Three Factors at Three Levels Factorial Experimental Modelling Of Lead Recovery Of Baban Tsauni (Nigeria) Lead-Gold Ore

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ABSTRACT
An empirical model was developed for froth flotation treatment of Baban Tsauni lead ore. Beneficiation of run-off mines is always necessary in order to reduce metal extraction cost. Metal recovery in froth flotation is a function of particle size, collector dosage and conditioning environment of the cell and this work investigated the effects of these parameters. This work applied a factorial experimental design for three factors at three levels. It adopted Box Behnken design of experiment. The empirical model developed established a mathematical relationship between lead recovery, particle size, collector dosage and alkalinity of the cell. Sodium ethyl xanthate was used as collector, solution of lime as pH regulator and pine oil as frother. Analysis of the model revealed that the level of deviation of the predicted lead recoveries from actual values range from -0.011% to 6.87%. The study established that particle size for optimum lead recovery was 144.74µm, at a collector dosage of 0.0436g/tonne and pH value of 8.112.

Keywords: Froth flotation, metal recovery, concentration, experimental design, empirical model.

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1. INTRODUCTION

Energy consumption per tonne of metal produced from its ore depends on the grade of feed (per cent concentration of valuable metal). This implies that feeds of higher grades cost less to extract (Wills and Napier-Munn, 2006). Moreover the cost of transportation decreases with increase in ore grade (Egbe, et al, 2013). Beneficiation is the process by which the concentration of the valuable constituent in an ore is increased while impurities are reduced to practically acceptable levels at economic recovery. The X-Ray fluorescence (XRF) test is often used for determination of the elemental composition (assay or grade) of raw ore, concentrates and tailings (Wills and Napier-Munn, 2006; Egbe et al., 2013; Muriana et al., 2014). Egbe, et al, (2013) indicated that gravity separation of Baban Tsauni lead-Gold ore, yielded lead recovery of 95.76% at a grade of 55%. This implies that further treatment of the concentrate by froth flotation is required in order to meet specifications for lead metal extraction. The focus of this research is to develop an empirical model for the response of the ore to froth flotation. Flotation parameters of interest are particle size (x_1), collector dosage (x_2) and cell alkalinity in term of pH value (x_3), while the measured response was lead recovery (y). Wills and Napier-Munn, (2006) indicated that recovery is calculated by applying Equation (1),

$$R = 100\frac{Cc}{Ff} \qquad (1)$$

where R= recovery, C= mass of concentrate, c = assay of concentrate, F= mass of feed and f = assay of feed sample.

Devor *et al.* (2007) stated that the objective, in empirical model, is to experiment with various combinations of the important design parameters for the purpose of identifying the particular combination that optimize performance measures. A well structured factorial experimental design makes it possible for the effects of several variables on the performance of a system to be determined with a few experimental runs and a line of action for product or process improvement is identified. The usual practice is to use the data generated from the designed experiment to develop a modelling equation which is subjected to significance test and analysis of variance to determine quality of the model before validation and optimization (Devor *et al*., 2007; Montgomery and Runger, 2003; Ross, 2004; Chandra, 2001).

Attainment of a local optimum requires a minimum of a quadratic modelling equation which is only possible with experiments conducted at three levels. A full three factors at three levels experiment requires twenty seven runs, 3^3 (Montgomery and Runger, 2003). Mee (2007) generated a fractional design of Box Behnken in order to further reduce the number of the test runs required. Box Behnkem design of experiment was adopted in this work.

2. MATERIAL AND METHOD

2.1 Material Preparation

Eighty kilograms (80kg) of ore samples collected from four mining pits at Baban Tsauni, Gwagwalada, Nigeria, were subjected to initial preparation involving mixing, crushing, grinding and sieving. Each set of close sized particles obtained from sieving operation were passed through Jones riffling sampler to get homogenous samples for all experimental runs.

2.2 Experimental Design Method

Fifteen runs were used in line with Box Behnken design of experiment. Details of experimental runs and random order of experimentation are presented in Table 1 while Table 2 presents parameter design for this research.

Table 1: Experimental test run conditions of the 3^{3^*} factorial design for froth flotation.

Test run	Particle size X_1	Collector X_2	pH X_3	Response Lead recovery y	Run order
A_1	-1	-1	0	Y_1	1
A_2	1	-1	0	Y_2	3
A_3	-1	1	0	Y_3	2
A_4	1	1	0	Y_4	5
A_5	-1	0	-1	Y_5	4
A_6	1	0	-1	Y_6	12
A_7	-1	0	1	Y_7	8
A_8	1	0	1	Y_8	13
A_9	-0.02035	-1	-1	Y_9	11
A_{10}	-0.02035	1	-1	Y_{10}	6
A_{11}	-0.02035	-1	1	Y_{11}	10
A_{12}	-0.02035	0	0	Y_{12}	14
A_{13}	-0.02035	0	0	Y_{13}	9
A_{14}	-0.02035	0	0	Y_{14}	7
A_{15}	-0.02035	0	0	Y_{15}	15

Table 2: The coded and actual values of independent variables for froth flotation test runs.

Coded values (X_k)	x_1= Particle size (microns)	x_2= Collector concentration (ml)	x_3= pH Value
-1	67.1 (-90+50)	1	7.0
0	138.13 (-180+106)	2	8.5
+1	212.132 (-250+180)	3	10.0

The relationship between coded values, X_k, and the actual values, x_k (Table 2) is,

$$X_k = 2\frac{(x_k - \bar{x}_k)}{(x_{kh} - x_{kl})} \qquad (2)$$

where X_k = coded value of the k^{th} factor (where k= 1, 2 and 3), x_k = actual value of the factor, x_{kh} = high value of x_k, x_{kl} = low value of x_k and $\bar{x}_k$ = arithmetic mean value of x_k. The sieve size required to produce a coded average of zero was not available. The nearest particle size with a coded value of -0.02035 was used as the intermediate level for particle size. The experiments were performed in random order so as to avoid structured error. The test conditions of the three factors (X_1, X_2, and X_3) for the validation test runs are presented in Table 3.

Table 3 Test conditions for validation test runs of froth flotation model.

Test Run	Particle size x1	Collector x2	pH x3
V_1	-0.59	0.8	0
V_2	-1.189	0.8	0
V_3	-1.189	0.8	0
V_4	-1.189	0.8	0
V_5	-1	0.8	0
V_6	-1	0.8	0
V_7	*-1*	*-1*	*0*

2.3 Experimental Method

A pulp of 31% solid by weight of ore corresponding to particle size x_1 (Table 1 and 2) and water was used for all flotation runs. Amount of collector (sodium ethyl xanthate) corresponding to x_2 was added to the pulp and the machine started with the air valve locked. After a minute the machine was stopped and the pulp pH was checked with a pocket pH meter. A solution of lime or sulphuric acid was added with the help of pipettes until a pH value corresponding to x_3 was attained (Table 1 and 2). Conditioning of the pulp continued for another 5minutes before 0.8ml of frother (pine oil) was added, followed by release of air into the cell. A scraper was used to collect the froth into trays.

When froth production ceased, the float (concentrates) and the residue (tailings) were washed and allowed to settle for about 20minutes. They were then decanted and filtered before drying to constant weight in an oven at a temperature of 110^{o}C. Finished products were weighed before samples were taken for assay analysis by XRF.

3. RESULTS AND DISCUSSION

The summary of lead recoveries for all test runs needed for developing the modelling equation is presented in Table 4.

Table 4: Lead recovery for each unique test

Test run	Feed weight (F)	Feed assay(f)	Weight of conc. (C)	Conc. assay (%)	Lead Recovery(%)
A_1	400	34.3212	118.8	66.04512	57.1524324
A_2	400	43.68996	127.6	77.73288	56.7562633
A_3	400	34.321	119.3	70.31208	61.101302
A_4	400	43.68996	134.8	65.11752	50.2280255
A_5	400	34.321	113.1	67.90032	55.9389746
A_6	400	43.68996	132.2	66.50892	50.3117834
A_7	400	34.321	148.8	59.18088	64.1452387
A_8	400	43.68996	129.7	65.30304	48.4653928
A_9	400	36.36192	85	68.45688	40.0063776
A_{10}	400	36.362	102.7	57.882	40.87
A_{11}	400	36.362	99.5	51.296	35.092
A_{12}	400	36.36192	108.5	52.78044	39.373
A_{13}	400	36.36192	102.6	58.06776	40.961
A_{14}	400	36.36192	99	58.25328	39.651
A_{15}	400	36.36192	103.2	56.3726	39.995

Note: (1) The mean lead recovery for the replicated test runs (A_{12}, A_{13}…A_{15}) = 40.0%.

The model equation is given by:

$$Y = B_0 + B_1 X_1 + B_2 X_2 + B_3 X_3 + B_{12} X_1 X_2 + B_{13} X_1 X_3 + B_{23} X_2 X_3 + B_{11} X_1^2 + B_{22} X_2^2 + B_{33} X_3^2 \quad (3)$$

where X_1, X_2, and X_3 are coded particle size, amount of collector and pH value respectively and B_0, B_1,……B_{33} were the unknown coefficients which were determined by using the responses from fifteen test runs (Table 5). The matrix form of Equation (3) was used with the calculation matrix presented in Table 6 on a MATLAB programme to calculate the coefficients of the modelling equation.

Table 6: Calculation matrix for froth flotation models

	B0	x_1	X_2	x_3	x_1x_2	x_1x_3	x_2x_3	x_1^2	x_2^2	x_3^2	Recovery
A_1	1	-1	-1	0	1	0	0	1	1	0	57.152
A_2	1	1	-1	0	-1	0	0	1	1	0	56.756
A_3	1	-1	1	0	-1	0	0	1	1	0	61.101
A_4	1	1	1	0	1	0	0	1	1	0	50.228
A_5	1	-1	0	-1	0	1	0	1	0	1	55.939
A_6	1	1	0	-1	0	-1	0	1	0	1	50.312
A_7	1	-1	0	1	0	-1	0	1	0	1	64.145
A_8	1	1	0	1	0	1	0	1	0	1	48.465
A_9	1	-0.02035	-1	-1	.02035	0.02035	1	.000414	1	1	40.006
A_{10}	1	-0.02035	1	-1	-.02035	0.02035	-1	.000414	1	1	40.87
A_{11}	1	-0.02035	-1	1	0.02035	-0.02035	-1	.000414	1	1	35.092
B_{av}	1	-0.02035	0	0	0	0	0	.000414	0	0	39.995

The variance of the replicated tests in respect of lead recovery (S^2_{Br}) became:

$S_{Br}^2 = (u_{Br1}-ū_{Br})^2/(n-1) + (u_{Br2}-ū_{Br})^2/(n-1) + (u_{Br3}-ū_{Br})^2/(n-1) +(u_{Br3}-ū_{Br})^2/(n-1)$ (4)

where u_{Bri} = individual responses in B_{ri} for i = 1, 2, 3 and 4, $ū_1$ = mean of the responses in B_{ri} and n = 4= number of replicates.

Substituting the responses yielded:
$S_{Br}^2 = (0.393124+0.923521+.121801+0.000025)/(4-1) = (1.438476)/3 = 0.479492$

The pooled variance (S_p^2) was the mean of all variance from replicated test runs. Thus,
$S_p^2 = S_{Br}^2 = 0.479492$

The sample variance of an effect was calculated by applying Equation (5),

$$S_E^2 = 4\frac{(S_p)^2}{N} \quad (5)$$

where N = the total number of test runs used for the model = 15.

Substituting yields,
$S_E^2 = 4(0.479492)/15 = 0.12786$

The standard error (s.e) of the effects is the square root of the sample variance and hence,
s.e = √(0.12786) = 0.35758

Sample variance of the average (S_{av}^2) is given by:

$$S_{av}^2 = \frac{(S_p)^2}{N} \quad (6)$$

= 0.479492/15 = 0.031966.

Therefore s_{av} = √(0.031966) = 0.17879

The calculated t-values were compared with the standard statistical t-value at a confidence level of 95% to determine the main effects and interaction effects that were significant. The coefficients and effect estimates calculated for lead recovery modelling are presented in Table 7. This table also presents the associated t-values for each estimated effect and the statistical t-value.

Table 7: Coefficients, effect estimates and associated t-values for lead recovery.

Coefficients	*Values*	*Effects*	*Effect estimates*	*Calculated t-values*	$t_{3,0.975}$
B_0	39.9056	E_0	39.9056	223.19817	3.182
B_1	-4.072	E_1	-8.144	-22.77532	3.182
B_2	0.6353	E_2	1.2706	3.5533307	3.182
B_3	0.3097	E_3	0.6194	1.7321998	3.182
B_{12}	-2.6453	E_{12}	-5.2906	-14.79557	3.182
B_{13}	-2.4872	E_{13}	-4.9744	-13.91129	3.182
B_{23}	1.5372	E_{23}	3.0744	8.5977963	3.182
B_{11}	15.8451	E_{11}	31.6902	88.624084	3.182
B_{22}	0.5586	E_{22}	1.1172	3.1243358	3.182
B_{33}	-1.0354	E_{33}	-2.0708	-5.791152	3.182

The associated t-values (Table 7) indicated that the effect estimates E_3 and E_{22} were not significant, at a confidence level of 95%, since their absolute values were less than $t_{2,0.975}$. This implied that their contributions to the model were negligible within the range of values used in the experiment. It also indicated that their mean effects were zero and hence their corresponding coefficients could not be included in the modelling equation for predicting future lead recovery by froth flotation of this ore.

The cumulative normal probability of the effect estimates for lead recovery is presented in Table 8 while the plot is shown in Figure 1.

Table 8: The cumulative normal probability of estimated effects of lead recovery

Effects	*Effect values*	*Normal probability*
E_1	-8.144	0.0556
E_{12}	-5.2906	0.1667
E_{13}	-4.9744	0.2778
E_{33}	-2.0708	0.388
E_3	0.6194	0.5
E_{22}	1.1172	0.6111
E_2	1.2706	0.7222
E_{23}	3.0744	0.8333
E_{11}	31.6902	0.94444

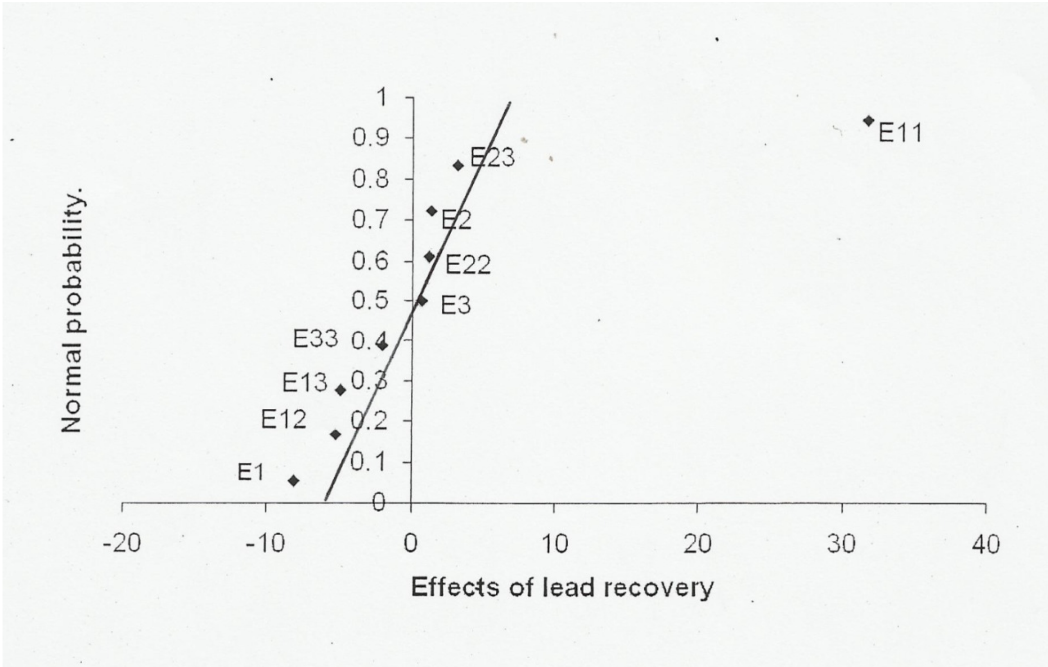

Figure 1: The normal probability plot for the effects of lead recovery

A line drawn near 0,0.5 point passed through E_3 and E_{22}. This again implied that these effect estimates were not significant and could be left out without considerable effect on the model. Thus the adopted model Equation 6 was modified to Equation 7.

$$Y_R' = 39.91 - 4.07X_1 + 0.64X_2 - 2.65X_1X_2 - 2.49X_1X_3 + 1.54X_2X_3 + 15.85X_1^2 - 1.04X_3^2 \quad 7$$

3.1 Model Analysis

The model was subjected to residual analysis to detect the presence of structured error. The residuals of the model for lead recoveries are shown in Table 9 and the last column presents the residuals as percentages of the actual recoveries and Figure 2 shows a cumulative normal probability plot of the model residuals.

Table 9: Lead recoveries and residuals for factorial froth flotation.

Test run	Particle size X_1	Collector X_2	pH X_3	Actual lead recovery y	Model lead recovery YR	Model lead residual	Per cent residuals
A_1	-1	-1	0	57.1524	56.535	0.6174	1.08027
A_2	1	-1	0	56.7563	53.695	3.0613	5.39376
A_3	-1	1	0	61.1013	63.115	-2.0137	-3.29567
A_4	1	1	0	50.228	49.675	0.553	1.10098
A_5	-1	0	-1	55.939	56.295	-0.356	-0.63641
A_6	1	0	-1	50.3118	53.135	-2.8232	-5.61141
A_7	-1	0	1	64.1452	61.275	2.8702	4.47454
A_8	1	0	1	48.4654	48.155	0.3104	0.64046
A_9	-0.02035	-1	-1	40.0064	39.7548	0.2516	0.6289
A_{10}	-0.02035	1	-1	40.87	38.0626	2.8074	6.8691
A_{11}	-0.02035	-1	1	35.092	36.7761	-1.6841	-4.7991
A_{12}	-0.02035	0	0	39.373	39.9994	-0.6264	-1.59094
A_{13}	-0.02035	0	0	40.961	39.9994	0.9616	2.3476
A_{14}	-0.02035	0	0	39.651	39.9994	-0.3484	-0.87867
A_{15}	-0.02035	0	0	39.995	39.9994	-0.0044	-0.011

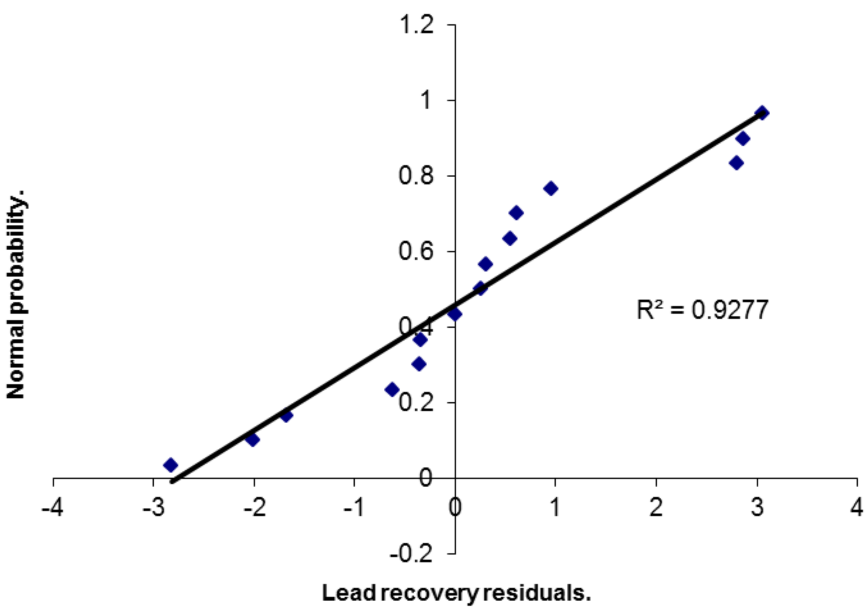

Figure 2: Normal probability plots of the model residuals for lead recovery.

The plot showed that the residuals have a mean centre of zero as expected if there were no structured errors (Montgomery and Runger, 2004; Devor, *et al.*, 2007). The level of deviation of the predicted lead recoveries from the actual values range from -0.011% to 6.87%. Only three values out of fifteen were higher than 5% and this was considered acceptable.

Thella and Venugopal (2011) indicated that the plot of the predicted values against the observed values must produce a linear relationship. Figure 3 presents a plot of predicted lead recovery against observed values and it showed a linear pattern as expected (Thella and Venugopal, 2011).

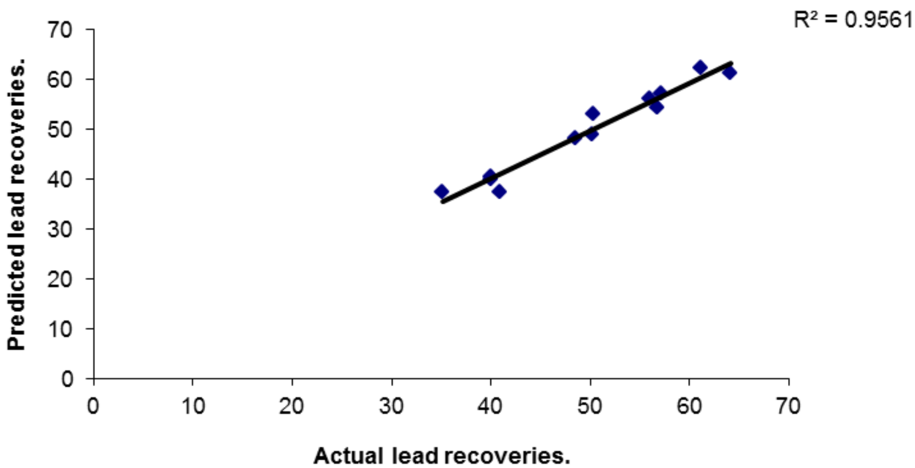

Figure 3: Plot showing predicted and observed values of lead recoveries.

The absence of structured error implied that the model equation is adequate for prediction of future results and particularly so when the conditions are within the range of values in the experiment.

The final modelling equation indicated that particle size smaller than the mean value of 106μm enhanced lead recovery and the interacting effects required that X2 and X3 should be positive for optimum recovery. Equation (7) indicates that for negative value of X1, a negative value of X3 (that is, pH values lower than 8.5) would only increase lead recovery when collector (xanthate) level is negative. This implied that at low pH the amount of xanthate had to be minimized in order to achieve good recovery. Moreover the last term of Equation (7) requires that increase in the absolute value of X3 impaired recovery. This deduction agreed with the common practice of operating sulphide ore at pH of about 8.5 (Bulatovic, 2010; Gupta, 2003; Wills and Napier-Munn, 2006).

3.2 Optimization.

Partial differentials of Equation (7) with respect to X1, X2 and X3 were obtained in order to determine the optimum conditions of the factors which enhance lead recovery. The partial differentials were equated to zero and solved to get the respective values of X1, X2 and X3. This gave optimum values of X1= 0.0912 (particle size x1= 144.74μm), X2 = -0.2019 (xanthate additon x2 = 1.798ml) and X3 = -0.2587 (pH value x3 = 8.112). The factors were tested in turn in the vicinity of the optimum values to determine if they were minima or maxima values. Figure 4 showed that the particle size of 144.74μm was the upper limit of particle size for enhanced recovery while Figure 5 showed that a pH of 8.112 was the upper limit for enhanced recovery when the amount of xanthate remained constant at 1.798ml (or dosage of 0.0436g/tonne). The foregoing observations indicated that when xanthate concentration was constant, lead recovery increased with decrease in particle size provided the pH in the flotation cell remained at the optimum level.

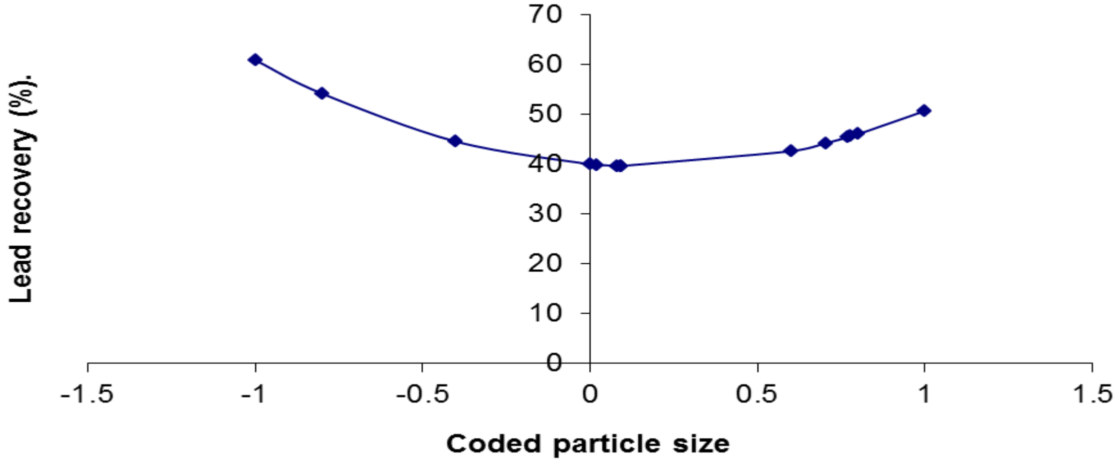

Figure 4: Lead recoveries as function of particle size when collector and pH remained at optimum values.

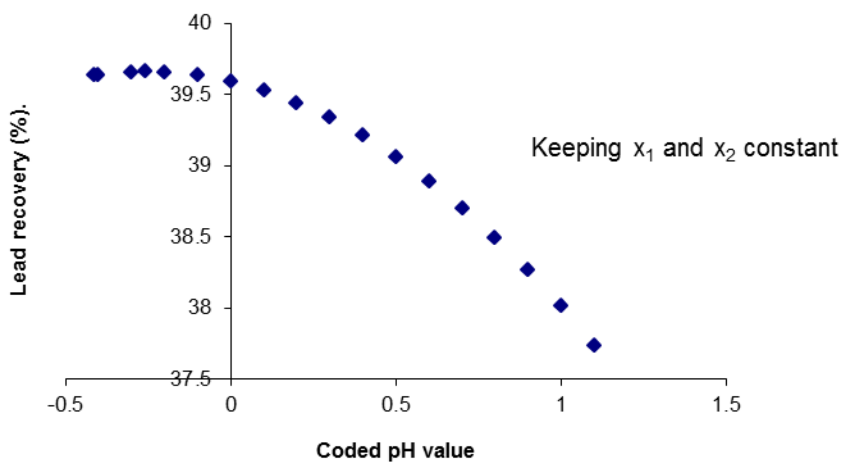

Figure 5: Lead recoveries as function of pH when particle size and collector remained at optimum values.

3.5 Validation/Optimum Test Results

The summaries of lead recoveries obtained from validation test runs that were carried out under optimum conditions are presented in Table 10 and lead recovery residuals are presented in Tables 11.

Table 10: Summary of lead recoveries from validation test runs

Test run	Feed weight (F)	Feed assay(f)	Weight of conc. (C)	Conc. assay (C)	Lead Recovery(%)
V_1	400	32.095	122.4	54.27	51.74208
V_2	400	31.5384	151.23	61.78	74.06043
V_3	400	31.5384	154.2	54.914	67.12245
V_4	400	31.5384	138.14	68.364	74.85956
V_5	400	32.1877	140.6	58.07	63.4143
V_6	400	66.51	252.3	75.1356	71.25512
V_7	147.7	51.776	45.5	71.24	42.38638

Table 11: Lead recovery residuals for validation tests of flotation model.

Test Run	Particle size x1	Collector x2	pH x3	Actual Recovery	Predicted YR	Recovery residual
V_1	-0.5899	0.8	0	51.742	49.587	2.155
V_2	-1.1888	0.8	0	74.060	70.174	3.887
V_3	-1.1888	0.8	0	67.122	70.174	-3.051
V_4	-1.1888	0.8	0	74.860	70.174	4.686
V_5	-1	0.8	0	63.414	62.457	0.957
V_6	-1	0.8	0	71.255	62.457	8.798
V_7	*-1*	*-1*	*0*	*42.386*	*56.535*	*-14.149*

The results presented in Tables 11 show close agreement between the predicted and the experimental values. The highest deviation in the five bulk flotation samples was 6.26% and the lowest was 1.51%. The recovery of the pre-concentrated samples showed remarkable positive deviation of 12.35% from the predicted value. The most likely reason being a more effective use of reagents due to prior reduction in the concentration of gangue minerals. Moreover the multi-gravity separation method used in preparing the sample removed contamination of particle surface by fine (dust) particles and thereby exposing clean mineral surfaces for more effective adsorption of reagents. The validation results have shown that the response of Baban Tsauni lead ore to froth flotation was adequately represented by the model Equation 7, in terms of lead recovery.

4. CONCLUSION

This research has developed an empirical model that predicts the lead recovery of Baban Tsauni lead ore for beneficiation of the ore by froth flotation method. It was established that the model is free of structured errors. The empirical model developed established a mathematical relationship between lead recovery, particle size, collector dosage and alkalinity of the cell. It established effectiveness of sodium ethyl xanthate as collector, solution of lime as pH regulator and pine oil as frother, for froth flotation treatment of the ore. Analysis of the model established that the level of deviation of the predicted lead recoveries from actual values range from -0.011% to 6.87%. The study established that particle size for optimum lead recovery was 144.74μm, at a collector dosage of 0.0436g/tonne and pH value of 8.112.

5. RECOMMENDATIONS

A good prediction of ore recovery under specified processing conditions will be of greater value in beneficiation when a model for prediction of metallic grade enrichment is available. It is recommended that an empirical model should be developed to predict metallic grade enrichment that is associated with predicted recovery.

One major challenge faced in this research was scarcity of froth flotation reagents. It is therefore recommended that development of flotation reagents be vigorously pursued in order to achieve self sustainance in solid mineral processing.

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