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Optimization of reagent dosages for copper flotation using statistical technique

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Abstract: The effects of Z11 and AP407 collectors as well as AF65 and AF70 frothers were evaluated in the rougher flotation circuit of the Sungun copper concentrator plant using 2^4 full factorial design. Response functions were produced for both Cu grade and recovery and optimized within the experimental range. The optimum reagent dosages were found to be 12.01 g/t Z11, 11 g/t AP407, 3 g/t AF65 and 5 g/t AF70 to attain the maximum Cu grade (8.17%). The reagent dosages of 12 g/t Z11, 11 g/t AP407, 3 g/t AF65 and 15 g/t AF70 produced the maximum Cu recovery (86.44%). The collector distribution demonstrated that the distribution pattern of (32%, 32%, 20%, 16%) can produce the best recovery (87.75%) in comparison to other examined distribution patterns.

Key words: copper sulphide ores; flotation reagents; modelling; optimization

1 Introduction

Froth flotation is a process used for selectively separating hydrophobic materials from hydrophilic. Various factors, such as the type and quantity of chemicals added[1−2], the bubble size[3], stator and rotor configuration[4] and residence time[5] influence the performance of a flotation unit.

The types and quantity of the reagents are the most important part of the flotation process. In commercial plants, the control of reagent additions is the most important aspect of the flotation strategy[6].

Reagent schemes used for the treatment of porphyry copper and copper-molybdenum ores are relatively simple and usually involve lime as a modifier, xanthate as the primary collector, and a secondary collector that includes dithiophosphates, mercaptans, thionocarbamates and xanthogen formates. The frothers, such as methyl isobutyl carbonyl, TEB (alkoxy paraffin and pine oil), Dow 250, Dow 1012 (glycols), HP700 and HP600 (alcohols in amine oxide) are typically used in the flotation of porphyry copper and copper molybdenum ores[6].

In copper flotation plants, an increase of 1%−2% in recovery and/or grade is economically remarkable. In this work, reagent optimization is a very important issue, and much time and attention are spent on the optimization of flotation reagents in order to provide the most effective separation and concentration results.

The advantages of the statistical design of experiments over classical treatment of one variable at a time were demonstrated in mineral processing industry[7−14]. One of these statistical techniques was the factorial design test, which was used to study several factors and determine their main effects and interactions[15].

The aim of the present work is to use statistical techniques to optimize the dosages of four flotation reagents, the collectors of Z11(isopropyl xanthate sodium), AP407 (a mixture of mercapto benzothiazole and dithiophosphate), the frothers of AF65 (ether polyglycol) and AF70 (methyl isobutyl carbonyl) in the rougher circuit of the Sungun Copper Concentrator Plant to achieve the maximum grade or recovery. By using this procedure, the main effects and interactions of these reagents on copper flotation performance are determined. The main steps in the current work are: 1) designing and performing of experiments on laboratory scale; 2) analysis of experimental results to determine the significant factors influencing the copper grade and recovery in the rougher stage; 3) determination of copper grade and recovery equations as functions of the reagent dosages; 4) establishing optimum reagent dosages to

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maximize the grade or recovery; 5) optimization of reagent distribution in the rougher circuit.

2 Sungun Copper Concentrator Plant

The Sungun copper mine is located in the eastern Azerbaijan Province in northwestern Iran. The total mineral resource of the Sungun copper reserve, at 0.25% Cu cut-off grade, is estimated to be 800 million tons and believed to exceed 1 billion tons. The total minable ore based on mine design and production scheduling is 400 million tons with an average Cu grade of 0.62%.

In the Sungun Concentrator Plant, the crushed ore is fed into a semi-autogeneses (SAG) mill to produce a product with K_{80} = 3 mm (Fig.1). The SAG mill products are then transferred to the two ball mills, where the ore is ground to a level of K_{80} = 80 μm. Lime, collectors of Z11 and AP407 are added to the ball mill feed as well. Then the product is discharged into a rougher flotation conditioner tank. The pH level of the feed with respect to the rougher flotation is measured in this conditioner tank and lime slurry is added. Additionally, the frothers AF65 and AF70 are added to the conditioner tank. The conditioner tank overflow enters the 12 rougher flotation tank cells which are grouped into 6 banks of 2 cells each. Further reagents (collectors) are added to the flotation cells 3, 5 and 9. The collector distribution pattern is 32% for ball mill feed, 32% for the third rougher flotation cell, 20% for the fifth rougher flotation cell and 16% for the ninth rougher flotation cell.

Tailings from the last rougher flotation bank together with cleaner scavenger tailing form the final plant tailing output stream. The rougher concentrate obtained from each cell is combined with the cleaner scavenger concentrate and pumped to the regrind hydrocyclone clusters. Hydrocyclone underflow, after lime addition, reports to the regrind ball mill. The regrind ball mill is operated in a closed circuit with the hydrocyclone cluster. The hydrocyclone overflow (K_{80} = 40 μm) is transferred to two cleaner column cells. Concentrate obtained from the cleaner columns is transferred to the re-cleaning column cell to produce the final concentrate with a 30% (nominal) copper grade and about 84% total Cu recovery.

3 Experimental

3.1 Material

The copper ore sample used in this study was prepared from the SAG mill feed. The sample with K_{80} =80 μm was prepared after two stages of laboratory crushing, including jaw and roll crushers and ball mill grinding. Chalcocite (0.36%), chalcopyrite (0.37%), covellite (0.2%) and pyrite (6.9%) are the sulfide minerals in the representative sample with Cu content 0.61%, Cu oxide content 0.054% and Fe content 3.33%.

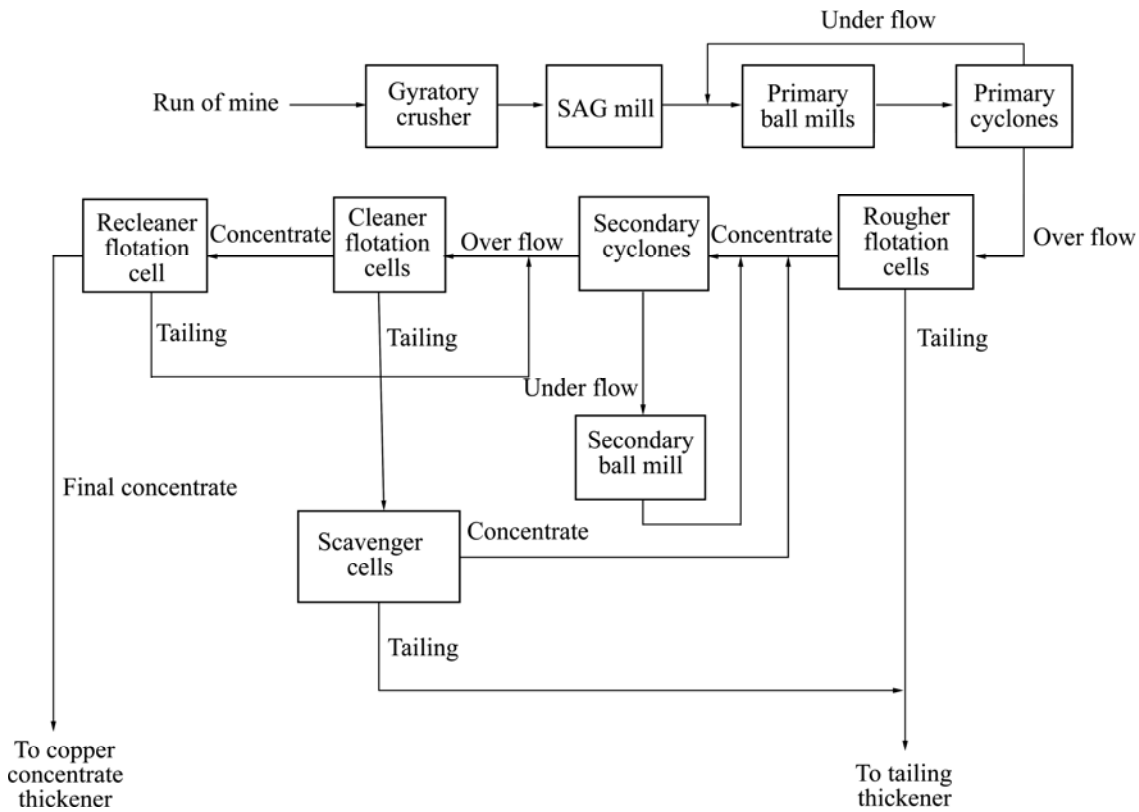

Fig.1 Flow sheet for Sungun copper flotation plant

3.2 Methods

The flotation studies were performed according to the full factorial design of experiments. The variables studied were the dosages of the Z11 and AP407 collectors and the AF65 and AF70 frothers. The variables and levels of full factorial design in both coded and actual values are presented in Table 1. The higher and lower levels are presented with +1 and -1 signs. The averages of reagent dosages used in 90 operating days in the plant which constituted the sampling period, were considered as the centre points in the experimental design.

Table 1 Variables, symbols and levels used for full factorial design

Variable	Symbol	Dosage/(g·t^{-1})		
		Low level	Center level	High level
		−1	0	+1
Z11	X_1	12	22	32
AP407	X_2	11	21	31
AF65	X_3	3	6	9
AF70	X_4	5	10	15

A Denver flotation machine was used for the flotation studies. The sample (1 175 g) was mixed with water to adjust the solid concentration to 34% and conditioned in a 3 L flotation cell for 3 min. The pH value was then adjusted to 11.8 using lime before adding the reagent. The reagents were added according to experimental design in predetermined quantities. The slurry was further conditioned after the addition of each reagent, i.e. 2.5 min for the collectors and 1 min for the frothers. An impeller speed of 1 250 r/min, a flotation time of 16 min which is rougher than the flotation time of 33 min scaled down from the Sungun plant were applied for all the experiments. The froth and tailing were collected separately, filtered and analyzed for copper grade and recovery calculations.

After optimizing the reagent dosages according to the distribution of (100%, 0, 0, 0), an optimization of collector distribution was performed. The collector distribution patterns of (32%, 32%, 20%, 16%) (current distribution in the plant), (25%, 25%, 25%, 25%), (75%, 10%, 15%, 0) and (40%, 10%, 40%, 10%) relative to the ball mill feed, the third rougher flotation cell, the fifth rougher flotation cell and the ninth rougher flotation cell were examined in the laboratory, respectively. The froth collecting time of 960 s was divided into 160 s for simulating the first and second rougher flotation cells, 160 s for simulating the third and fourth rougher flotation cells, 320 s for simulating the fifth to eighth rougher flotation cells and 320 s for simulating the ninth to twelfth flotation cells.

4 Results and discussion

4.1 Statistical design

16 sets of tests were necessary based on the $N = 2^n$ equation, where N and n are the number of tests and variables, respectively. As a basic principle in the design of the experiments, 4 experiments were carried out at the base level to estimate both error and standard deviation. The statistical design was performed using Design-Expert software. A matrix for 4 variables and the corresponding grade and recovery of copper are shown in Table 2. Analysis of variance was performed to evaluate the significance of the effects and the interactions among the investigated factors. An effect is considered to be significant if its significance level is greater than 95%, which means the model with a P-value lower than 0.05 could be considerable.

Table 2 Full factorial design with coded values and results for both Cu grade and recovery

Run	X_1	X_2	X_3	X_4	Observed results	
					Recovery/%	Cu grade/%
1	1	1	−1	1	83.80	5.72
2	1	−1	1	1	83.81	3.56
3	0	0	0	0	83.40	3.87
4	1	−1	−1	1	84.78	3.93
5	1	1	−1	−1	79.59	5.46
6	−1	−1	−1	−1	67.40	8.58
7	1	1	1	1	84.17	3.83
8	−1	1	−1	1	85.05	3.93
9	−1	1	1	1	85.26	4.19
10	0	0	0	0	79.38	4.90
11	−1	1	−1	−1	81.75	4.67
12	1	−1	1	−1	85.75	4.95
13	1	1	1	−1	84.82	5.32
14	−1	−1	−1	1	87.03	3.78
15	0	0	0	0	80.00	4.19
16	0	0	0	0	81.50	4.27
17	−1	−1	1	1	85.35	4.42
18	−1	1	1	−1	85.26	5.10
19	−1	−1	1	−1	75.12	4.42
20	1	−1	−1	−1	80.50	5.89

4.1.1 Model equation for Cu recovery

To evaluate the significance of effects for recovery, an analysis of variance was conducted at 95% confidence intervals. The result is shown in Table 3. Based on the result, the following model was obtained using regression analysis:

$$y_r=82.47+0.94X_1+1.25X_2+1.23X_3+2.44X_4-1.55X_1X_2-1.7X_1X_4-1.58X_2X_4-1.49X_3X_4+1.74X_1X_2X_4 \quad (1)$$

where y_r is the predicted Cu recovery; X_1, X_2, X_3 and X_4 are coded values for Z11, AP407, AF65 and AF70, respectively.

Table 3 Analysis of variance for recovery

Source	Sum of squares	df	Mean square	F value	P-value Prob > F
Model	367.27	9	40.81	23.61	< 0.000 1 S
X_1	14.06	1	14.06	8.14	0.019
X_2	24.9	1	24.9	14.41	0.004 2
X_3	24.11	1	24.11	13.95	0.004 7
X_4	95.36	1	95.36	55.18	< 0.000 1
X_1X_2	38.69	1	38.69	22.39	0.001 1
X_1X_4	46.44	1	46.44	26.88	0.000 6
X_2X_4	40.13	1	40.13	23.22	0.000 9
X_3X_4	35.34	1	35.34	20.45	0.001 4
$X_1X_2X_4$	48.23	1	48.23	27.91	0.000 5
Curvature	6.23	1	6.23	3.60	0.090 1 NS
Residual	15.55	9	1.73		
Lack of fitness	5.94	6	0.99	0.31	0.897 3 NS
Pure error	9.61	3	3.2		
Cor total	389.05	19			

NS: not significant; S: significant; CV = 1.60%, R^2 = 95.94%, Adj. R^2 = 91.87%

According to the analysis variance (Table 3), the Fisher's F–test with a very low probability value ($P_{\text{Model}}>F$)<0.000 1 indicates the model is highly significant. To evaluate the fitness of Eq.(1), a regression-based determination coefficient R^2 should be determined[16−17]. When the values of R^2 are close to 1, the model offers an appropriate explanation of the variability of experimental values to the predicted values[17−18]. The recovery model presents a high determination coefficient R^2= 0.96, explaining 96% of the variability in the response. The adjusted determination coefficient (Adj. R^2= 0.9187) was also satisfactory and confirmed the significance of the model. The observed Cu recoveries and the predicted values obtained using Eq.(1) are shown in Table 4 and Fig.2. The model also showed that the lack of fitness ($P_{\text{Model}}>F$)=0.8973 was not significant; thus, the model was considered to be adequate for prediction within the range of variables employed. A low value for the coefficient of variation was observed (CV = 1.60%), which indicated both the precision and the reliability of the experiments. The P-value of curvature in the factorial experiment for recovery was 0.0901, which is higher than 0.05, indicating a non-significant curvature measured by the difference between the average of the centre points and the factorial points in the design space.

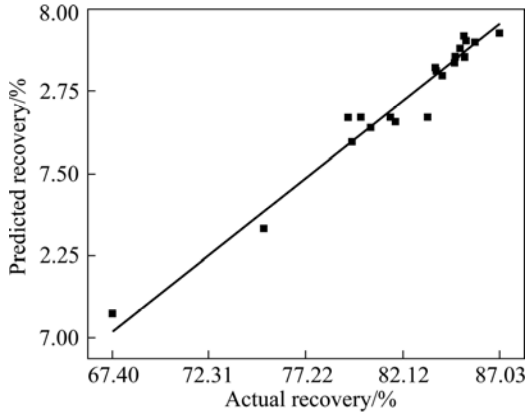

Fig.2 Predicted recoveries versus actual recoveries

Table 4 Observed and predicted values for Cu grade (K, %) and recovery (y_g)

	Run Order	$1/K^{1/2}$ Observed	$1/K^{1/2}$ Predicted	Recovery/% Observed	Recovery/% Predicted
1	6	0.341 39	0.349 69	67.4	68.55
2	20	0.412 04	0.425 69	80.5	80.41
3	11	0.462 74	0.453 52	81.75	80.79
4	5	0.427 96	0.438 60	79.59	79.49
5	19	0.475 65	0.463 25	75.12	73.97
6	12	0.449 46	0.436 45	85.75	85.84
7	18	0.442 80	0.456 34	85.26	86.22
8	13	0.433 55	0.422 05	84.82	84.92
9	14	0.514 34	0.501 21	87.03	86.45
10	4	0.504 43	0.516 13	84.78	84.55
11	8	0.504 4	0.488 30	85.05	85.41
12	1	0.418 12	0.412 30	83.80	84.24
13	17	0.475 65	0.484 66	85.35	85.93
14	2	0.529 99	0.518 94	83.81	84.04
15	9	0.488 53	0.499 06	85.26	84.90
16	7	0.510 97	0.525 85	84.17	83.73
17	15	0.488 53	0.483 13	80.00	81.07
18	10	0.451 75	0.483 13	79.38	81.07
19	16	0.483 93	0.483 13	81.50	81.07
20	3	0.508 32	0.483 13	83.40	81.07

The main effect of all the variables on recovery was significant at 95% confidence intervals. Positive coefficients of X_1, X_2, X_3 and X_4 variables indicated a positive effect for increasing recovery. The order of influences was $X_4 > X_2 > X_3 > X_1$. The effect of AF70 (X_4) was both highly significant and positive. Among the interactions, X_1X_2, X_1X_4, X_2X_4 and X_3X_4 had negative coefficients, while $X_1X_2X_4$ had a positive coefficient.

4.1.2 Model equation for Cu grade

To assess the significance of effects for the Cu grade, analysis of variance was conducted at 95% confidence intervals. The results are shown in Table 5. Based on these results, the following model was obtained using regression analysis:

$$y_g=0.46+0.014X_3+0.031X_4-0.012X_1X_2-0.012X_2X_4+0.01X_1X_2X_3+0.015X_1X_3X_4+0.017X_2X_3X_4 \quad (2)$$

where y_g is the predicted $1/K^{1/2}$ X_1, X_2, X_3 and X_4 are coded values of Z11, AP407, AF65 and AF70, respectively. The value of y_g produced a better correlation than Cu grade; therefore, it was considered as the dependent variable instead of Cu grade.

According to the analysis variance (see Table 5), the Fisher's F–test with a probability value ($P_{Model} > F$) = 0.000 1 indicated that the model was significant. The model for the grade presented a determination coefficient of R^2 = 0.89, explaining 89% of the variability in the response. The adjusted determination coefficient for the grade was 83.09%. The observed y_g and the predicted values obtained using Eq.(2) are shown in Table 4 and Fig.3. The model also shows the lack of fitness ($P_{Model} > F$) = 0.803 is not significant; thus, the model is considered to be adequate for predicting within the range of variables employed. A low value for coefficient of variation was observed (CV = 4.03%), which indicated both the precision and reliability of the

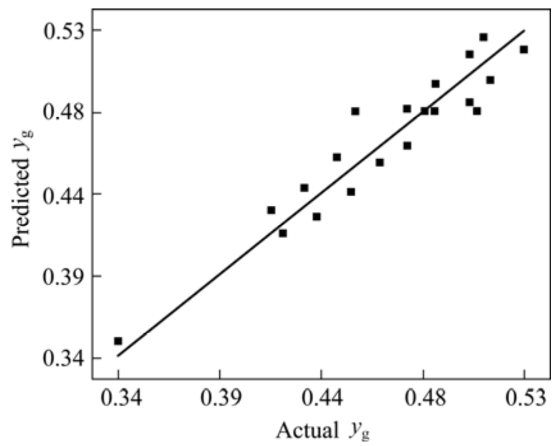

Fig.3 Predicted y_g versus measured values

experiments. The P-value of curvature in the factorial experiment for the grade was 0.069 3 which is more than 0.05, indicating a non-significant curvature in the design space.

The main effects of AF65 (X_3) and AF70 (X_4) on the Cu grade are significant at 95% confidence intervals. The most important effect was for AF70 (X_4) which was negative. The effect of AF65 (X_3) was also negative. The X_1X_2 and X_2X_4 interactions had a positive effect, while $X_1X_2X_3$, $X_1X_3X_4$ and $X_2X_3X_4$ all had negative effects on the Cu grade.

4.2 Three-dimensional (3D) response surface and cube plots for Cu grade and recovery

The 3D response surface plots are a graphical representation of the regression equation generally used to visualize the relationship between the response and experimental levels of each variable and the type of interactions between the variables to deduce the optimum conditions[16, 19].

Table 5 Analysis of variance for grade

Source	Sum of squares	df	Mean square	F Value	P-value Prob > F
Model	0.034	7	4.810×10^{-3}	13.64	0.000 1 S
X_3	3.057×10^{-3}	1	3.057×10^{-3}	8.67	0.013 4
X_4	0.016	1	0.016	44.45	< 0.000 1
X_1X_2	2.421×10^{-3}	1	2.421×10^{-3}	6.86	0.023 8
X_2X_4	2.277×10^{-3}	1	2.277×10^{-3}	6.46	0.027 4
$X_1X_2X_3$	1.740×10^{-3}	1	1.740×10^{-3}	4.93	0.048 3
$X_1X_3X_4$	3.731×10^{-3}	1	3.731×10^{-3}	10.58	0.007 7
$X_2X_3X_4$	4.765×10^{-3}	1	4.765×10^{-3}	13.51	0.003 7
Curvature	1.429×10^{-3}	1	1.429×10^{-3}	4.05	0.069 3 NS
Residual	3.880×10^{-3}	11	3.528×10^{-4}		
Lack of fit	2.231×10^{-3}	8	2.789×10^{-4}	0.51	0.803 0 NS
Pure error	1.649×10^{-3}	3	5.498×10^{-4}		
Cor total	0.039	19			

NS: not significant; S: significant; CV = 4.03%; R^2 = 89.67%; Adj. R^2 = 83.09%

Fig.4 shows the 3D response surface relationship between Z11(X_1) and AP407(X_2) with recovery at the centre level of AF65(X_3) and AF70(X_4). It is obvious that the highest recovery could be achieved with the maximum level of AP407(X_2) and the minimum level of Z11(X_1).

Figs.5−7 are response surface plots for the interactions X_1X_4, X_2X_4 and X_3X_4, respectively. It is seen that the recovery steadily increased with an increase of AF70(X_4) and a decrease of other variables. It is obvious that the addition of AF70 had a great effect on recovery.

Fig.8 shows the 3D response surface relationship between Z11(X_1) and AP407(X_2) on the Cu grade at the centre level of AF65(X_3) and AF70(X_4). It is obvious that the highest grade could be achieved with the maximum levels of AP407(X_2) and Z11(X_1) and with the minimum levels of Z11(X_1) and AP407(X_2).

Fig.9 shows the 3D response surface relationship between AP407(X_2) and AF70(X_4) on the Cu grade at the centre level of Z11(X_1) and AF65(X_3). It is obvious that the highest grade could be achieved with the minimum level of AP407(X_2) and AF70(X_4).

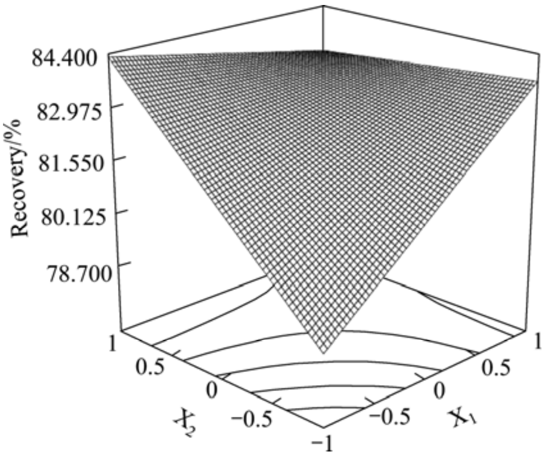

Fig.4 Effects of Z11(X_1) and AP407 (X_2) on recovery at centre level of AF65(X_3) and AF70(X_4)

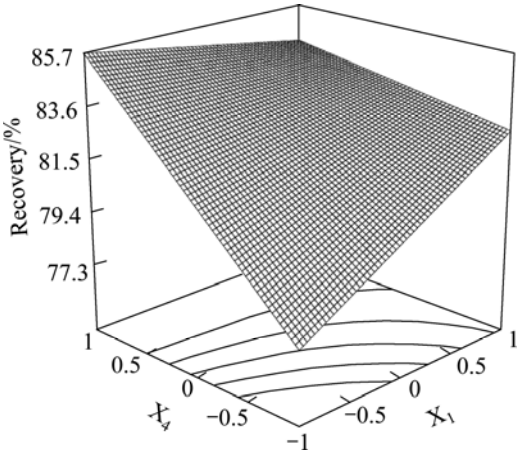

Fig.5 Effects of Z11(X_1) and AF70 (X_4) on recovery at centre level of AF65(X_3) and AP407 (X_2)

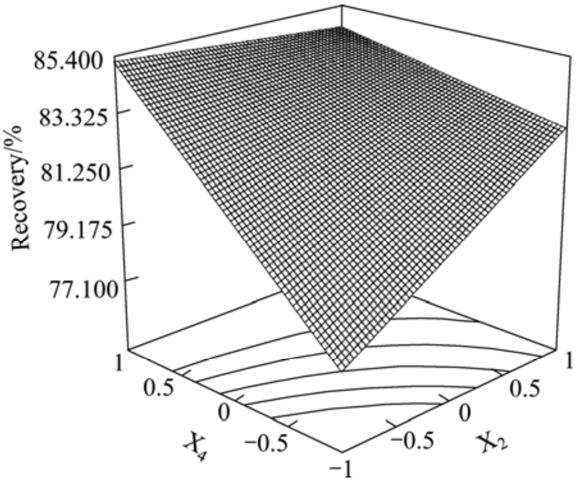

Fig.6 Effects of AP407(X_2) and AF70 (X_4) on recovery at centre level of AF65(X_3) and Z11(X_1)

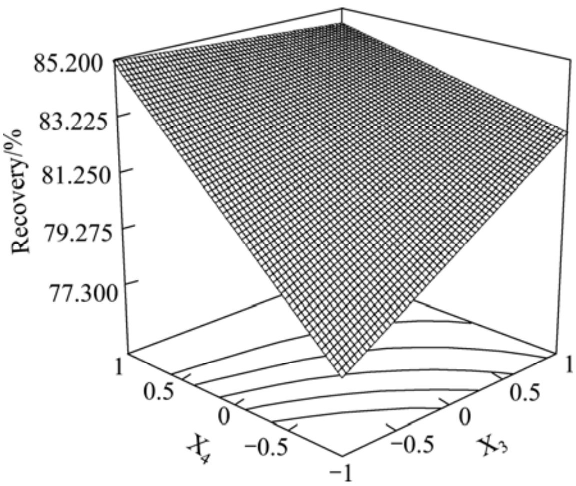

Fig.7 Effects of AF65 (X_3) and AF70 (X_4) on recovery at centre level of AP407 (X_2) and Z11(X_1)

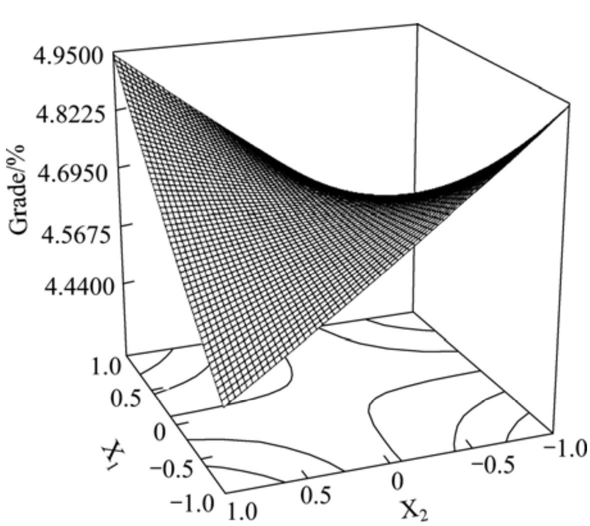

Fig.8 Effects of Z11(X_1) and AP407(X_2) on Cu grade at centre level of AF65(X_3) and AF70(X_4)

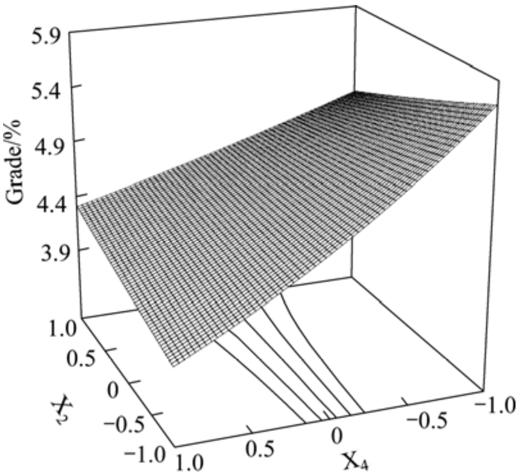

Fig.9 Effects of AP407(X_2) and AF70 (X_4) on Cu grade at centre level of AF65(X_3) and Z11(X_1)

4.3 Optimization studies for Cu grade and recovery

The optimum levels of the variables to maximize the Cu grade within the studied range were obtained by solving the regression equation using the Design-Expert software. The optimum variables were found to be −0.999 (12.01 g/t) for Z11, −1 (11 g/t) for AP407, −1 (3 g/t) for AF65 and −1 (5 g/t) for AF70 with a prediction of an Cu grade of 8.175%, respectively.

In the same way, the optimum levels of the variables to maximize recovery within the range studied were obtained by solving the regression equation using the Design-Expert software. The optimum variables were found to be −1 (12 g/t) for Z11, −1 (11 g/t) for AP407, −1 (3 g/t) for AF65 and +1 (15 g/t) for AF70 with a prediction of 86.447% recovery, respectively.

4.4 Optimization of collector distribution

Because of the large specific surface of fine particles in comparison to coarse particles, the consumption of the collector which was required to produce a specified degree of particle coverage for fine particles was much higher than that for coarse ones. In a flotation system that consists of fine and coarse particles, with a specified dosage of collector, fine particles use most of the collector, leaving insufficient collector available to produce the hydrophobic coverage which is required to float the coarse particles[20−23]; therefore, a suitable distribution of reagents along the flotation bank could increase both recovery and the grade on occasion.

Different collector distribution patterns were examined at the optimum levels of the reagent dosages for maximizing recovery (12 g/t Z11, 11 g/t AP407, 3 g/t AF65 and 15 g/t AF70). There was an opportunity for increasing copper grade in the subsequent cleaner and recleaner stages; additionally, in the current rougher circuit of the Sungun plant, the rougher tailing was rejected to the tailing dam. Therefore, distribution studies were performed on the reagent dosages to maximize recovery.

The flotation results for different selected distribution patterns (32%, 32%, 20%, 16%)[a], (32%, 32%, 20%, 16%)[b], (25%, 25%, 25%, 25%), (75%, 10%, 15%, 0) and (40%, 10%, 40%, 10%) are shown in Table 6. Each pattern was tested three times, and the average was reported. The distribution pattern of (32%, 32%, 20%, 16%)[a] was that of the current dosages of the rougher circuit of the plant, and in the pattern of (32%, 32%, 20%, 16%)[b], the reagents are in the optimized dosages for maximizing recovery in this study.

Table 6 Cu grade and recovery with different distribution patterns of collector

Distribution pattern	Recovery/%	Grade/%
(32%, 32%, 20%, 16%) [a]	86.75	3.98
(32%, 32%, 20%, 16%) [b]	87.75	4.58
(25%, 25%, 25%, 25%)	86.27	4.43
(75%, 10%, 15%, 0)	87.88	3.397
(40%, 10%, 40%, 10%)	86.91	3.14

a: With current reagent dosages in plant, b: With optimized reagent dosages to maximize recovery

It can be seen that the distribution of (32%, 32%, 20%, 16%)[b] is the most suitable one to produce maximum copper recovery and the consumption of reagents can be decreased from 22 to 12 g/t for Z11, 21 to 11 g/t for AP407 and 6 to 3 g/t for AF65, respectively. The only reagent with an increasing dosage is AF70 with 15 g/t comparing to the current consumption in the plant of 10 g/t.

5 Conclusions

1) A two-level full factorial design was used for modelling and optimizing four copper flotation reagents, namely, Z11 and AP407 as collectors and AF70 and AF65 as frothers.

2) The mathematical equations for both Cu grade and recovery were achieved by using sets of experimental data and Design-Expert software package.

3) The high correlation coefficients of the model equations for grade (R^2 = 0.89) and recovery (R^2 = 0.96) indicate that the predicted values are in good agreement with the observed values.

4) Optimization of regression equations to maximize the Cu grade was performed using the Design-Expert software. The dosages of 12.01 g/t Z11, 11 g/t AP407, 3 g/t AF65 and 5 g/t AF70 were optimal dosages with a prediction of 8.175% Cu grade.

5) The optimal dosages to achieve maximum recovery were found to be 12 g/t Z11, 11 g/t AP407, 3 g/t AF65 and 15 g/t AF70 with a prediction of 86.447% recovery. These results were achieved with a collector distribution pattern of (100%, 0, 0, 0).

6) At the optimal operating conditions for maximizing recovery, different collector distributions were examined in the laboratory. The results show that the distribution of (32%, 32%, 20%, 16%) can produce the highest recovery (87.75%) among the examined patterns.

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