Optimization of Flotation Process for Reduction of Alumina and Silica from Screw Classifier Overflow of an Iron Ore Washing Plant

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Abstract

Reverse flotation process was optimized by an experimental programme based on statistical analysis for reduction of silica and alumina levels from screw classifier overflow so as to enrich iron values. Flotation of alumina and silica bearing minerals with Sokem 521C and starch as collector and depressant respectively was studied to estimate their optimum levels at different particle sizes. A two-level three factor design of experiments showed that particle size is insignificant in the ranges of study. Tests on an orthogonal design of the hexagonal type were then carried out to determine the effects of the other two variables, on the response, Selectivity Index (SI), a measure of separation efficiency of iron values from alumina and silica. Regression equations were developed as models and response contours were plotted. Maximum response (SI of 2.25) has been optimized at 0.306 kg/t of amine collector, 1.0043 kg/t of starch at a particle size of 40 µm.

Keywords: Reverse flotation; iron ore; alumina; silica; selectivity index; statistical analysis

1. Introduction

Flotation is a complex process involving many variables. The selection of cationic amine collector, Sokem 521C, for the reduction of alumina and silica from the slimes generated as the overflow of screw classifier of an operating iron ore washing plant was discussed in an earlier study[i]. Out of the four cationic collectors, Sokem 521C was selected for further investigations. It is difficult to optimize it through a series of trial and error experiments. Results obtained for the effects of a variable on the measured response of the system using the one variable at a time technique can be misleading. This is due to interactions between factors; the observed effects at one set of conditions may not be reproduced at some other set of conditions.

Experimental programmes using statistical designs offer an effective method to solve this problem [ii]. The design is amenable to analysis of variance, tests of significance, mathematical modeling and optimization. Among a set of variables, the significant ones that have a direct bearing on the desired response could readily be ascertained and all possible interactions sorted out separately.

In mineral processing research, the statistical approach has found much use lately [iii, iv]. In this paper, optimization of the reverse flotation of iron ore slimes for reduction of alumina and silica using a statistical approach is presented. The experiments were first carried out using two- level three factor design. Analysis of variance was carried out to determine the significance of the factors. Experiments based on an orthogonal design of the hexagonal type were then carried out to determine the effects of variables, on selectivity index (SI), a measure of separation efficiency of iron values from gangue (alumina and silica). Based on regression equations, response contours were plotted and the maximum response in terms of SI was optimized.

2. Materials and Methodology

2.1 Materials

The details of the plant flow sheet, material’s size, chemical, mineralogical and liberation characteristics, reagents used, selection of cationic collector and the flotation process methodology are outlined elsewhere [v, vi, vii]. However, in the present investigation, the grinding period was so optimized as to get a product of d_{80} : 20 µm and flotation tests were conducted in a single stage by adding the entire collector in one installment.

2.2 Statistical design

Initial screening tests were conducted using a 2^3 factorial design [viii]. Even though the reverse flotation of iron ore is affected by different variables, three of the factors, viz., concentration of amine collector, Sokem 521C, concentration of starch as depressant for iron bearing minerals and particle size (d_{80}, 80% passing size) were studied at two levels. The two levels of the factors were fixed on the basis of flotation results obtained earlier. The variables were coded between -1 and +1, where -1 represented the low level and +1 the high level of the factor. The levels of coding of factors are indicated in Table 1 and the possible combinations of the 2 levels and 3 factors are given in Table 2. The experimental error variance was obtained through duplicated experiments as shown in Table 3. The calculations of the effects and mean squares based on Yate’s method are shown in Table 4. The ‘F’ test for significance of each factor and their interactions was carried out at 99% confidence level. An orthogonal design of hexagonal type was used to determine the influence of two variables which are found to be significant. Regression equation was developed. The response contours were plotted and optimum conditions were determined.

3. Results and Discussion

Out of the four collectors, viz., Soken 503C, Sokem 504C, Sokem 520C and Sokem 521C, the collector Sokem 521C was selected for further investigations [i]. The selected collector is said to be ether amine based. Optimization of the flotation process using the collector Sokem 521C, depressant starch and particle size of the slimes is discussed in this paper.

Table 1 Levels of factors for 2^3 design

Factor	Level of Factors	
	Low (-)	High (+)
‘A’ - Collector, ‘Sokem 521C’	0.2 kg/ton	0.4 kg/ton
‘B’ - Depressant, ‘Starch’	0.5 kg/ton	1.5 kg/ton
‘C’ Particle size (d_{80})	20 µm	40 µm

Table 2 Treatment combinations for 2^3 design

Treatment Combination Symbol	Amine collector (A)	Starch Depressant (B)	Particle size (d_{80}) (C)
1	-	-	-
a	+	-	-
b	-	+	-
ab	+	+	-
c	-	-	+
ac	+	-	+
bc	-	+	+
abc	+	+	+

(-) represents the lower level of the factor
(+) represents the higher level of the factor

Flotation of alumina and silica bearing minerals is controlled by several variables such as type of amine collector, pH, type of starch, starch preparation, conditioning period, water quality and particle size. From the previous experience, it was decided to study the effect of only three parameters, viz., dosage of collector, dosage of starch and particle size of the slimes, on the main response, the selectivity index (SI). SI was defined by Douglas [ix] and given by

$SI=[(R-C)*(c-f)*100]/[(100-C)(c_{max.}-f)]$ …(1)

where ‘R’ = % recovery of iron in the concentrate, ‘C’ = % weight of the concentrate, ‘c’ = % iron of the concentrate, ‘$c_{max.}$’ = maximum (theoretical) iron in the concentrate and ‘f’ = % iron of the feed. An index of 100 is indicative of a perfect separation between the valuable minerals and the gangue or waste; an index of zero indicates no separation. The numerical value of this selectivity index, as defined, is adjusted for the variations in head assay. As such, therefore, it served as a useful measure of the efficacy of the separation process on a number of different samples as well. The results of 8 tests corresponding to 2^3 factorial design are shown in Table 3.

Table 3 Results of 2^3 factorial design experiments
Feed: 500g (%Fe: 60.43, %SiO_2:6.88, %Al_2O_3:3.26)

Treatment combination	Responses (for the concentrate generated)					
	%Fe	%SiO_2	%Al_2O_3	Wt, %	%Fe recovery	SI
1	62.12	5.43	2.92	70.6	73.09	1.41
a	63.55	4.23	2.38	28.8	30.49	0.73
b	60.89	6.34	3.49	82.6	83.95	0.35
ab	63.38	4.26	2.41	27.1	28.65	0.62
c	62.55	4.99	2.64	54.0	56.40	1.09

ac	63.59	4.19	2.33	28.0	29.58	0.68
bc	62.22	5.25	2.79	67.6	69.76	1.18
abc	63.43	4.13	2.28	25.0	26.73	0.68
Repeat tests						
ab	63.55	4.16	2.39	27.3	28.94	0.69
ab	63.13	4.57	2.59	29.6	31.21	0.61
ab	63.41	4.25	2.45	28.7	30.42	0.71
ab	63.24	4.26	2.40	30.4	32.26	0.74
ab	63.25	4.26	2.36	30.4	32.31	0.76

For the repeat tests, Mean (μ) = (0.69 + 0.61 + 0.71 + 0.74 + 0.76)/5 = 0.702

SSE = $\sum(X - \mu)^2 = (0.69-0.702)^2 + (0.61-0.702)^2 + \ldots + (0.76-0.702)^2 = 0.01348$

MSE = SSE/d.f = 0.01348/5-1 =$3.37*10^{-3}$ where d.f is degrees of freedom.

Calculation of the effects and mean squares by Yate's Algorithm Method [ii] for SI is illustrated in Table 4. The main factor and interactions which are significant at 99% confidence level based on F-tests [ii], are also shown in Table 4. It was found that factor A, namely, amount of collector Sokem 521C was the most significant followed by factor B (starch dosage). The effect of particle size was not significant at the level used in the study. Among the interactions, BC was more significant. Such an observation indicates that the response – variables relationship is more complicated and a simple linear model may not suffice.

The next step is to assume a suitable mathematical model relating the response with respect to the significant variables [x]. From an analysis of the above factorial design, it is clear that the particle size is not a significant factor in the ranges of study. Hence, it is now possible to eliminate this factor so as to confine ourselves to a more compact and better design that would fit the assumed model with known values of least square coefficients.

Table 4 Calculation of effects and mean squares by Yate's Algorithm

Response: Selectivity Index (SI)

Factors	Sum of squares	d.f	Mean squares	'F' = (Mean squares) / MSE
Main Effects				
A *	0.2178	1	0.2178	64.63
B *	0.1458	1	0.1458	43.26
C	0.0338	1	0.0338	10.03
Two Factor Interactions				
AB *	0.09245	1	0.09245	27.43
AC	0.03125	1	0.03125	9.27
BC *	0.19845	1	0.19845	58.89
Three Factor Interactions				
ABC *	0.1325	1	0.1352	46.53

Factors with (*) indicate that they show high significance at 99% confidence level.

A general model represented in the following form was assumed.

$$Y = B_0 + B_1X_1 + B_2X_2 + B_{11}X_1^2 + B_{22}X_2^2 + B_{12}X_1X_2 \quad \ldots \quad (2)$$

where X_1 and X_2 are the two variables and B_0, B_1 and B_2 are the first order least square coefficients. B_{11} and B_{22} are the second order coefficients while B_{12} is the cross factor coefficient representing interaction between the factors. To achieve satisfactory fit, an orthogonal model of the hexagonal type was adopted. The design points along with results are given in Table 5. The range of variables is also given and they are coded to cover the range of +1 to -1. The experimental points derived after coding were tested at random at a fixed particle size of 40 μm with other conditions fixed as before to determine the respective responses.

Table 5 Hexagonal design of experiments with particle size fixed at 40 µm

Test No.	Design levels		Factor levels		SI
	X_1	X_2	Sokem 521C (kg/t)	Starch (kg/t)	
1	1	-1	0.4	0.5	0.68
2	1	0	0.4	1.0	2.76
3	1	1	0.4	1.5	0.68
4	-1	0	0.2	1.0	1.51
5	0	0	0.3	1	2.58
6	-1	-1	0.2	0.5	1.09
7	-1	1	0.2	1.5	1.18

Code X_1 = (Concentration of Sokem 521C – 0.3)/0.1
Code X_2 = (Concentration of Starch – 1.0) / 0.5

A Forward Doolittle technique [ii, iii] was used to determine the values of coefficients in the model represented above to generate regression equation relating the response SI to the two parameters affecting it. The coefficients were calculated and the equation obtained was as shown below.

$$Y = 2.251 + 0.0567X_1 + 0.0225X_2 – 0.4443X_1^2 - 1.2235X_2^2 - 0.0225X_1X_2 \quad …. \quad (3)$$

In order to optimize the process, we need to find the conditions where we get the maximum response. For that we differentiate the regression equation with respect to X_1 and X_2 and set them equal to zero.

Differentiating equation (3) with respect to X_1 and X_2 and equating it to zero,

$$0.0567 – (2*0.4443X_1) – (0.0225X_2) = 0$$
$$0.0225 – (2*1.2235X_2) – (0.0225X_1) = 0$$

X_1 = 0.06359, i.e., optimum dosage of Sokem 521C= 0.306 kg/t

X_2 = 0.00861, i.e., optimum dosage of starch = 1.0043 kg/t of starch

We find that the maximum response (SI) near 2.26 is obtained when we have Sokem 521C concentration of 0.306 kg/t and a starch concentration of 1.0043 kg/t . Since we have got higher responses as indicated by the hexagonal design, we owe this result to the variance between the regression curve and the experimental values. In order to estimate the nature of the fit, Multiple Regression Coefficient was calculated.

R^2 = (Variance due to B_1 … B_{12}) / (Total Variance – Variance due to B_0)

$R^2 = 0.7742$.

If the value of R^2 exceeds 0.75, the agreement between the experimental data and the mathematical model is considered sufficient.

For better visualization, the surface contours of the response (SI) as well as surface plots of the regression equation are included (Figures 1 & 2).

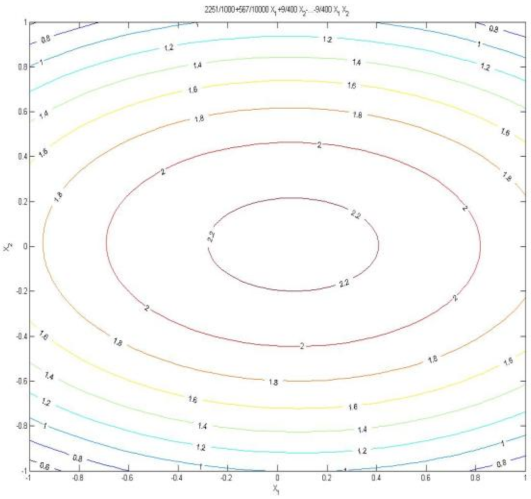

Figure 1 Surface contours for the response, selectivity index

4. Conclusions

1. Statistical design of experiments and analysis of data were used for optimization of reverse flotaion process of iron ore slimes generated as overflow of screw classifier of an operating washing plant.

2. 2^3 factorial experiments using the variables – concentration of the collector Sokem 521C, concentration of starch and the particle size of the slimes – were carried out at two levels – low and high. Yate's analysis and calculations of effects of factors showed that concentrations of Sokem 521C and starch are significant at 99% confidence level.

3. Orthogonal design of hexagonal type experiments have been carried out, regression equation developed, and the fit measured using the regression coefficient. Response contours have been plotted.

4. The parameters have been optimized at 0.306 kg/t of Sokem 521C , 1.0043 kg/t of starch and particle size of 40 µm at a maximum response of Selectivity Index, 2.26 .

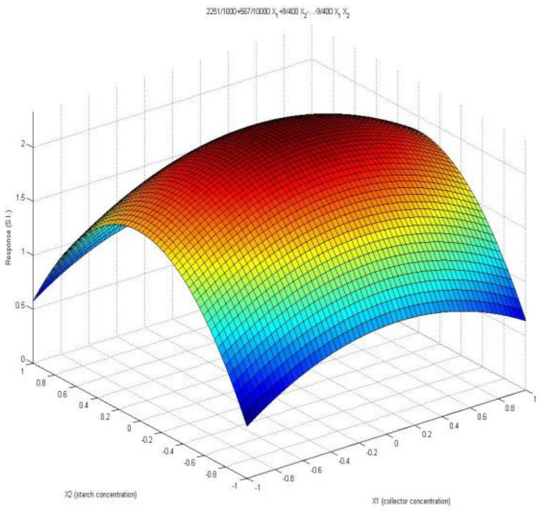

Figure 2 Surface plots for the regression equation

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