

Research Article

Optimization of Flocculation and Sedimentation Parameters of Total Tailing Filling Material Based on Response Surface Method

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Using sulfide ore and oxide ore tailings of a tin mine in Yunnan as raw materials, 17 groups of flocculation settling tests were designed by Box-Behnken in Design-Expert software, and the settling velocity and underflow concentration of mixed tailings with sulfur/oxygen ratio of 5:5 were studied. The effects of volume concentration, flocculant unit consumption, and flocculant concentration on settling velocity and underflow concentration or mixed tailings with sulfur/oxygen ratio of 5:5. The best mixing parameters of mixed tailings are volume concentration of 17.66%, flocculant unit consumption of 39.589 g/t, and flocculant concentration of 0.195%. At this time, the settling speed is 2.231 mm/s and the underflow concentration is 65.289%. The flocculation settling velocity under this parameter condition was tested, and the experimental results were consistent with the predicted results of the model. This surface method can be used to design the flocculation and settlement test of mine tailings, which has certain guiding significance for the selection of parameters in actual production of mines.

1. Introduction

With the continuous development of social economy, people's demand for mineral resources is higher and higher. The deepening of resources and the large-scale stacking of solid waste have become the difficult problems facing the development of mining industry. Filling method is widely used as a clean and efficient mining method, which can solve the stacking of surface tailing waste [1–3].

The filling method needs to make tailings and other solid wastes into slurry and transport them to the goaf. The thickening and dehydration of tailings is the premise of the filling pulping process. The traditional tailing dewatering method is natural sedimentation, which can improve the sedimentation efficiency by increasing the sedimentation area. However, with the progress of beneficiation technology, the particle size of tailings becomes smaller and smaller, and the tailings become finer and finer. There are some problems in the use of natural sedimentation, such as serious fine particle overflow and slow sedimentation speed [4]. Adding flocculant to the tailing slurry can improve the settling velocity of tailings and reduce

the overflow concentration [5]. Therefore, many scholars have carried out a lot of research work on the process of flocculation sedimentation. By studying the settling velocity of muddy liquid surface, based on the settling time, unit consumption of flocculant, and settling velocity of muddy liquid surface, the action mechanism of unit consumption of flocculant in different settling stages was obtained [6]. The law of multifactor flocculation and sedimentation is studied, and the important factors affecting flocculation and sedimentation are selected by the Design-Expert software, and the parameters are optimized [7]. Through the static flocculation sedimentation test of total tailings, the solid flux model of total tailing flocculation sedimentation is established, and the influence law of different feeding speed on sedimentation in the process of dynamic flocculation sedimentation is explored [8]. The support vector machine (SVM) regression prediction model is established, and the model parameters are optimized by genetic algorithm to predict the settlement velocity [9]. A simple regression model of sedimentation rate was obtained by fitting the static flocculation sedimentation test according to the feed concentration and unit consumption of flocculant [10].

It can be seen from the above reference that a series of tests are needed to determine the parameters of flocculation and sedimentation. Summarize the rules from the experiment, and finally determine the addition range of parameters. The surface response method has some advantages in this process. The test scheme can be designed by determining the variation range of independent variables, and the overall test can be simplified, and at the same time, the optimization scheme within the variation range of independent variables can be obtained.

In this paper, aiming at the two kinds of tailings of sulfide ore and oxide ore of a tin mine, the indoor static flocculation sedimentation test of sulfide ore tailings, oxide ore tailings, and three mixed tailings with different sulfur oxygen ratio are carried out to study the influence of different particle size composition of tailings on the sedimentation rate. For the mixed tailings with sulfur oxygen ratio of 5:5, the response surface method is used to design the test, explore the influence law of various parameters on flocculation sedimentation effect, and optimize the feeding parameters.

2. Experimental Materials

2.1. Basic Physical Parameters of Total Tailings. There are mainly two kinds of tailings of a tin mine in Yunnan: oxidized ore tailings and sulfide ore tailings. Specific parameters are shown in Table 1.

As shown in Table 1, the apparent density of sulfide tailings is 3042 kg/m³. The apparent density of oxidized tailings is 2861 kg/m³. The density of sulfide tailings is higher. The loose density of sulfide tailings is 1365 kg/m³. The loose density of oxidized tailings is 1245 kg/m³. The stacking compactness of sulfide tailings is 0.449, and the porosity is 0.551. The stacking compactness of oxidized tailings is 0.435, and the porosity is 0.565. In terms of loose density, packing compactness, and porosity, they are relatively close.

From Table 2, showing the mineral composition analysis, it can be seen that the main components of the tailings are calcite and mica, accounting for 40–50% and 23–35%, respectively. The chemical properties of the tailings are relatively stable, and there is no chemical reaction with water.

2.2. Tailing Grading. The two kinds of tailings are obviously different in appearance, gradation, and basic physical parameters. The grading analysis of two kinds of tailings is carried out. The detailed results of tailings grading are shown in Figure 1.

It can be seen from the figure that the tailings of sulfide ore are mainly distributed below -500 mesh, accounting for 40.87%. The average particle size of sulfide tailings is $84 \,\mu\text{m}$. The median particle size was $40 \,\mu\text{m}$. The particle size of 60% tailings is $53 \,\mu\text{m}$. Oxidized tailings are mainly distributed below -500 mesh, accounting for 46.11%, and the median particle size is $31 \,\mu\text{m}$. 60% of oxidized ores is below $40 \,\mu\text{m}$. The average particle size and median particle size of oxidized ore tailings are smaller than sulfide ore tailings, which shows that in morphology, oxidized ore tailings are finer than sulfide ore tailings. Both kinds of tailings are distributed below -500 mesh. Generally speaking, the particle size of the two filling

TABLE 1: Basic physical parameters of sulfide ores.

Туре	Apparent density/ (kg/m ³)	Loose density/ (kg/m ³)	Packing compactness	Porosity
Sulfide ores	3042	1365	0.449	0.551
Oxidized ores	2861	1245	0.435	0.565

TABLE 2: Analysis results of mineral composition of tailings.

Mineral name	Mineral content of sulfide ores (%)	Mineral content of oxidized ores (%)
Calcite	40-50	35-40
Mica	25-35	25-40
Fluorite	5-10	5-10
Dolomite	5-10	5-15
Amphibole	1–5	5-10

tailings is too fine [11, 12]. If natural settlement is adopted, it will cause problems such as too slow speed, insufficient underflow concentration, and high overflow concentration. Therefore, it is necessary to adopt flocculation settlement method to improve the settlement effect of tailings and realize stable sand discharge of sand bin.

3. Flocculation Sedimentation Mechanism

The polymer flocculant aggregates the particles through "bridging action" to form flocculation groups [13, 14]. The action mechanism is that some active groups in the molecules are adsorbed on the surface of the particles, the rest extend into the solution to form unstable molecules, and the particles with vacancies on other surfaces contact these extended parts to form aggregates, so as to complete the "bridging" between the particles. If the flocculant is excessive and the polymer active group completely wraps the particles, there is no other vacancy on the particle surface for the extended active group to contact, and the bridging action will not be completed. Therefore, the addition amount of flocculant has a reasonable range, and the larger the flocculant is, the better the flocculation effect is. Different flocculant types will produce different effects. Polymer flocculants can be generally divided into anionic, cationic, and nonionic types. Because different types of flocculants have different molecular composition and flocculation structure, and their action forms and effects are significantly different in solution environments with different pH values and different charges, the polymer flocculant rt301 with polyacrylamide model is selected for flocculation sedimentation test in this test.

4. Flocculation Sedimentation Test of Mixed Tailings

4.1. Experimental Design. In order to study the interaction between flocculant and tailing slurry sedimentation velocity and underflow concentration, a tailing flocculation sedimentation test was designed. Considering that in the actual

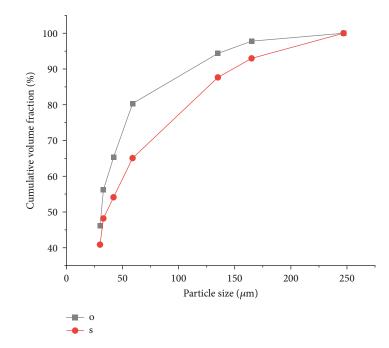


FIGURE 1: Grain size composition of full tailings.

production and application of the mine, sulfide tailings and oxidized tailings will be mixed and discharged in the proportion of 5:5 in most cases. The volume concentration range of the mixed slurry discharged by the concentrator is 15%-21%. Most research results believe that the main influencing factors of flocculation sedimentation are slurry volume concentration, flocculant unit consumption, and flocculant solution concentration. Therefore, based on the above conditions, the volume concentration of test slurry is designed to be 15%-21%, the unit consumption of flocculant is 20~60 g/T, and the flocculant concentration is 0.1~0.3%.

Response surface method [15, 16] can intuitively optimize the feeding parameters and predict and analyze the objectives. It can not only conduct single-factor analysis but also consider the impact of parameter interaction on the objectives. The volume concentration X_1 of tailing slurry, unit consumption X_2 of flocculant, and flocculant concentration X_3 were selected as independent variables, and the sedimentation velocity Y_1 and underflow concentration Y_2 were selected as response values. Box-Behnken in the Design-Expert software is used to design the test scheme of three factors and three levels and analyze the influence law of the interaction of various variables on sedimentation velocity and underflow concentration. The levels of various factors are shown in Table 3.

4.2. Test Result. In the settlement test, the settlement velocity of solid particles in the slurry is mainly determined by observing the position of the solid-liquid interface, as shown in the figure. The settling velocity and underflow concentration of solid particles are calculated by recording the height and time of solid-liquid interface.

Figure 2 shows the settlement process of tailings with the passage of time. With the passage of time, the height of settlement interface is getting lower and lower. The settlement

TABLE 3: Factors and levels of response surface methodology.

Independent variable	Horizontal coding			
	-1	0	1	
Volume concentration $(X_1)/(\%)$	15	18	21	
Unit consumption of flocculant $(X_2)/(g/t)$	20	40	60	
Flocculant solution concentration $(X_3)/(\%)$	0.1	0.2	0.3	

speed of tailings can be calculated by reading the height of settlement interface.

The specific test scheme and results are shown in Table 4. The multiple regression equations of response value, sedimentation velocity (Y_1) , and underflow concentration (Y_2) are obtained by multiple fitting of the test results with design expert:

$$\begin{split} Y_1 &= -0.63 + 0.35X_1 + 0.02X_2 + 1.94X_3 - 1.25 \\ &\times 10^{-4}X_1X_2 - 0.067X_1X_3 - 7.5 \times 10^{-3}X_2X_3 \\ &- 0.012X_1^2 - 2.19 \times 10^{-4}X_2^2 - 1.525X_3^2, \end{split} \tag{1}$$

$$\begin{split} Y_2 &= 7.197 + 4.07X_1 + 0.48X_2 + 47.42X_3 - 1.125 \\ &\times 10^{-3}X_1X_2 + 1.09X_1X_3 - 0.18X_2X_3 - 0.096X_1^2 \quad (2) \\ &- 5.34 \times 10^{-3}X_2^2 - 149.9X_3^2. \end{split}$$

During the experiment, the experimental concentration, flocculant unit consumption and flocculant concentration can be determined. However, the granularity of tailings in each experiment has a small amount of uncertainty. Therefore, under the same experimental conditions, there will be some differences in the experimental results, but these

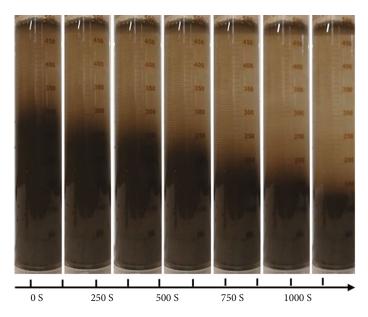


FIGURE 2: Schematic diagram of settlement test.

TABLE 4:	Response	surface	test	design	and	results.

Number	С	Code value			l value	Predicted value	
	X_1	X_2	X_3	Y_1	Y_2	Y_1	Y_2
1	0	0	0	2.21	65.76	2.20	65.58
2	-1	0	1	2.38	60.66	2.37	60.47
3	1	0	-1	1.81	65.12	1.82	65.31
4	0	0	0	2.18	65.38	2.20	65.58
5	-1	1	0	2.28	60.41	2.28	60.28
6	0	-1	1	2.11	62.12	2.11	62.26
7	0	-1	-1	2.12	61.88	2.12	61.56
8	0	0	0	2.19	65.81	2.20	65.58
9	1	-1	0	1.74	64.89	1.73	65.02
10	1	0	1	1.74	66.22	1.74	65.95
11	0	1	1	2.04	61.29	2.04	61.61
12	-1	0	-1	2.37	60.87	2.37	61.14
13	1	1	0	1.68	65.01	1.68	64.96
14	0	1	-1	2.11	62.49	2.11	62.35
15	0	0	0	2.22	65.75	2.20	65.58
16	0	0	0	2.19	65.19	2.20	65.58
17	-1	-1	0	2.31	60.02	2.31	60.07

differences are very small, and there will be no serious deviation from the correctness of the experimental results.

4.3. Reliability Test of Regression Model Based on Response Surface Method. The model reliability of multiple regression equations of sedimentation velocity (Y_1) and underflow concentration (Y_2) is tested. Figure 3 shows the comparison between the predicted values of the sedimentation velocity and underflow concentration model and the test values. It can be seen from Figure 3 that the test values are evenly distributed on the fitting line and both sides. It can be seen that the error between the two is small, which proves that the sedimentation velocity and underflow concentration model are reliable.

Goodness of fit (R^2) represents the difference between the measured value and the regression value, and the value is between 0 and 1. The closer R^2 is to 1, the higher the reliability of the model is. The R^2 values of sedimentation velocity and underflow concentration are 0.998 and 0.99, respectively, which are close to 1, which means that the model is very reliable. The *p* value of the model represents the significance of each factor, and the *p* value less than 0.05 represents the significance of the model. The *p* values of the regression models of sedimentation velocity and underflow concentration are less than 0.0001, and the residuals of the two regression models are not significant, which represent that the model is very significant.

5. Influence of Response Surface Parameters on Flocculation Sedimentation

5.1. Effect of Single Factor of Response Surface Parameters on Flocculation Settlement of Mixed Tailings. It can be seen from Figure 4 that the influence law of various factors on flocculation sedimentation is as follows:

- Keeping the unit consumption of flocculant and flocculant concentration unchanged, with the increase of volume concentration, the sedimentation rate gradually decreases and the impact is more intense. The underflow concentration first gradually increases and then tends to be flat, which indicates that the impact of the continuous increase of the initial feed volume concentration on the underflow concentration is decreasing
- (2) Keep the volume concentration and flocculant concentration unchanged. With the increase of

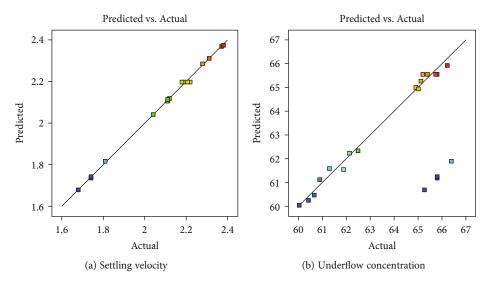


FIGURE 3: Comparison between experimental value and predicted value.

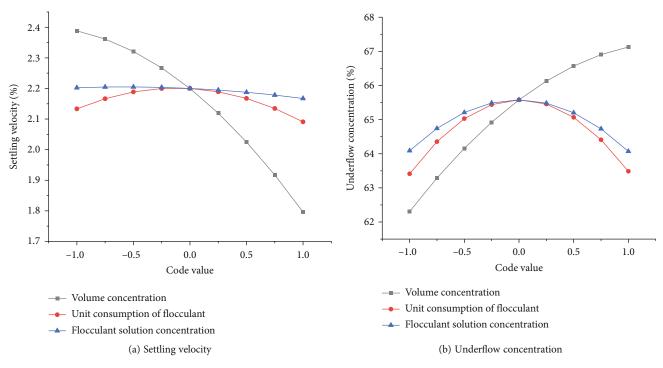


FIGURE 4: Single-factor analysis of response surface.

flocculant unit consumption, the sedimentation velocity and underflow concentration increase first and then decrease and reach the maximum in the range of 37-41 g/T. This shows that if the unit consumption of flocculant exceeds the optimal action range, the sedimentation effect will become worse. This is because the fine tailings adsorb a large number of flocculating molecules to produce too much floc water, which will worsen the driving effect of coarse particles on fine particles. The steric effect of a large number of polymer adsorption membranes makes the tailing particles repel each other; the siltation layer occupying the bottom space hinders the downward sedimentation of particles and worsens the compaction and dehydration process in the later stage, and too much flocculation structure reduces the underflow concentration

(3) Keep the volume concentration and unit consumption of flocculant unchanged. With the increase of flocculant concentration, the sedimentation velocity changes little, and the underflow concentration increases first and then decreases, reaching the maximum at about 0.2%.

Source	Sum of square	df	Mean square	F value	p value	
Model	0.7995	9	0.0888	495.5	< 0.0001	Significant
A-volume concentration	0.7021	1	0.7021	3916.17	< 0.0001	
B-unit consumption of flocculant	0.0036	1	0.0036	20.15	0.0028	
C-flocculant solution concentration	0.0025	1	0.0025	13.67	0.0077	
AB	0.0002	1	0.0002	21.25	0.2996	
AC	0.0016	1	0.0016	8.92	0.0203	
BC	0.0009	1	0.0009	5.02	0.06	
A^2	0.0489	1	0.0489	272.66	< 0.0001	
B^2	0.0324	1	0.0324	180.84	< 0.0001	
C^2	0.001	1	0.001	5.46	0.0521	
Residual	0.0013	7	0.0002			
Lack of fit	0.0002	3	0.0001	0.216	0.8807	Not significant

TABLE 5: Variance analysis of settlement velocity model.

TABLE 6: Analysis of variance of underflow concentration model.

Source	Sum of square	df	Mean square	F value	p value	
Model	82.37	9	9.15	78.59	< 0.0001	Significant
A-volume concentration	46.46	1	46.46	399.01	< 0.0001	
B-unit consumption of flocculant	0.0105	1	0.0105	0.0903	0.7726	
C-Flocculant solution concentration	0.0006	1	0.0006	0.0053	0.9442	
AB	0.0182	1	0.0182	15.33	0.7042	
AC	0.429	1	0.429	3.68	0.0964	
BC	0.5184	1	0.5184	4.45	0.0728	
A^2	3.12	1	3.12	26.84	0.0013	
B^2	19.17	1	19.17	164.66	< 0.0001	
C^2	9.46	1	9.46	81.25	< 0.0001	
Residual	0.8152	7	0.1165			
Lack of fit	0.5089	3	0.1696	2.22	0.2288	Not significant

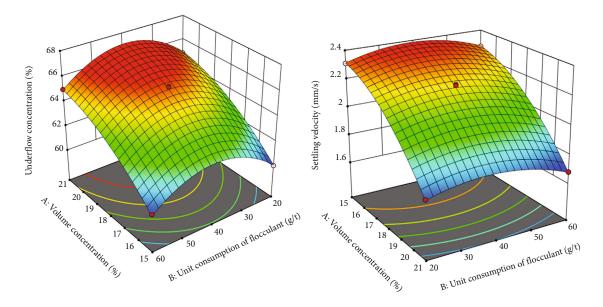


FIGURE 5: Interaction analysis of response surface factors.

5.2. Effect of Interaction of Response Surface Parameters on Flocculation and Sedimentation of Mixed Tailings. From the variance analysis of sedimentation rate and underflow concentration (as shown in Tables 5 and 6), it can be seen that the F value of volume concentration and flocculant unit consumption AB is the largest, which means that the interaction between them has the most significant impact on the flocculation and sedimentation of mixed tailings. Keep the flocculant concentration unchanged at 0.2%. Analyze the impact of volume concentration and flocculant unit consumption on sedimentation rate and underflow concentration, as shown in Figure 5. It can be seen from the figure that the sedimentation rate and underflow concentration are affected by the unit consumption of flocculant, and both increase first and then decrease. The best unit consumption of flocculant is in the range of 37-41 g/T. The increase of volume concentration makes the sedimentation velocity gradually smaller. The volume concentration increases from 15% to 18%, the velocity decrease range is about 7.9%, the volume concentration increases from 18% to 21%, and the velocity decrease range is about 18.36%, which indicates that the influence on the sedimentation velocity becomes more and more intense with the continuous increase of volume concentration. The underflow concentration increases with the increase of volume concentration, the volume concentration increases from 15% to 18%, the increase range of underflow concentration is about 5.2%, while the volume concentration increases from 18% to 21%, and the increase range of underflow concentration is about 1.8%, which indicates that the influence on underflow concentration decreases with the increase of volume concentration. With the increase of volume concentration, the influence of unit consumption of flocculant on sedimentation velocity and underflow concentration is decreasing, which proves that the influence of volume concentration on sedimentation effect is greater than that of unit consumption of flocculant.

5.3. Parameter Optimization. The Design-Expert software is used to optimize the results of flocculation sedimentation test. The underflow concentration and sedimentation velocity are the largest. The best parameters calculated by the model are feed volume concentration 17.66%, flocculant unit consumption 39.589 g/t, and flocculant concentration 0.195%. The sedimentation velocity is 2.231 mm/s, and underflow concentration is 65.289%. Taking the volume concentration of 17.7%, the unit consumption of flocculant 39 g/T, and the flocculant concentration of 0.2% as the actual test parameters, a flocculation and sedimentation test was designed.

The settling velocity is 2.247 mm/s, and the underflow concentration is 65.77%, which is basically consistent with the optimization results. The test results prove the correctness of the optimization model.

6. Conclusion

In this paper, the slurry flocculation sedimentation tests under different slurry volume concentration, flocculant unit consumption, and flocculant solution concentration are designed, and the test results are analyzed and studied by response surface method. The following conclusions are drawn:

- The effect of flocculation and sedimentation is not only affected by single factors, but also by the interaction between factors, especially the interaction of volume concentration and unit consumption of flocculant
- (2) Based on the research results of response surface method, the optimal mixing parameters of mixed ore tail mortar with sulfur oxygen ratio of 5:5 are predicted. The volume concentration is 17.66%, the unit consumption of flocculant is 39.589 g/t, the flocculant concentration is 0.195%, the settlement velocity is 2.231 mm/s, and the underflow concentration is 65.289%

Data Availability

The data that support the findings of this study are available from the corresponding author upon reasonable request.

Conflicts of Interest

The authors declared no potential conflicts of interest with respect to the research, authorship, and/or publication of this article.

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