Optimization of Flotation Process of Zinc Oxide Ore by Response Surface Methodology

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Abstract: According to the test results, the flotation process parameters of refractory zinc oxide ore will be optimized by the response surface methodology in this paper. The regression model between Zn recovery and sodium sulfide concentration, depressant(F1) concentration and sodium carbonate concentration was established. It can be known that the model is highly significant and test error is very small, and the concentration of sodium sulfide has great influence to the recovery of Zn concentrate. The verification tests results show that the model is reliable and can predict accurately the zinc recovery. It can be concluded that the optimizing process parameters are sodium sulfide 7500g/t, depressant concentration 1050g/t, and sodium carbonate concentration 2200g/t, which can provide the technical basis for the comprehensive utilization of low grade refractory zinc oxide ore.

1. Introduction

As the primary source of zinc, natural sulfide ores are steadily getting depleted and for future demand, there is an obvious recognition that the recovery of zinc from oxidized ores is an alternative. It is well known that the main processing methods are flotation, acid leaching and ammonia leaching for refractory zinc oxide ore which contains high-content alkaline gangues [1]. However, if the ore is treated just by flotation, its recovery is very low due to the poor processing efficiency of zinc oxide. In this case, the massive zinc metal are lost in the slime resulting in waste of resources. If the raw ore is directly processed by hydrometallurgy, high-content alkaline gangues will consume a large amount of acid leading to very high beneficiation cost [2]. The disadvantages of ammonia leaching process are serious environmental pollution, high energy consumption and equipment investment [3]. A new dressing-metallurgy combination process can avoid effectively the above problems. In recent years, the rapid development of acid leaching-extraction-electrodeposition technology, greatly promote the zinc hydrometallurgical process [3-5]. Nevertheless, most of zinc oxide ore has the characteristics of low grade, high oxidation rate and high-content alkaline gangues, is inappropriate to adopt directly the method of acid leaching treatment, require pretreatment of flotation and enrichment of zinc concentrate grade to more than 15% [6]. Under the premise of ensuring zinc recovery rate, removing the calcium and magnesium alkaline gangues.

Response surface methodology is the product of combining mathematical method and statistical method, with the scientific design and intuitive three-dimensional curved surface and contour output, product design and process optimization are the favor of the researchers. However, in terms of flotation process for refractory zinc oxide ore, the application of response surface methodology has not been reported. According to the test results and the response surface methodology, the flotation process conditions of refractory zinc oxide ore will be optimized by the author, providing the technical basis for the comprehensive utilization of low grade refractory zinc oxide ore with high-content alkaline gangues.

2. Experiment

2.1 Materials

The samples used in this work were taken from the Lanping mine located in the west of Yunnan province, China. Complete physical, chemical and mineralogical characterization was carried out before performing the flotation tests. The chemical analysis and phase analysis results of zinc oxide sample are shown in Table 1 and Table 2, respectively. Reagents such as sodium carbonate, sodium sulfide, F1(modified depressant) and HHA (modified amine collector) were used in this investigation.

Table 1 Chemical analysis of zinc oxide sample

Component	Zn	Pb	Fe	SiO_2	CaO	MgO	Al_2O_3
Content(%)	7.45	1.12	5.36	26.78	22.51	2.16	2.53

Table 2Phase analysis of zinc oxide sample

Zinc phase analysis	Zinc carbonate	Zinc silicate	Zinc sulfide	Others	Total Zn
Content(%)	3.75	3.14	0.48	0.08	7.45
Distribution rate(%)	50.34	42.15	6.44	1.07	100.00

2.2 Experimental method and apparatus

The flotation experiments containing two rougher were done in the XFD flotation machine and the impeller speed was 1998 rpm. Sodium carbonate with a sample of 500g in weight was added into the rod mill to be ground together for pH adjustment and dispersing slime. Slurry containing 500g ground sample was fed to flotation cell (1.5L) and the pulp density was 30%. A 2% F1 solution was used for depressant of gangues, and then Na_2S was added to the pulp and conditioned for 5 min. After conditioning, pH reached to 10.5～11 which was considerably suitable for adding other reagents especially collector HHA. Finally, the zinc concentrate and tailing were filtered, dried, weighed and analyzed when flotation operation were finished.

2.3 Designs of response surface methodology

According to the experiment principle of Box-Behnken Design, the three factors Na_2S concentration(X_1), F1 concentration(X_2) and Na_2CO_3 concentration(X_3) which influenced significantly flotation indexes were selected. Experimental design of 3 factors 3levels was carried out on the basis of single factor test. The Table3 shows the experimental factors and levels in terms of the equation $x_i=(X_i-X_0)/\Delta X$. x_i is code value of the argument, X_i is real value of the argument, X_0 is real value of the argument at the test center point, ΔX is change step of the argument.

Table 3 Experimental factors and levels

Factor	Code	Level		
	x_i	-1	0	+1
Na_2S concentration(g/t)	X_1	5000	6500	8000
F1 concentration(g/t)	X_2	0	750	1500
Na_2CO_3 concentration(g/t)	X_3	0	2000	4000

Notes: x1=(X1-6500)/1500; x2=(X2-750)/750; x3=(X3-2000)/2000.

3. Results and discussion

3.1 Experimental results

The recovery of Zn concentrate was used as the response value. According to the Box-Behnken Design, the corresponding test scheme and results are shown in Table4 in terms of the change of the three factors.

Table 4 Box-Behnken Design arrangement and the experimental data

No.	Code			Real vale			Recovery of Zn concentrate(%)
	x_1	x_2	x_3	X_1	X_2	X_3	
1	0	0	0	6500	750	2000	88.76
2	0	1	-1	6500	1500	0	87.57
3	1	1	0	8000	1500	2000	87.94
4	0	1	1	6500	1500	4000	86.12
5	1	-1	0	8000	0	2000	88.42
6	1	0	1	8000	750	4000	90.05
7	0	0	0	6500	750	2000	88.76
8	1	0	-1	8000	750	0	87.58
9	0	-1	-1	6500	0	0	77.08
10	0	-1	1	6500	0	4000	80.59
11	-1	0	1	5000	750	4000	71.96
12	0	0	0	6500	750	2000	88.76
13	0	0	0	6500	750	2000	88.76
14	0	0	0	6500	750	2000	88.76
15	-1	0	-1	5000	750	0	72.33
16	-1	1	0	5000	1500	2000	70.14
17	-1	-1	0	5000	0	2000	75.13

3.2 Model establishment and significance test

The experimental data of zinc recovery in table 4 were implemented multivariate regression fitting by using the Expert Design software. The quadratic multivariate regression equation of taking Zn recovery as the objective function can be obtained:

$$y = 88.76 + 8.05x_1 + 1.32x_2 + 0.52x_3 + 1.13x_1x_2 + 0.71x_1x_3 - 1.24x_2x_3 - 5.36x_1^2 - 3.00x_2^2 - 2.92x_3^2 \quad (1.1)$$

The validity of mathematical model is analyzed and tested, and the results are shown in table 5.

Table 5 Variance analysis of regression equation on zinc recovery

Source	Sum of squares	Degree of freedom	Mean square	F value	P value
Model	763.01	9	84.78	10.17	0.0029
x_1	518.90	1	518.90	62.24	< 0.0001
x_2	13.91	1	13.91	1.67	0.2374
x_3	2.16	1	2.16	0.26	0.6262
x_1 x_2	5.09	1	5.09	0.61	0.4604
x_1 x_3	2.02	1	2.02	0.24	0.6379
x_2 x_3	6.15	1	6.15	0.74	0.4188
x_1^2	120.80	1	120.80	14.49	0.0067
x_2^2	37.80	1	37.80	4.53	0.0707
x_3^2	35.99	1	35.99	4.32	0.0763
Residual	58.36	7	8.34		
Pure error	0.00	4	0.00		
Total deviation	821.37	16			

Notes: P<0.001 is extremely significant difference, P<0.01 is highly significant difference, P<0.05 is significant difference.

According to the results of table 5, it can be known that the model is highly significant and test error is very small. Table 5 also illustrates the concentration of sodium sulfide has great influence to the recovery of Zn concentrate.

3.3 Response surface analysis and optimization

The results are shown in Fig.1 in terms of the response surface plot on Zn recovery in the condition of varying Na_2S concentration and F1 concentration when Na_2CO_3 concentration is 2000g/t. As can be seen from the plot in Fig.1, the Zn recovery raises the maximum and then decreases slightly along with increasing Na_2S concentration and F1 concentration at the same time. Zn recovery did not change as increasing F1 concentration at low Na_2S concentration. Zn recovery first increases and then decreases when increases F1 concentration in the condition of high Na_2S concentration. Fig.2 illustrates the response surface plot on Zn recovery as a function of the different Na_2S concentration and Na_2CO_3 concentration. The Zn recovery can reach the maximum along with increasing Na_2S concentration when F1 concentration is 750g/t and Na_2CO_3 concentration is 2000g/t. Zn recovery shows a trend of decline when Na_2CO_3 concentration is more than 2000g/t.

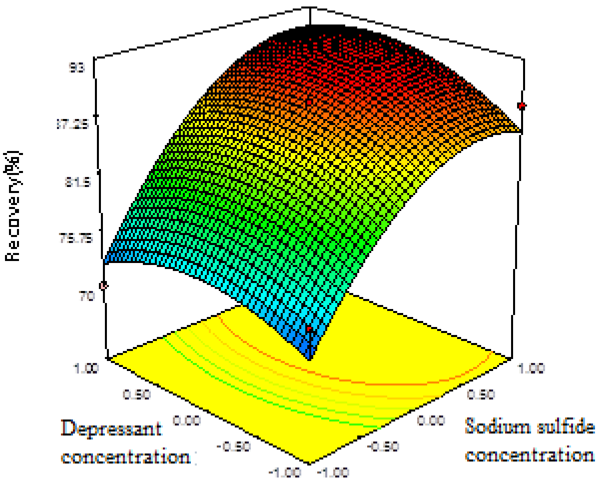

Fig 1

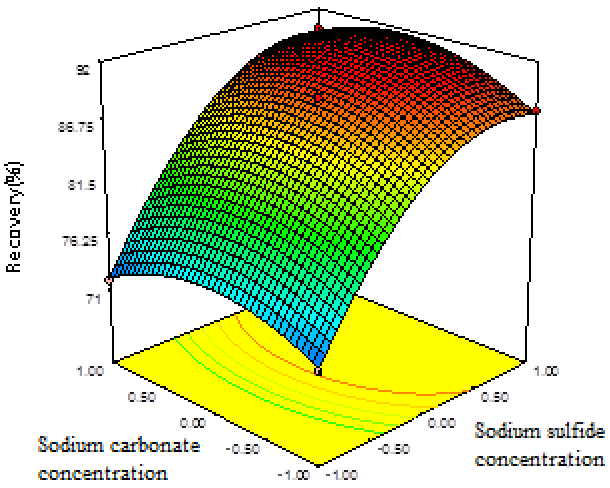

Fig 2

Fig1 Response surface plot for the effects of sodium sulfide concentration and depressant concentration on Zn recovery

Fig2 Response surface plot for the effects of sodium sulfide concentration and sodium carbonate concentration on Zn recovery

In order to determine the value of the optimal point, the quadratic multivariate regression equation of taking Zn recovery as the objective function was solved first order partial derivative, and making it equal to zero. The following set of equations can be got.

$$
\begin{aligned}
-10.72x_1 + 1.13x_2 + 0.71x_3 &= -8.05 \\
1.13x_1 - 6x_2 - 1.24x_3 &= -1.32 \\
0.71x_1 - 1.24x_2 - 5.84x_3 &= -0.52
\end{aligned}
\qquad (1.2)
$$

To solve the equation group 1.2: x_1=0.79, x_2=0.35, x_3=0.11, that is the optimal level value of the three factors. After transforming, the highest Zn recovery 92.22% can be gained when Na_2S concentration is 7685g/t, F1 concentration is 1012.5g/t, and Na_2CO_3 concentration is 2220g/t.

3.4 Verification test

In order to test the reliability of the results obtained by the response surface method, three sets of parallel verification tests were conducted under the conditions of the selected conditions(Na_2S concentration is 7500g/t, F1 concentration is 1050g/t, and Na_2CO_3 concentration is 2200g/t). The results show that the average Zn recovery is 91.08%. So we can know that the theory predicted value of the Zn recovery is very close to the experimental value and the error is only 1.14%. Therefore, the optimal parameters of flotation process are accurate and reliable based on the response surface method, which has practical value.

4. Conclusions

(1) The Zn content in the original zinc oxide sample used in the study is 7.45%, and the contents of major zinc phase such as carbonate zinc, silicate zinc and sulfide zinc are 3.75%, 3.14% and 0.48%, respectively. It should be noted that the oxidation rate of the sample reaches as high as 93.56%, and there are 42.15% silicate zinc, which confirms that the sample is refractory zinc oxide ore of low grade.

(2) According to the results of variance analysis of quadratic multivariate regression equation on zinc recovery, it can be known that the model is highly significant and test error is very small, and the concentration of sodium sulfide has great influence to the recovery of Zn concentrate.

(3) The regression model between Zn recovery and sodium sulfide concentration, depressant concentration and sodium carbonate concentration was established. The verification tests results show that the model is reliable and can predict accurately the zinc recovery. It can be known that the optimizing process parameters are sodium sulfide 7500g/t, depressant(F1) concentration 1050g/t, and sodium carbonate concentration 2200g/t.

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