MODELLING OF GALENITE FLOTATION FROM A DISSEMINATED LEAD AND ZINC ORE USING CENTRAL COMPOSITE DESIGN METHOD

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Abstract - In this work, modelling of galena flotation from a disseminated Lead and Zinc (Pb-Zn) ore containing 0,83% Pb was investigated. In flotation experiments, while the solids ratio and depressant amount (independent variables-parameters) were changed, the collector type and amount, the frother type and amount, particle size, pH, flotation time and the amount of two surfactants were kept constant. The response variables-dependent variables (Pb content and Pb Recovery) were described with mathematical models by using statistical design method. Central Composite Design (CCD) method which is one of the principle response surface methods, was used in statistical design of experiments, analysis of results and modelling of response variables. Two quadratic mathematical models were developed for Pb grade and Pb recovery.Analysis of variance showed that models, created for Pb grade and Pb recovery, were found statistically significant in 95% confidence level. Predicted vs actual values of Pb grade and Pb recovery were also given in graphically. It was found that predicted values represent approximately 96% and 94% of actual Pb grade and Pb recovery, respectively. The high values of R-square (R^2) indicate that the models developed for two response variables are powerful.

Keywords - Central Composite Design, Modelling, Pb flotation, Pb-Zn ore.

I. INTRODUCTION

Lead and zinc metals are utilized widely in electrical, mechanical, military, metallurgical, chemical, light and medical industries. Sulfide ores are the main resources for lead and zinc minerals [1], [2]. Froth flotation is the main beneficiation method to achieve lead and zinc sulfide minerals separation [3]-[6]. There are two basic approaches to obtain lead and zinc separation by flotation. First, flotation of lead sulphide is carried out and zinc sulphide and gangue minerals are depressed by various depressants and second, the bulk flotation of lead and zinc sulphide is carried out to obtain a concentrate of lead and zinc, followed by the lead and zinc separation [7]. Apart from concentration of lead and zinc by flotation, there are a few literatures that reports concentrations of lead and zinc by gravity and hydrometallurgical methods with remarkable success [8]-[13].

The Balya Lead Mines are situated in the Western part of Turkey. These mines are very possible that have been worked since ancient times as they are situated near Troy. The oldest lead pieces found in Troy are thought to be originated from Balya lead mines. But regular operations with the application of scientific and modern mining methods were started about 1880s [14].

Balya lead mine is a (dacite-limestone) contact / metasomatic type ore deposit. The deposits of Balya is occurred mainly in vein type and disseminated type ores. The combined lead and zinc grade are reported as high as 30% in vein type ore while that of disseminated type ore is around 1% to 4%. The sulfide minerals mainly consist of galena, sphalerite, pyrite, and chalcopyrite [15]. In mine, 2400 tons of ore is processed at the mineral processing plant daily producing 366 tons of Pb and Zn concentrate [16].

In this study, flotation studies were carried out to obtain a lead rich concentrate with highest lead recovery. In flotation, there are many variables that affect flotation performance, i.e. collector type and amount, frother type and amount, particle size, pH, flotation time, solids ratio and depressant amount. These are called independent variables-parameters. Among these, the collector type and amount, frother type and amount, particle size, pH, flotation time were kept constant, while the solids ratio and the depressant amount were changed in this work. The solid ratio, depressant amount and their interactions which are the effective parameters on the Pb grade of concentrate have been described with mathematical models by using Central Composite Design (CCD) method in the flotation of low-grade lead and zinc ore. CCD is one of the response surface methods that allows to obtain more data with limited number of experiments [17]. The optimum conditions of solid ratio and depressant amount to obtain the highest Pb grade and highest Pb recovery have been determined.

II. MATERIALS AND METHODS

A. Sample

Samples were kindly supplied by Balya Lead and Zinc Concentration Company from the disseminated type ores. Ore is comminuted in the crashing and grinding plant in Balya to minus 0.200 mm before feeding to flotation cells. Studied samples were taken from the exit of the ball mill at the end of the grinding circuit. The predominant minerals of ore are reported as

galena, sphalerite and chalcopyrite while quartz, calcite, pyrite and jips are the main gangue minerals [9], [15]. The Pb, Zn and Fe contents of sample are analyzed as 0.83%, 0.60% and 11.15%, respectively.The flotation reactives used in the flotation experiments were obtained from the Balya Lead and Zinc Concentration Company Laboratories. Ethyl Xanthate and Methyl Isobutyl Carbinol (MIBC) were used as collector and frother, respectively. $ZnSO_4$ and $FeSO_4$ were used as depressants for sphalerite and pyrite, respectively. Although NaCN is not used in the plant but It was used in this study as an additional depressant to improve the depression of sphalerite and pyrite and obtained from Eskisehir Osmangazi University, Mining Engineering Department, Mineral Processing Laboratories.

B. Method

Flotation experiments were carried out by using laboratory type Denver Floatation cell with a volume of 1.5 liter cell at the Mineral Processing Laboratory of Mining Engineering Department of Eskisehir Osmangazi University. In the flotation experiments, particle size, pH, collector amount, frother amount, collector conditioning time and flotation duration were kept constant at -0.200 mm, 9.5, 25 g/t, 17.5 g/t, 12.5 min and 30 seconds, respectively. The dosages of $ZnSO_4$ and $FeSO_4$ used as depressants for sphalerite and pyrite were 750 g/t and 300 g/t, respectively. These conditions were advised by the Balya Lead and Zinc Concentration Company Laboratory technicians. In the experiments, solid ratio and the additional depressant (NaCN) dosage were tested and the optimum conditions were determined. After each flotation experiments, Pb content of floatation products was analyzed and Pb recovery (R_{Pb}) calculated from the equation (1) as given below:

$$R_{Pb} = 100*(c*C)/(f*F) \quad (1)$$

where C is the amount of concentrate (%), c is the Pb grade of concentrate (%), F is the amount of feed (%) and f is the Pb grade of feed (%).

Statistical design of the experiments was performed by using Design Expert version 11.0 software. The value of α used in the calculation of axial points was taken as 1.00 (face centered). Before the systematic experiments, preliminary experiments were carried out to determine the range of parameters. In these tests, minimum (-) and maximum (+) levels for solid ratio and depressant dosage were determined as 10–40% and 0–75 g/t, respectively. Independent variables-parameters and their levels are given in Table 1. 13 experiments were carried out randomly, to determine the effects of two parameters on response variables. Variance analysis for both response variables were performed, and regression models were created in 95% confidence level.

Parameter	Actual levels		
	Low (-1)	Center (0)	High (+1)
A- Solid Ratio (%)	10	25	40
B- Depressant amount (g)	0	37.5	75

Table 1–Parameters and their levels

III. RESULTS AND DISCUSSION

According to the design, planned by software, 13 experiments were carried out randomly. Table 2 shows the design matrix with coded values and the results of Pb contents and Pb recoveries of concentrates. As can be seen from Table 2, the highest Pb content is obtained as 47.25% at high level of dispersant dosage. At this point Pb recovery is 79.08%. Significant increase in Pb content was observed with a remarkable Pb recovery. This attributed to the usage of NaCN as a depressant for of gangue minerals such as sphalerite and pyrite.

Number of Experiment	A	B	Pb (%)	R_{Pb} (%)
1	1	1	45.69	64.01
2	0	0	38.86	74.96
3	0	0	36.22	80.19
4	-1	0	39.17	75.56
5	0	-1	23.15	47.53
6	-1	-1	21.18	56.17
7	0	1	47.25	79.08

8	-1	1	38.54	66.45
9	0	0	41.37	79.38
10	1	0	38.57	69.34
11	1	-1	19.45	42.19
12	0	0	39.79	77.84
13	0	0	37.22	76.38

Table 2–Design matrix with coded factors and results of experiments

Variance analysis (ANOVA) for Pb content and Pb recovery (response variables) according to the results of the experiments were prepared, and regression models were created.

The results of ANOVA of Pb content are given in Table 3. Model coefficients, p values, regression coefficient values (R^2) and predicted R^2 ($P\text{-}R^2$) values are also given in the same Table. As a result of this variance analysis, the regression model for Pb content, created according to the coded values of the terms, is given in equation (2).

$$Pb(\%)=39.27+0.80*A+11.28*B+2.22*AB-1.83*A^2-5.50*B^2 \quad (2)$$

This model is found to be statistically significant as its p-value (<0.0001) is less than 0.05. It is clearly seen form Table 3 that experimental error is insignificant as the p-value of lack of fit (0.3680) is much greater than 0.05. Predicted results versus actual results are given in Fig. 1 graphically. As can be seen from this figure, predicted Pb content values represent 96.43% of actual values.

Source	Sum of squares	P value	Model coefficients
Model	920.91	<0.0001 (Significant)	39.27
A (Solid Ratio; %)	3.87	0.4019	0.80
B (Depressant Amount; g/t)	763.88	<0.0001	11.28
AB	19.71	0.0840	2.22
A^2	9.27	0.2100	-1.83
B^2	83.60	0.0043	-5.50
Lack of Fit	17.37	0.3680 (not Significant)	
R^2		0.9643	
$P\text{-}R^2$		0.8314	

Table 3–ANOVA for Pb content

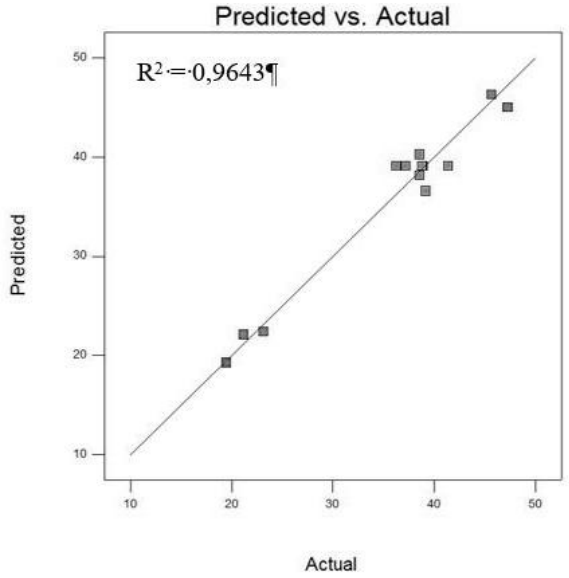

Fig. 1– Relationship between actual and predicted Pb contents of concentrate.

The most effective parameter for Pb content is depressant dosage (B). Both linear and quadratic effects of depressant dosage are found to be significant as p values are less than 0.05 for both B and B^2. (Table 3). Solid ratio (A) is not an effective parameter for Pb content. Both linear and quadratic effects are insignificant as p values greater than 0.05 for both A and A^2 (Table 3). It can be clearly seen from Table 3

that the interaction of solid ratio and depressant dosage (AB) is insignificant as p-value (0.0840) is well above 0.05. The response surface of this interaction is given in Fig. 2. As can be seen from this figure, Pb content of concentrate did not change significantly with changing solid ratio at low depressant dosage. Although Pb content of concentrate is increased slightly at high depressant dosage with increasing solid ratio, this increase did not indicate a significant interaction between solid ratio and depressant dosage.

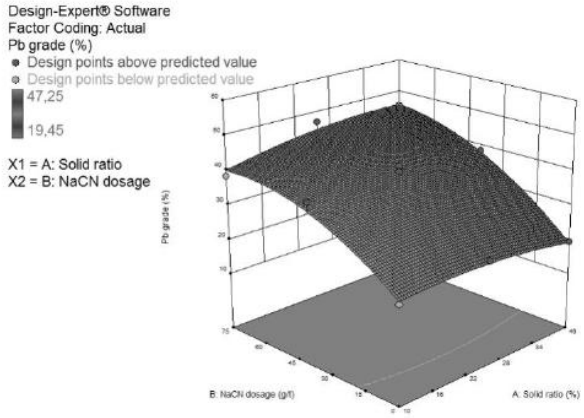

Fig. 2– The response surface of double interaction between solid ratio (A) and depressant dosage (B)

The results of ANOVA of Pb recovery are given in Table 4. Model coefficients, p values, R^2 and P-R^2 values are also given in the same Table. As a result of this variance analysis, the regression model for Pb recovery, calculated according to the coded values of the terms, is given in equation (3).

$R_{Pb}(\%)=77.86-3.77*A+10.61*B+2.88*AB-5.69xA^2-14.83*B^2$

This model is found significant as its p-value (0.0002) is less than 0.05. Predicted Pb recovery values versus actual Pb recovery values are given in Fig. 3 graphically. As can be seen from this figure, predicted R_{Pb} values represents 94.78% of actual R_{Pb} values. It is clearly seen form Table 4 that experimental error is insignificant as the p-value of lack of fit (0.0586) is greater than 0.05.

Source	Sum of squares	P value	Model coefficients
Model	1816.65	0.0002 (Significant)	77.86
A (Solid Ratio; %)	85.43	0.0445	-3.77
B (Depressant Amount; g/t)	675.22	0.0002	10.61
AB	33.29	0.1709	2.88
A^2	89.30	0.0411	-5.69
B^2	607.52	0.0003	-14.83
Lack of Fit	81.84	0.0586 (not Significant)	
R^2		0.9478	
P-R^2		0.5521	

Table 4–ANOVA for Pb recovery (R_{Pb})

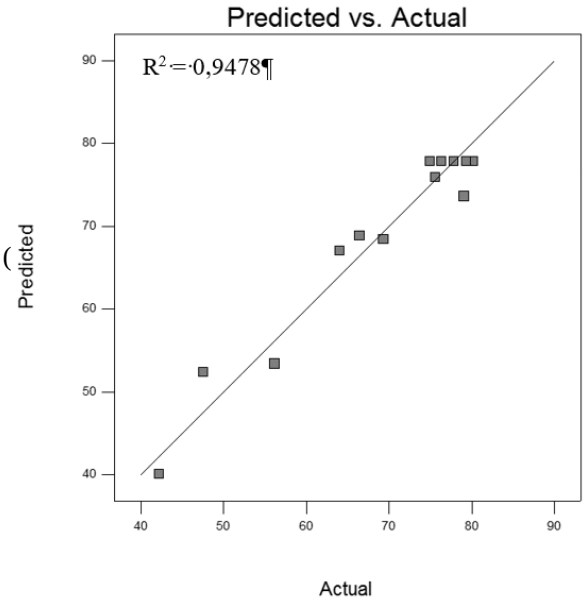

Fig. 3– Relationship between experimental and predicted Pb recovery

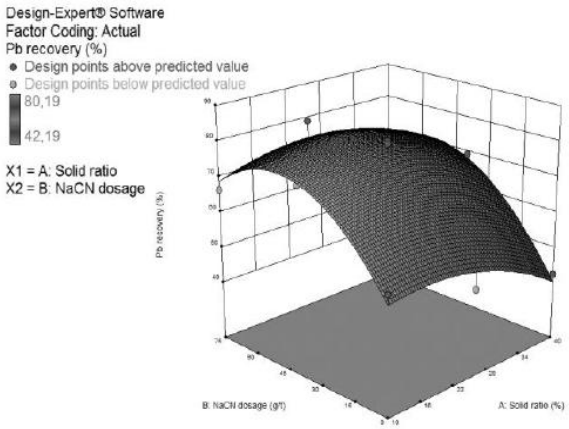

Fig. 4– The response surface of double interaction between solid ratio (A) and depressant dosage (B)

Both linear and quadratic effects of solid ratio and depressant dosage are found to be significant as p values are less than 0,05 for both parameters (Table 4). It can be clearly seen from Table 4 that the interaction of solid ratio and depressant dosage (AB) is insignificant as p-value (0.1709) is much greater than 0.05. The response surface of this interaction is given in Fig. 4. As can be seen from Fig. 4 that R_{Pb} is decreased slightly with increasing solid ratio at low depressant dosage. R_{Pb} is increased at the beginning but decreased afterwards with changing solid ratio showing a parabolic curve at high depressant dosage, this variation did not indicate a significant interaction between solid ratio and depressant dosage.

IV. CONCLUSION

The experimental results revealed that the Pb content of concentrate was obtained as 47.25% with 79.08% Pb recovery under the conditions of solid ratio of 25% and depressant dosage of 75 g/t. Maximum Pb recovery was obtained as 80.19% with 36.22% Pb content under the conditions of solid ratio of 25% and depressant dosage of 37.5 g/t. Solid ratio and depressant dosage that thought be effective parameters on the Pb grade and Pb recovery and their interactions are described with a mathematical model by using Central Composite Design method (CCD). The variance analysis has shown that both models obtained for Pb grade and Pb recovery were statistically significant. It has also shown that the effect of depressant dosage is higher than that of solid ratio. The graphics that present the actual values vs predicted values for two response variables, show that the regression coefficients are 0.9643 and 0.9478 for models created for solid ratio and depressant dosage, respectively. The high values of R-square (R^2) indicate that the models developed for two response variables are powerful.

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