260	1	1.260	1.244	0.282
D	1.144	1	1.144	1.129	0.305
CD	3.276	1	3.276	3.233	0.092
D^2	29.941	1	29.941	29.546	< 0.0001
Residual	15.201	15	1.0134		
Pure Error	1.635	5	0.327		
Cor Total	60.0698	29			
R^2=0.75	**Q^2 =0.609**	**R^2 Adj. =0.698**		**Cond. no. =2.401**	
Water Turbidity					
Model	4.66751	14	0.333	3.26238	0.015
A	0.051	1	0.051	0.496	0.492
B	0.308	1	0.308	3.015	0.103
C	1.047	1	1.047	10.245	0.006
D	0.443	1	0.443	4.334	0.055
BC	1.426	1	1.426	13.952	0.002
A^2	0.676	1	0.676	6.614	0.021
Residual	1.533	15	0.102		
Pure Error	0.088	5	0.018		
Cor Total	6.200	29			
R^2 =0.68	**Q^2 =0.466**	**R^2 Adj. =0.591**		**Cond. no. =2.401**	
Pulp Density					
Model	0.004	14	0.000254	4.649	0.003
A	5.14E-05	1	5.14E-05	0.941	0.347
B	0.0004	1	0.0004	6.811	0.020
C	0.0006	1	0.0006	10.144	0.006
D	0.0002	1	0.0002	2.897	0.109
BC	2.77E-05	1	2.77E-05	0.506	0.048
BD	0.0007	1	0.0007	12.689	0.003
A^2	0.0013	1	0.0013	23.452	0.0002
B^2	0.0004	1	0.0004	6.509	0.0221
Residual	0.0008	15	5.47E-05		
Pure Error	6.96E-05	5	1.39E-05		
Cor Total	0.0044	29			
R^2 =0.839	**Q^2 =0.61**	**R^2 Adj. =0.778**		**Cond. no. =3.392**	

3.2. Investigating the main parameters with the greatest effect on the process

Figure 4 (A) shows that the water turbidity decreases with the increase in the consumption of flocculant. High flocculant dosage causes more particles to be attached to the flocculant chains. This result is accordance with another research work [36,37]. Based on the results, it was detected that the flocculant dosage has the greatest effect on the viscosity response. According to the diagram in Figure 4 (B), it can be seen that with the increase of the Flocculant dosage from 0.5 to 3.5 g/ton in the central range of temperature and pH=6.5, the viscosity of the pulp decreases from 49.05 cSt to

17.54 cSt and again by increasing the flocculant dosage up to 6.5 g/ton, the viscosity of the settled pulp increases up to 45.84 cSt. In the central and constant values of the pulp solid percentage, when the flocculant dosage is low, few particles would be absorbed and settled by the flocculant, and the majority of the particles probably settle without flocculant adsorption. Therefore, free flocculant particles could be placed in the empty space of the flocs (Figure 5 (A)). In this condition, the density of particles and flocs could be increased and causes an increase in the density of the settled pulp, which leads to an increase in viscosity. The decrease in viscosity when the flocculant dosage is in the central range indicates that the particles could be completely absorbed by the flocculant chains.

The formation of flocs and the placing of flocs together causes the empty space between the flocs to be occupied by water, which in addition to reducing the density of the settled pulp, also reduces the viscosity of the pulp (Figure 5 (B)).

According to Figure 5 (C), it can be interpreted that increase in viscosity with increasing flocculant dosage means the absorption space of the flocculant chain is not completely filled by the particles and the connection between the flocs by the flocculant chain prevents the flowing of the settled pulp, which indicates floc adhesion to flocculant chains [38,39].

Excess flocculant can re-charge and stabilize the particles, thereby increasing the viscosity and dispersion of the suspension [40]. The placement of flocs and the connection between flocs can also be checked from the density of the settled pulp.

The density of settled pulp in tests N23, N24 and N25 shows that increasing the flocculant dosage leads to a decrease in density, because the way the flocs are placed and the presence of pore water between the flocs reduces the density of the settled pulp.

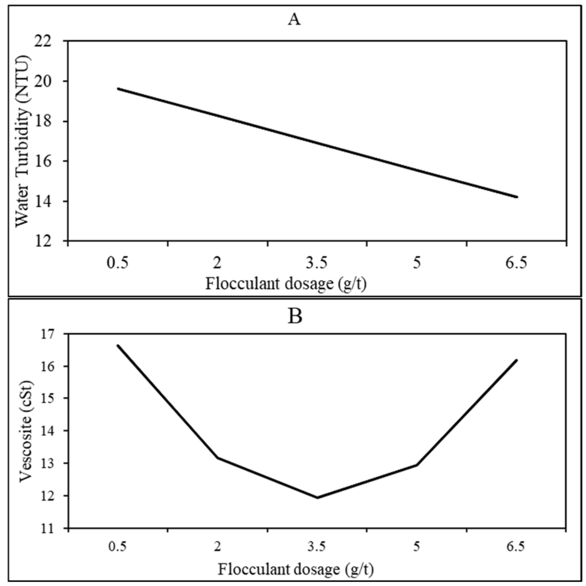

Figure 4. (A) Effect of Flocculant dosage on water turbidity (B) Effect of Flocculant dosage on pulp viscosity, (other factors in the central range).

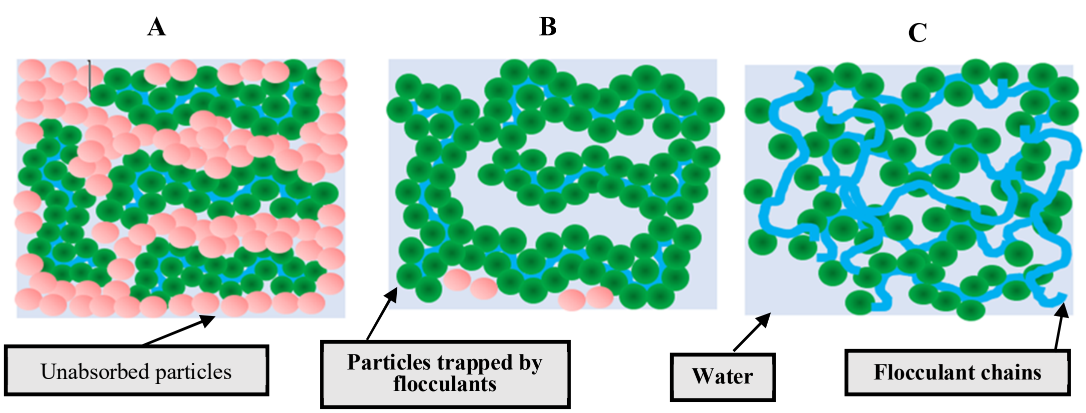

Figure 5. Placement of flocs in different dosage of flocculant. (A) low dose, (B) medium dose, (C) high dose.

It can be seen from Figure 6 (A) that the solid percentage significantly effects on the particle settling speed. That is, with the increase of the solid percentage, the sedimentation rate of the particles decreases, the main reason of which is the collision of the flocs during the sedimentation. In the low pulp solid percentage, free fall conditions prevail in the sedimentation process, which increases the sedimentation rate among the particles. In the pulp with a high solid percentage, the falling condition has turned into a barrier-falling state, which can result in a decrease in the settling speed of the particles.

The hindered-settling velocity increases when the particle size increases due to aggregation, and the suspension permeability increases because of channels formed in the settling sediment [41,42]. According to Figure 6 (B), the pulp solid percentage has considerable effect of water turbidity. The high percentage of solids makes the separated water clear, because in a situation the fine particles are tramped between the coarse particles and flocs. The shape of the cylinders in the values of low solid percentage (right) and high solid percentage (left) is shown in Figure 7. By evaluating the diagram in Figure 6 (C), the increase in solid percentage leads to an increase in pulp density after sedimentation. Undoubtedly, the effect of the weight of the particles and the compression of the pulp in the settled part causes a significant increase in the density after the pulp settles [41].

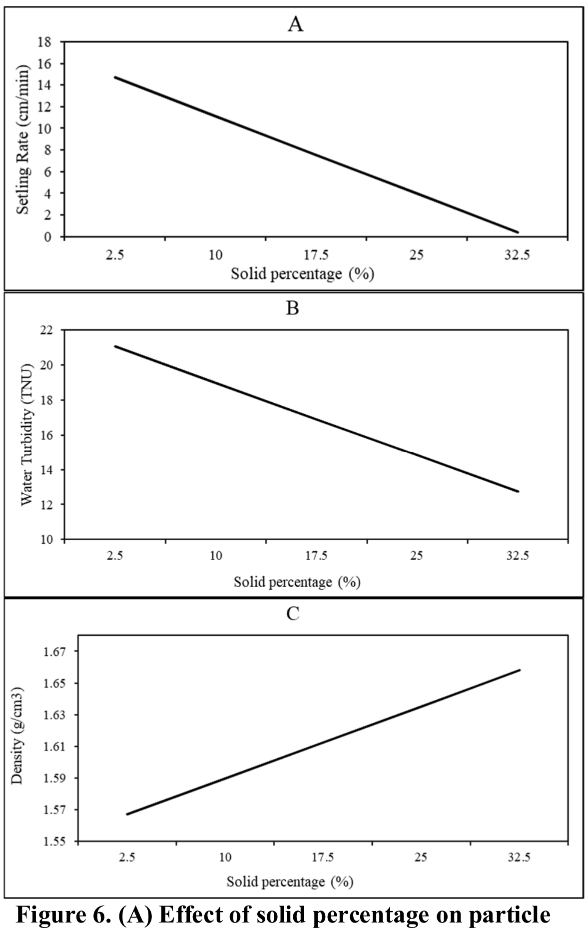

Figure 6. (A) Effect of solid percentage on particle settling speed, (B) The effect of pulp solid percentage on water turbidity, (C) Effect of pulp solid percentage on settled pulp density. (In the central value of other factors).

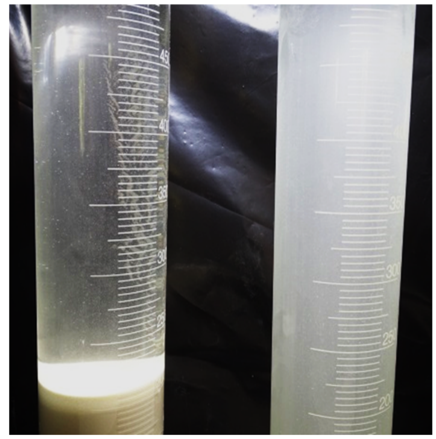

Figure 7. The effect of solid percentage values on water turbidity, Low solid percentage (right), High solids percentage (left).

3.3. Effect of interactions

The results showed that at the low levels of flocculant dosage (2 g/t) an increase in solid percentage causes a significant decrease in the viscosity of the settled pulp from about 14.6 cSt to 11.6 cSt (Figure 8). In contrast, in high dosage of flocculant (5 g/t), an increase in the solid percentage of the settled pulp causes an increase in the viscosity of the pulp, which shows significant interaction between flocculant dosage and pulp solid percent parameters. The viscosity behavior of the pulp in high dosage of flocculant shows that in the low amounts of solid percentage, due to the low percentage of solid particles, the formed flocs do not exert enough weight force to compress the flocs and the pulp is less dense and a significant amount of water remains between the flocs, which reduces the viscosity. With the increase of the solid percentage, the rate of formation and the number of formed flocs with high weight increases, which leads to the compaction of the pulp in the settled part, and on the other hand, the majority of solid particles that are placed between the flocs during settling cause an increase pulp density associated with an increase in pulp viscosity. By increasing the solid percentage in a low flocculant dose, the viscosity decreases. The low dosage of flocculant causes the number of flocs formed in different values of solid percentage to be different. In low amounts of solid percentage, the surface of the floc chain may be completely surrounded by particles, that is, all the particles have been converted into flocs by the flocculant, which causes the flocs to connect to each other and increase the viscosity.

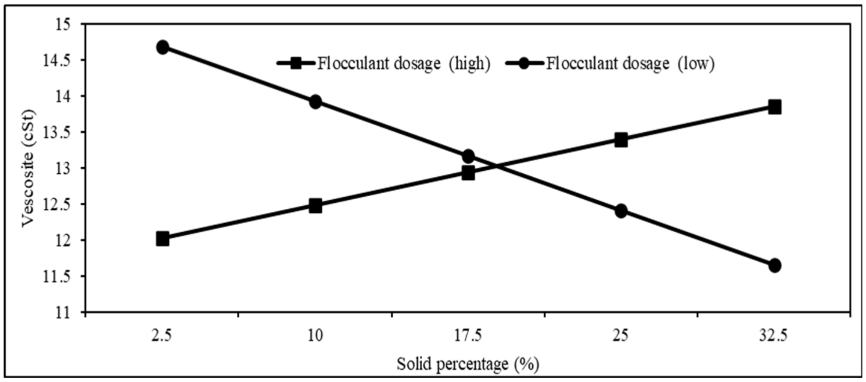

Figure 8. The effect of solid percentage on pulp viscosity of the settled part at high and low levels of Flocculant dosage (central range of other parameters).

If the amount of solid percentage increases, a small part of the solid particles will be absorbed and settled by the floc, and the majority of the particles will be settled individually. Due to the fact that the dimensions of the coarse flocs are larger than the solid particles, they settle at a faster rate and are placed in the lower part of the container, and due to the irregular shape of the flocs, part of the water remains between the flocs. The lack of connection of the major part of solid particles and the presence of pore water between the flocs causes the continuity between the flocs and particles not to occur and causes the viscosity to decrease.

Floc properties depend on solid, liquid, mixed, and flocculant properties [43]. Figure 9 indicates water turbidity at different pH values and solid percentage. Figure 9 indicates water turbidity at different pH values and solid percentage. The performance of anionic flocculants is greatly weakened in the acidic range, so that in small amounts of flocculants, the sedimentation process is carried out without flocculants. In the case where flocculant is not used, due to the positive charge of the edges and the negative charge of the kaolinite surface, the edge-surface connection structures are dominant and cause the coagulation of fine particles suspended in water and increase the transparency of the water [38]. These charges are created due to the isomorphous substitution of Si and Al atoms by transition metal ions as impurities in the mineral crystal structure [44].

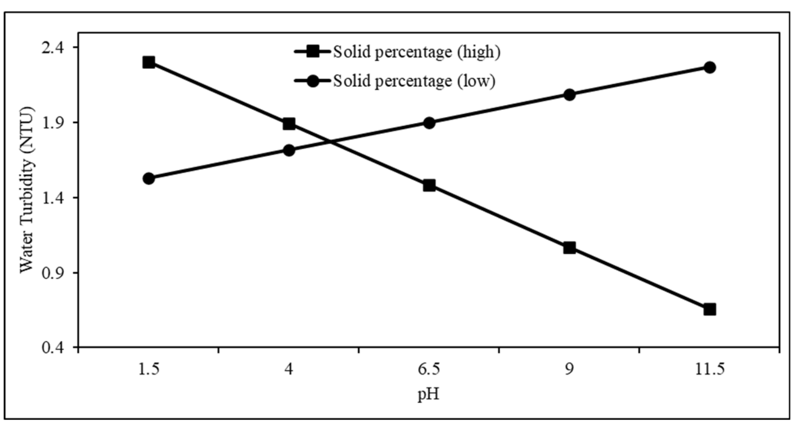

Figure 9. Effect of pulp pH on water turbidity at high and low levels of solid weight percentage (central range of other parameters).

Due to the fact that in low pulp solid percentage values, the number of suspended particles is less compared to high pulp solid percentage values, it reduces water turbidity. According to Figure 10 the flocculant does not play a main role in the settling of suspended particles in acidic pH and the low water turbidity in low amounts of pulp solid percentage is due to the few numbers of suspended particles.

In the alkaline pH range, the potential of colloidal particles to remain suspended is higher due to the electric direction of the particle surface. In the pulp with a high solid percentage, due to the higher number of formed flocs compared to the pulp with a low solid percentage, the probability of suspended particles hitting the flocs is higher.

If the turbidity of the water is checked in the alkaline range of pH in different values of the solid percentage, the turbidity of the water has decreased in the high values of the pulp solid percentage in order to collect the suspended particles by the flocs, but when the solid percentage is lower, the probability of the floc hitting the suspended particles is reduced.

Figure 11 indicates interaction between pH, flocculant dosage on settle pulp density. Increasing flocculant dosage in the acidic pH rose pulp density, while it decreased in the alkaline pH. As discussed previously, in the enough amounts of flocculant, there is a phenomenon of bridging between the flocs, and the empty space between the flocs is filled by water which leads to a decrease in the density of the settled pulp. This trend observed in the alkaline pH.

On the other hand, in the acidic pH range, flocculant performance is weakened in small amounts. Sedimentation is the same as in conditions without flocculant in acidic pH with low amounts of flocculant, whereas the performance of flocculant is affected, due to the high amount of flocculant, part of the particles by the flocculant is settled. Therefore, the amount of remaining flocculant is not so high that it may lead to the formation of a bridge between the flocs. As a result, the density of settled pulp has increased.

Figure 10. Effect of pulp pH on water turbidity when pulp solid percentage is high, Acidic pH (right), Alkaline pH (left).

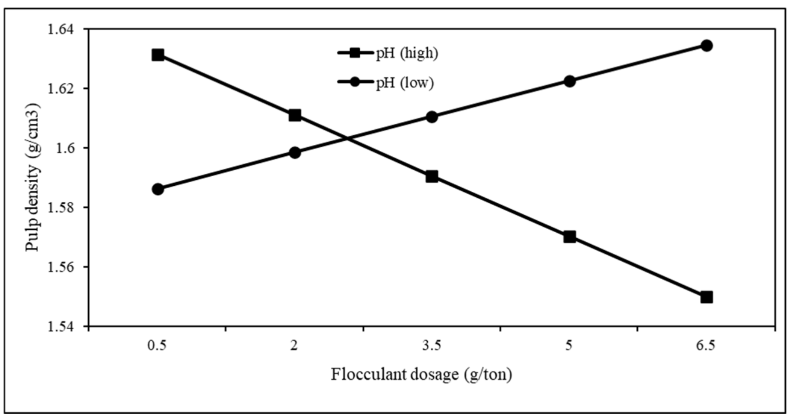

Figure 11. The effect of the Flocculant dosage on the density of settled pulp at high and low pH levels, (other factors in the central range).

3.4. Process optimization

The objective of response surface optimization is to find a desirable location in the design space. This could be a maximum, a minimum, or an area where the response is stable over a range of factors. In this research, a simultaneous optimization technique was used (by Modde software) for optimization of multiple responses.

The surfaces generated by linear and quadratic models can be used to indicate the direction in which the original design must be displaced in order to attain the optimal conditions. However, if the experimental region cannot be displaced due to physical or instrumental reasons, the research must find the best operational condition inside the studied experimental condition by visual inspection. Responses were defined as maximum and minimum and factors were evaluated in different modes.

Using the software, the optimum point of the process was determined, which is shown in out-of-range values column of the table 4. Since the design used in this study is of CCC type, the optimization mode was performed by considering the factors in the out-of-range range.

The software obtained the optimal conditions when the temperature is 36.9°C, pH 1.5, solid percentage 2.5%, and the flocculant dosage is 6.5 g/t. In these conditions, the sedimentation rate was 14.53 cm/min, water turbidity was 5.24 NTU, pulp density was 1.62 g/cm^3 and pulp viscosity was 12.17 cSt. Considering that the pH adjustment is always done using chemicals, the use of chemicals causes contamination of the water separated from the solid phase, and this process causes disturbances in kaolin processing.

As this research has been studied for an industrial situation, therefore, adjusting the pH and temperature will not be possible from an economical and process point of view. On the other hand, it will not be possible to adjust the solid percentage on an industrial scale without installing equipment and spending money. Different optimization conditions are shown in Table 4.

The consumption of minimum flocculant will be adjusted to the minimum amount if possible, considering that the return water will be used in the kaolin processing circuit.

Table 4. Optimization in the range of factors and response selected by Modde software.

Case	out-of-range values	within range	Operating range	Repeat test
Temperature (°C)	36.92	30	21.5	21
pH	1.5	4	7.5	7.5
Solid percentage (%)	2.5	10	23.96	24
Flocculant dosage (g/ton)	6.5	5	5	5
Viscosity (cSt) Maximize	12.17	5.87	5.1	8.51
Water turbidity (NTU) Minimize	5.24	5.76	15.7	14
Pulp density (g/cm^3) Maximize	1.62	1.58	1.61	1.61
Settling rate (cm/min) Minimize	14.53	13.11	13.23	12.54

To ensure the correctness of the optimization ranges, repetition tests were performed. It was done within the operational range predicted by the software. By repeating the experiment in the specified range, the results were more similar, so that the sedimentation rate was 12.54 cm/min, the water turbidity was 14 NTU, the pulp density was 1.61 g/cm and the pulp viscosity was 8.51 cSt.

Figure 12 shows the response surface for viscosity responses, settled pulp density, settling velocity and water turbidity. The behavior of each of the responses are shown in different amounts of solid percentage and flocculant dosage with the central values of pH and temperature.

As it is clear in the picture, the curvature of the surfaces shows the quadratic nature of the model.

5. Conclusions

Mineral analysis shows that the major part of the solid phase of silica is of the quartz type, which is important in the sedimentation and separation of particles. Results indicated that the pulp solid percentage has the largest negative coefficient in response to the initial settling rate, and then it has the largest negative coefficient in the water turbidity response.

In low pulp solid percentages, free fall conditions prevail in the sedimentation process, which increases the sedimentation rate. In the pulp with a high solid percentage, the falling condition has turned into a barrier-falling state, which can result in a decrease in the settling speed of the particles. On the other hand, with the increase in pulp solid percentage, the water in the remaining part becomes clearer, and the rate of sedimentation decrease. The high percentage of solids makes the separated water transparent, because in conditions where the solid percentage of the pulp is high the fine particles could be stuck between the coarse particles and it settles, which makes the water clear. An increase in solid percentage leads to an increase in pulp density. Undoubtedly, the effect of particle weight and pulp compression in the settled part causes a significant increase in pulp density.

Flocculant dosage has the greatest effect on the viscosity response. Increasing the flocculant dosage water clarity improved as the pH increases; when the solid percentage is at a low level, water turbidity increases. In low amounts of flocculant, due to the proper coverage of the surfaces and density of the flocs, it has increased the density. On the contrary, in acidic pH, it is clear that the low amount of flocculant caused the flocculation not to take place well, but with the increase in the flocculant dosage, the pulp density also increased. Optimization of the process showed that if the flocculant consumption is 5 g/t in the industrial operating parameters in terms of temperature, solid percentage, and pH, the sedimentation operation can be performed. In these defined conditions, by repeating the experiment, the results were more similar, so that pulp viscosity 12.54 cSt, water turbidity 14 NTU, pulp density 1.61 g/cm, and sedimentation speed 8.51 cm/min were obtained.

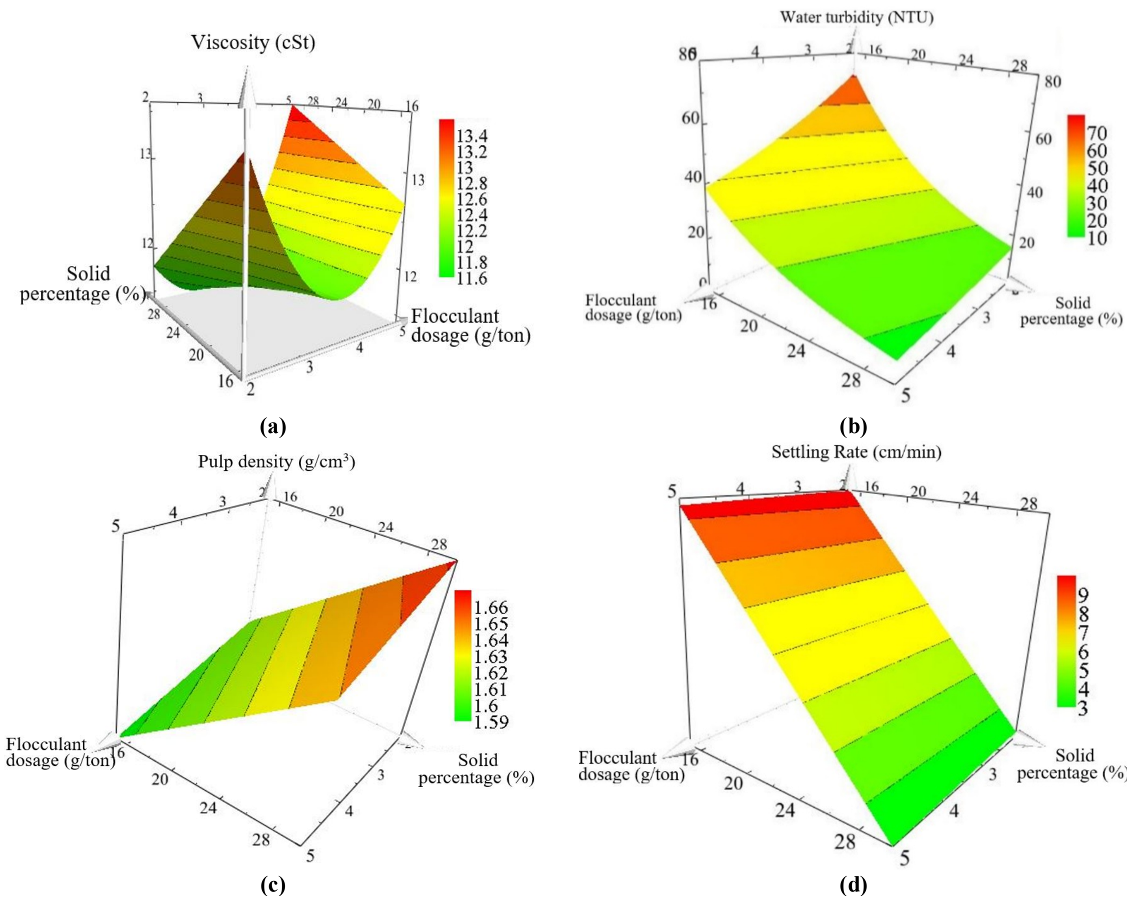

Figure 12. Response surface for viscosity (A), water turbidity (B), settled pulp density (C) and settling rate (D).

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بهینه سازی پارامترهای ته نشینی باطله نهایی کارخانه فرآوری کائولن زونوز با استفاده از RSM

سید محسن زمزمی و جواد وظیفه مهربانی*

گروه فرآوری مواد معدنی، دانشکده مهندسی معدن، دانشگاه صنعتی سهند، شهر جدید سهند، تبریز، ایران

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* نویسنده مسئول مکاتبات: mehrabani@sut.ac.ir

چکیده:

در این تحقیق، فرآیند ته نشینی فاز جامد از فاز مایع به طور همزمان بر روی پاسخ های مختلف با استفاده از روش سطح پاسخ (RSM) بهینه شد. اثر درصد جامد، دوز لخته‌ساز، دما و pH خمیر بر واکنش‌های سرعت ته‌نشینی جامد، کدورت آب، ویسکوزیته و چگالی خمیر ته نشین‌شده مورد ارزیابی قرار گرفت. نتایج نشان داد که با افزایش دوز فلوکولانت از ۰.۵ به ۳.۵ گرم در تن، ویسکوزیته پالپ ته نشین شده از ۴۹.۰۵ cSt به ۱۷.۵۴ cSt کاهش می‌یابد. مقادیر بالاتر pH خمیر کاغذ و همچنین مقدار کم درصد جامد منجر به کدورت آب می‌شود که نشان دهنده عدم تماس بین لخته‌ها و ذرات معلق است. نتایج نشان داد که درصد جامد پالپ و دوز لخته‌کننده‌ها مهم‌ترین پارامترها در پاسخ‌ها هستند. شرایط آزمایش بهینه در حالت صنعتی با استفاده از لخته‌ساز ۵ گرم بر تن، درصد جامد ۲۳/۹۶ درصد، pH=۷/۵ دمای خمیر ۲۱/۵ درجه سانتی‌گراد به دست آمد که در آن شرایط، سرعت ته‌نشینی، ویسکوزیته خمیر، دانسیته خمیر و کدورت آب ۱۳/۱۳،۲۵ و ۱۳/۱ و ۱۳/۱۳ سانتی‌متر بر سانتی‌متر بر دقیقه پیش‌بینی شد. به ترتیب ۱۵.۷ NTU. آزمون تکرار در شرایط بهینه پیش‌بینی‌شده مدل انجام شد و شرایط بهینه پیش‌بینی‌شده را تأیید کرد.

کلمات کلیدی: نرخ تسویه، دم کردن، بهینه سازی، RSM، لخته سازی.