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Predictive Analysis of Dissolved Lead Based on Leaching Time and Reaction Temperature during Hydro-processing of Galena in Ferric Nitrate Solution

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Abstract

Predictive analysis of dissolved lead was carried out based on the leaching time and reaction temperature during hydro-processing of galena in ferric nitrate solution. The analysis was actualized using a two-factorial model derived from experimental results. The evaluated standard errors when the dissolved lead concentration for various values of reaction time as obtained from experiment and derived model-predicted results were subjected to statistical analysis were 0.3481 and 0 % respectively. Evaluation of generated results indicates that the rate of dissolution of lead as obtained from experiment and derived model-predicted results were 0.082 and 0.085 ppm/mins respectively. Results of deviational analysis indicate that the maximum deviation of the model-predicted dissolved lead relative to the experimental results is less than 8%. This implies a derived model confidence level above 87% as well as over 0.87 response coefficients for dissolved lead dependence on the leaching time and reaction temperature.

Keywords: Prediction, Dissolved lead, Galena, Ferric Nitrate, Leaching time, Reaction temperature.

1. Introduction

Enyimgba galena deposit is a part of a continuous PbS-ZnS ore deposit in Ebonyi state of Nigeria that stretches from Ameka to Enyimgba. Preliminary report [1] on the deposit shows that both sphalerite and galena occur in massive aggregates. Galena appears to be more abundant than blend in the eastern (Enyimgba) ore bodies of the field. The major problem associating conventional processing of this ore type is the inevitable generation of large amount of sulphur dioxide and sulphur trioxide gaseous pollutant which has become a source of concern to recent lead-zinc producers due to high demand for environment pollution processes [1-18].

Research [19] carried out on the sedimentation analysis of Ishiagu galena indicates that the average grain size of Ishiagu galena concentrate is approximately 100µm. The researcher also found that the mechanism of bioleaching of Ishiagu galena concentrates was indirect mechanism. This was sequel to the dominance of Fe^{3+} ions over H^{+} during the leaching process.

The possibility of extracting lead using microbes have been evaluated and found feasible [20]. The research reported that during bioleaching of Ishiagu lead-zinc ore, using mixed cultures of Acidithiobacillus Ferrooxidans, Acidithiobacillus Thiooxidans and Leptospirillum Ferrooxidans, higher silica contents of the ore reduce acidity, iron mobility and oxidation.

Researchers [21] have reported that Acidithiobacillus Ferrooxidans are able to oxidize ferrous ions and the reduced sulphur compounds while Acidithiobacillus Thiooxidans are able to oxide only reduced sulphur compound. Similar research [22] has proved that arsenic can be reduced in a complex galena concentrate by Acidithiobacillus Ferrooxidans. The results of the investigation reveal that arsenopyrite was totally oxidized. The sum of arsenic remaining in solution and removed by sampling represents from 22 to 33% in weight (yield) of the original content in the mineral. The rest of the biooxidized arsenic form amorphous compounds that precipitate galena (PbS) was totally oxidized too, anglesite ($PbSO_4$) formed is virtually insoluble and remains in the solids. The maximum rate of arsenic dissolution in the concentrate was found using the following levels of factors; small surface area of particle exposure; low pulp density, injecting air and adding the leaching medium to the system. It was also found that ferric chloride and carbon dioxide decreased the arsenic dissolution rate. Bioleaching kinetic data of arsenic solubilization were used to estimate the dilution rate for a continuous culture. Calculated dilution rates were relatively small (0.88 - 0.103day^{-1}) [22].

Report [23] has also shown that galena concentrate could be leached in ferric chloride brine. The results of the investigation reveal several advantages of ferric chloride over the reagents as a leaching media which includes that it exhibits substantially faster dissolution rates for most sulphides, it is regenerated easily by chlorination of ferrous chloride leaching by-products, and it has greater potential for the treatment of complex sulfides.

The aim of the present work is to carry out a predictive analysis of dissolved lead based on leaching time and reaction temperature during hydro-processing of galena in the ferric nitrate solution.

3. Materials and methods

The galena samples used in this study were collected from the deposit, at Enyimgba, Abakaliki, Ebonyi State. The galena which was in association with other minerals (valuable and gangue) was obtained in lumps of about 500 mm. These lumps were crushed and the galena cubes isolated from the gangue by careful hand picking. The isolated galena crystals were further crushed and a set of screen used to size them into fines 10 x 20 mesh. Based on the atomic absorption spectrometric analysis carried out, the samples used contain 86.55% Pb, indicating that the sample was essentially pure.

Ferric nitrate solution and the galena were kept in separate cylindrical flask and placed in the water bath to attain the desired temperature. Once the temperature was reached the leaching solution was transferred into the vessel containing the galena sample and stirring commenced. Also 0.1M $Fe(NO_3)_3$ was used at the temperature (50^oC) in the presence of 1.0M $NaNO_3$. In all the experiments, 0.5 gram each of galena was leached in 500 mls solution which is equivalent to 1 gram of galena in 1 litre of solution. A 5 mls sample each of solution was withdrawn at predetermined time intervals and filtered. Furthermore, 2 mls of this stock solution was further diluted to 100 mls and sampled for analysis.

Table 1: Variation of concentration of dissolved lead with leaching time and reaction temperature [24]

(γ)	(υ)	(ζ)
50	20	1.90
50	40	3.98
50	80	7.44
50	100	9.00
50	130	10.91

3.1 Model Formulation

Results generated from the experiment were used for the model formulation. Computational analysis of the results shown in Table 1, gave rise to Table 2 which indicate that;

$$\zeta - N\gamma \approx K + S \qquad (1)$$

Introducing the values of N, K and S into equation (2) reduces it to;

$$\zeta - 0.00012\gamma = 0.085\upsilon + 0.427 \qquad (2)$$

$$\zeta = 0.085\upsilon + 0.00012\gamma + 0.427 \qquad (3)$$

Where

(ζ) = Conc. of dissolved lead (ppm)
(υ) = Leaching time (mins)
(γ) = Reaction temperature (^0C)
N = 0.00012, K = 0.085, and S = 0.427. These are empirical constant (determined using C-NIKBRAN [25]

4. Boundary and Initial Condition

Galena was placed in cylindrical flask 30cm high containing leaching solution of ferric nitrate. The leaching solution is non flowing (stationary). Before the start of the leaching process, the flask was assumed to be initially free of attached bacteria and other micro organism. Initially, the effect of oxygen on the process was assumed to be atmospheric. In all cases, weight of lead used was 0.5g. The reaction temperature used was 50^{o}C. The reaction time range used 20 - 130 mins. Ferric nitrate concentration used was 0.1M.

The leaching process boundary conditions include: atmospheric level of oxygen (considering that the cylinder was open at the top) at both the top and bottom of the ore particles in the gas and liquid phases respectively. A zero gradient was assumed for the liquid scalar at the bottom of the particles and for the gas phase at the top of the particles. The sides of the particles were assumed to be symmetries.

5. Model Validation

Table 2: Variation of ζ - 0.00012ɤ with 0.085 υ + 0.427

ζ - 0.00012 γ	0.085 υ + 0.427
1.894	2.127
3.974	3.827
7.434	7.227
8.994	8.927
10.904	11.477

Equation (3) is the derived model. The validity of the model is strongly rooted on equation (2) where both sides of the equation are correspondingly approximately equal. Table 2 also agrees with equation (2) following the values of ξ - 0.00012ɤ with 0.085ϑ + 0.427 evaluated from the experimental results in Table 1.

Furthermore, the derived model was validated by comparing the concentration of dissolved lead predicted by the model and that obtained from the experiment. This was done using the 4^{th} Degree Model Validity Test Techniques (4^{th} DMVTT); statistical, graphical, computational and deviational analysis.

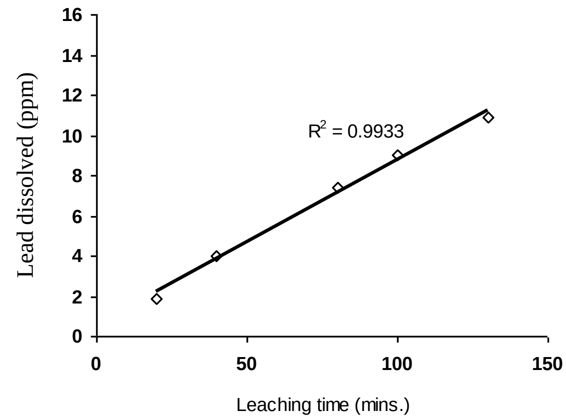

Fig.1: Coefficient of determination between dissolved lead concentration and leaching time as obtained from experiment

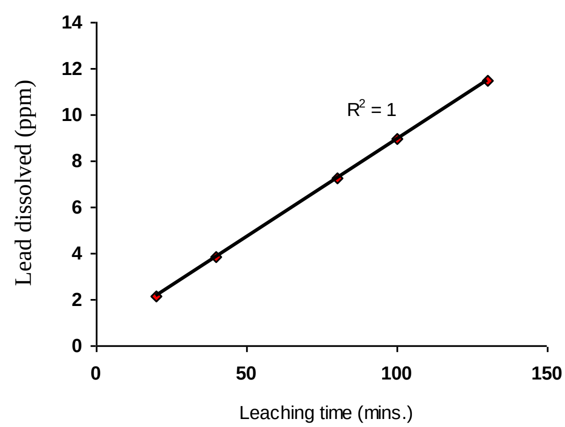

Fig.2: Coefficient of determination between dissolved lead concentration and leaching time as obtained from derived model

Statistical Analysis

Standard Error (STEYX)

The standard errors incurred in predicting the concentration of dissolved lead for each value of the leaching time considered as obtained from experiment and derived model were 0.3481 and 0 % respectively. The standard error was evaluated using Microsoft Excel version 2003.

Correlation (CORREL)

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The correlation coefficient between dissolved lead and leaching time were evaluated from the results of the derived model and experiment, considering the coefficient of determination R^2 from Figs. 2 and 3. The evaluation was done using Microsoft Excel version 2003.

$$R = \sqrt{R^2} \quad (4)$$

The evaluated correlations are shown in Table 3 These evaluated results indicate that the derived model predictions are significantly reliable and hence valid considering its proximate agreement with results from actual experiment.

Table 3: Comparison of the correlations evaluated from derived model predicted and ExD results based on leaching time

Analysis	Based on leaching time	
	ExD	D-Model
CORREL	0.9966	1.0000

Graphical Analysis

Comparative graphical analysis of Fig. 3 show very close alignment of the curves from the experimental (ExD) and model-predicted (MoD) dissolved lead.

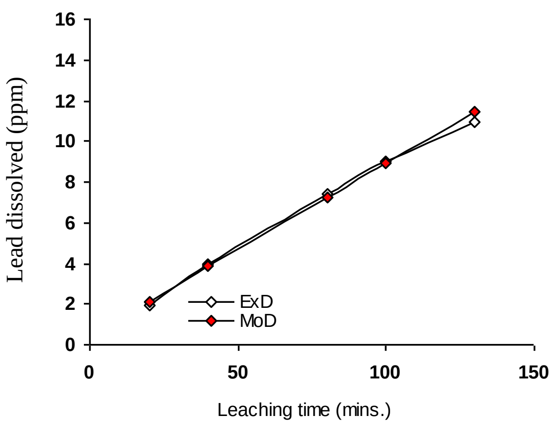

Fig.3: Comparison of dissolved lead concentrations (relative to leaching time) as obtained from experiment and derived model

Furthermore, the degree of alignment of these curves is indicative of the proximate agreement between both experimental and model-predicted dissolved lead.

Computational Analysis

Computational analysis of the experimental and model-predicted dissolved lead was carried out to ascertain the degree of validity of the derived model. This was done by comparing the concentration of dissolved lead per unit leaching time using experimental and model-predicted results.

Rate of dissolution of lead

The rate of dissolution of lead ζ_R was calculated from the expression;

$$\zeta_R = \frac{\Delta\zeta}{\Delta\upsilon} \quad (5)$$

Equation (6) is detailed as

$$\zeta_R = \frac{\zeta_2 - \zeta_1}{\upsilon_2 - \upsilon_1} \quad (6)$$

Where

$\Delta\zeta$ = Change in concentration of dissolved lead at two different leaching times υ_2, υ_1.

Considering the points (20, 1.9) & 130, 10.91), and (20, 2.133) & (130, 11.483) as shown in Figs 1 and 2, and designating them as ((ζ_1, υ_1) & (ζ_2, υ_2) for experimental and derived model predicted results respectively, and then substituting them into equation (6), gives the slopes: 0.082 and 0.085 ppm/mins. as rate of dissolution of lead respectively.

Deviational Analysis

The deviation Dv, of model-predicted dissolved lead from the corresponding experimental result was given by

$$Dv = \left[\frac{\zeta_{MoD} - \zeta_{ExD}}{\zeta_{ExD}}\right] \times 100 \quad (7)$$

Where

ζ_{MoD} and ζ_{ExD} are extracted lead concentration from experiment and derived model respectively.

Critical analysis of the concentration of dissolved lead obtained from experiment and derived model shows negative deviations on the part of the model-predicted values relative to values obtained from the experiment. This is attributed to the fact that the surface properties of galena and the physico-chemical interactions between the galena and the leaching solution which played vital roles during the leaching process were not considered during the model formulation. This necessitated the introduction of correction factor, to bring the model-predicted extracted lead concentration to those of the corresponding experimental values.

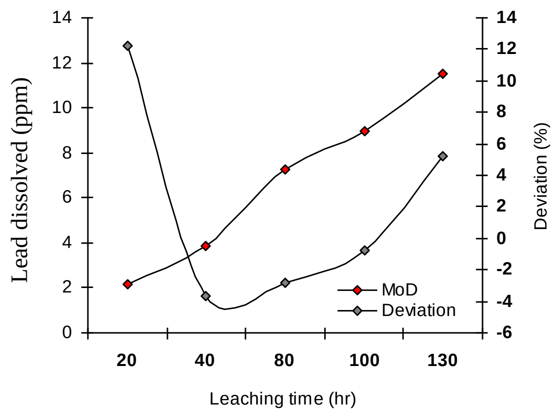

Fig.4: Variation of deviation with dissolved lead (relative to the leaching time)

Deviational analysis from Fig. 4 indicates that the maximum deviation of model-predicted dissolved lead from the experimental results is less than 13%. This translates into over 87% operational confidence and response level for the derived model as well as over 0.87 response coefficient of dissolved lead to the combined operational contributions of the leaching time and reaction temperature.

Consideration of equation (7) and critical analysis of Fig. 4 shows that the least and highest magnitudes of deviation of the model-predicted dissolved lead (from the corresponding experimental values) are – 0.74 and + 12.26. Figs. 1- 4 indicate that these deviations correspond to 8.933 and 2.133 ppm of dissolved lead as well as reaction times of 100 and 20 mins. respectively.

Correction factor, Cf to the model-predicted results is given by

$$Cf = -\left[\frac{\zeta_{MoD} - \zeta_{ExD}}{\zeta_{ExD}}\right] \times 100 \qquad (8)$$

Critical analysis of Figs. 1-5 indicates that the evaluated correction factors are negative of the deviation as shown in equations (7) and (8).

The correction factor took care of the negligence of operational contributions of the surface properties of the galena and the physico-chemical interactions between the galena and the leaching solution which actually played vital role during the leaching process. The model predicted results deviated from those of the experiment because these contributions were not considered during the model formulation. Introduction of the corresponding values of Cf from equation (8) into the model gives exactly the corresponding experimental values of dissolved lead.

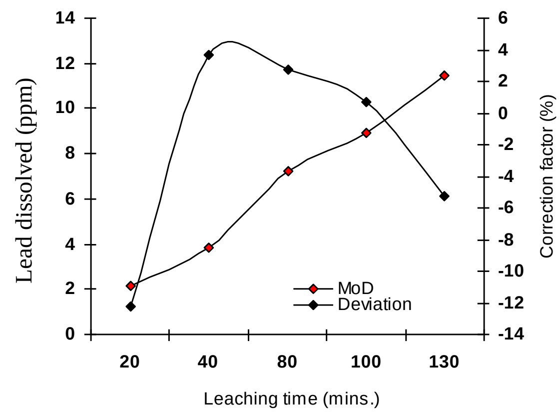

Fig.5: Variation of correction factor with dissolved lead concentration (relative to the leaching time)

Fig. 5 shows that the least and highest correction factor (to the model-predicted dissolved lead) are + 0.74 and – 12.26 %. Since correction factor is the negative of deviation as shown in equations (8) and (9), Figs. 1-5 indicate that these highlighted correction factors correspond to 8.933 and 2.133 ppm of dissolved lead as well as reaction times of 100 and 20 mins. respectively.

It is very pertinent to state that the deviation of model predicted results from that of the experiment is just the magnitude of the value. The associated sign preceding the value signifies that the deviation is a deficit (negative sign) or surplus (positive sign).

6. Conclusion

Predictive analysis of dissolved lead was carried out based on the leaching time and reaction temperature during hydro-processing of galena in ferric nitrate solution. A two-factorial model derived from experimental results was used as a tool for the analysis. The validity of the model was rooted on the expression $\zeta - 0.00012\gamma = 0.085\upsilon + 0.427$ where both sides of the expression are correspondingly approximately equal. Following statistical analysis of the concentration of dissolved lead for each value of the reaction time as obtained from experiment and derived model-predicted results, the evaluated standard errors were 0.3481 and 0 % respectively. Evaluation of generated results indicates that rate of dissolution of lead as obtained from experiment and derived model-predicted results were 0.082 and 0.085 ppm/mins. respectively. Results of deviational analysis indicate that the model-predicted dissolved lead shows a maximum deviation of less than 8% relative to the experimental results. This implies a derived model confidence level above 87% as well as over 0.87 response coefficients for dissolved lead dependence on the leaching time and reaction temperature.

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