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Implicit Assessment of Lead Extraction Based On Its As-Mined Content and Ferric Sulphate Concentration during Leaching In the Sulphate Solution

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Abstract-An implicit assessment of lead extraction was carried out based on its as-mined content in galena and ferric sulphate concentration during leaching in the sulphate solution. A two-factorial empirical model was derived following computational analysis of experimental results. The model was validated and used for the assessment. The validity of the model expressed as;

$$\xi = 3\upsilon + 0.0015\gamma + 0.3$$

was rooted on the expression $\xi - 0.0015\gamma = 3\upsilon + 0.3$ ***where both sides of the expression are correspondingly approximately equal. Evaluation of generated results indicates that for each value of the ferric sulphate concentration, the statistical analysis of the extracted concentration as obtained from experiment and derived model-predicted results show standard errors of 0.0266 and*** 3.04×10^{-9} ***% respectively. Extracted lead concentration per unit ferric sulphate concentration as obtained from experiment and derived model-predicted results were 3.35 and 3.0 ppm/ M respectively. Deviational analysis of model-predicted extracted lead concentration (from experimental result) was less than 11%. This invariably implies a derived model confidence level of above 89% as well as over 0.89 response coefficients for lead extraction dependence on its original content in the galena and ferric sulphate concentration.***

Keywords: Implicit assessment, Lead extraction, Galena Lead Content, Ferric sulphate solution.

I. INTRODUCTION

The vast usefulness of lead for industrial application stems on the ease at which it can be alloyed with many other metals and casts with little difficulty. Lead has good resistance to corrosion under a wide variety of conditions.

The properties of lead that make it useful in a wide variety of applications are density, malleability, lubricity, flexibility, electrical conductivity, and coefficient of thermal expansion, all of which are quite high; and elastic modulus, elastic limit, strength, hardness, and melting point, all of which are quite low.

For over ten years now, the basic method of lead extraction from galena has been hydrometallurgical process and conventional roasting. The hydrometallurgical route has received wide application because the method is environment friendly. This is sequel to the dissolution of produced gases (in the leaching solution) as the process progresses.

Studies [1-5] have evaluated the possibility of leaching galena in different acids. Attempts have been made [6] to extract lead from Ishiagu galena using different acids. The results of the investigation reveal that the concentration of lead after leaching with dilute nitric acid, sulphuric acid and aqueous solution of both acids are 39.5% w/w, 14.16% w/w and 15.18% w/w respectively. These results indicate that nitric acid gave the best result of recovery (84.2%), with original lead in the ore being 46.9% w/w.

Research [7] has shown that production of sufficiently soluble lead salts, from which very pure metal can be obtained by electrowinning is an alternative method for treating galena. On using hydrochloric acid (HCl) in the presence of chlorides, leaching of galena through non-oxidative method has been given much consideration, since it permits the direct conversion of lead sulfide into chloride as shown in the equation [7]:

$$PbS + 2HCl = PbCl_2 + H_2S \qquad (1)$$

In processes on a pilot-plant scale, reports [8-13] have shown that the accelerating effect of chlorides in the HCl solutions used for leaching galena has been widely accepted and considered applicable.

Results [14] of leaching galena in ferric chloride brine have revealed several advantages of ferric chloride over other reagents as a leaching media. These include exhibiting substantially faster dissolution rates for most sulphides, it is regenerated easily by chlorination of ferrous chloride leaching by-products. Also it has greater potential for the treatment of complex sulfides.

The aim of the present work is to carry out an implicit analysis of lead extraction based on its original lead content of galena and ferric sulphate concentration during leaching in the sulphate solution.

II. 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, 80 x 100, 60 x 80 mesh, 40 x 60, 20 x 40 mesh, 10 x 20 mesh and oversize. This range of particle size was used throughout the experiment. Based on the atomic absorption spectrometric analysis carried out, the samples used contain 86.55% Pb, indicating that the sample was essentially pure. Ferric sulphate 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. 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 extracted lead concentration with ferric sulphate concentration at original ore lead content 17.31ppm [15]

(γ)	(υ)	(ξ)
17.31	0.050	0.43
17.31	0.075	0.56
17.31	0.100	0.63
17.31	0.150	0.75
17.31	0.250	1.10

A. 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;

$$\xi - N\gamma = K\upsilon + S \qquad (2)$$

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

$$\xi - 0.0015\gamma = 3\upsilon + 0.3 \qquad (3)$$

$$\xi = 3\upsilon + 0.0015\gamma + 0.3 \qquad (4)$$

Where

(ξ) = Extracted lead conc. (ppm)

(υ) = Leaching temperature (^{o}C)

(γ) = Original Lead content of galena (ppm)

N = 0.0015, K = 3.0, and S = 0.3 are empirical constant (determined using C-NIKBRAN [16]

III. BOUNDARY AND INITIAL CONDITION

Galena was placed in cylindrical flask 30cm high containing leaching solution of ferric sulphate. 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 leaching temperature used was 50^0C. The reaction time used was 6 hrs. The range of Ferric sulphate concentration used was 0.05- 0.25, and H_2SO_4 concentration used was 0.1M. The pH range and used was 0.3 – 1.0.

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.

IV. MODEL VALIDATION

Table 2: Variation of $\xi - 0.0015\gamma$ with $3\vartheta + 0.3$

$\xi - 0.0015\gamma$	$3\upsilon + 0.3$
0.4044	0.4500
0.5344	0.5250
0.6044	0.6000
0.7244	0.7500
1.0744	1.0500

Equation (4) is the derived model. The validity of the model is strongly rooted on equation (3) where both sides of the equation are correspondingly approximately equal. Table 2 also agrees with equation (3) following the values of $\xi - 0.0015\gamma = 3\upsilon + 0.3$ evaluated from the experimental results in Table 1.

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

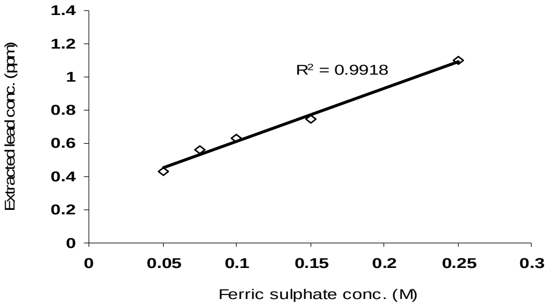

Fig.1: Coefficient of determination between extracted lead concentration and ferric sulphate concentration as obtained from experiment

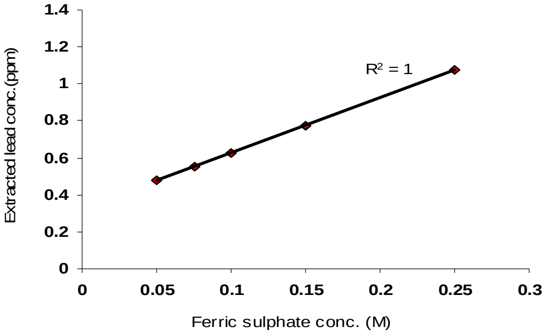

Fig.2: Coefficient of determination between extracted lead concentration and ferric sulphate concentration as obtained from derived model

A. Statistical Analysis

1) Standard Error (STEYX): The standard errors incurred in predicting lead yield for each value of the ferric sulphate concentration considered as obtained from experiment and derived model were 0.0266 and 3.04 x 10^{-9} % respectively. The standard error was evaluated using Microsoft Excel version 2003.

2) Correlation (CORREL): The correlation coefficient between extracted lead concentration and ferric sulphate concentration 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.

$$R = \sqrt{R^2} \qquad (5)$$

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 ferric sulphate conc.

Analysis	Based on ferric sulphate conc.	
	ExD	D-Model
CORREL	0.9959	1.0000

B. Graphical Analysis

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

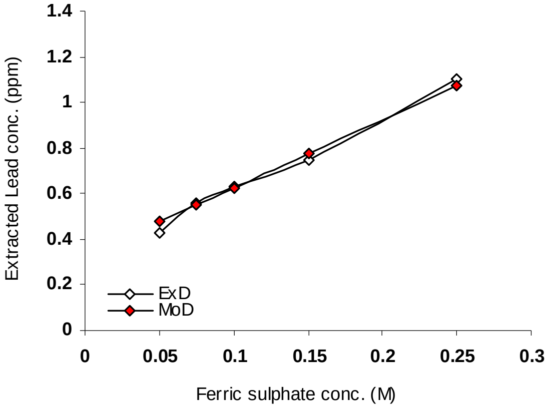

Fig.3: Comparison of extracted lead concentrations (relative to ferric sulphate conc.) as obtained from experiment and derived model

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

C. Computational Analysis

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

Extracted Lead concentration per unit ferric sulphate concentration

The extracted lead concentration per unit ferric sulphate concentration ζ_C was calculated from the expression;

$$\xi_C = \frac{\Delta\xi}{\Delta\upsilon} \qquad (6)$$

Equation (6) is detailed as

$$\xi_C = \frac{\xi_2 - \xi_1}{\upsilon_2 - \upsilon_1} \qquad (7)$$

Where

$(\Delta\xi)$ = Change in lead yield at two different ferric sulphate concentrations ϑ_2, ϑ_1.

Considering the points (0.05, 0.43) & (0.25, 1.1), and (0.05, 0.4756) & (0.25, 1.0756) as shown in Figs 1 and 2, and designating them as (ξ_1, υ_1) & (ξ_2, υ_2) for experimental and derived model predicted results respectively, and then substituting them into equation (7), gives the slopes: 3.35 and 3.0 ppm/M as extracted lead concentration per unit concentration respectively.

D. Deviational Analysis

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

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

Where

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

Critical analysis of the extracted lead concentration obtained from experiment and derived model shows low 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.

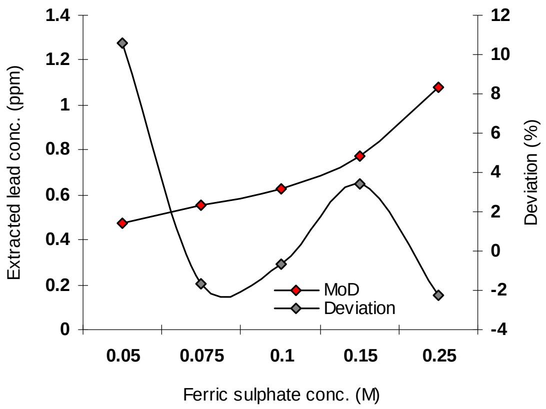

Fig.4: Variation of deviation with extracted lead concentration (relative to the ferric sulphate conc)

Fig. 4 shows that the maximum deviation of model-predicted extracted lead concentration from the experimental results is less than 11%. This translates into over 89% operational confidence and response level for the derived model as well as over 0.89 response coefficient of extracted lead concentration to the collective operational contributions of the ferric sulphate concentration and original lead content of the galena.

Consideration of equation (8) and critical analysis of Fig. 4 shows that the least and highest magnitudes of deviation of the model-predicted lead yield (from the corresponding experimental values) are – 0.7 and + 10.6. Figs. 1- 4 indicate that these deviations correspond to extracted lead concentrations: 0.6256 and 0.4756 ppm as well as ferric sulphate concentrations: 0.1 and 0.05M respectively.

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

$$\mathrm{Cf} = -\left[\frac{\xi_{\mathrm{MoD}} - \xi_{\mathrm{ExD}}}{\xi_{\mathrm{ExD}}}\right] \times 100 \qquad (9)$$

Critical analysis of Figs. 1-5 indicates that the evaluated correction factors are negative of the deviation as shown in equations (8) and (9).

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 (9) into the model gives exactly the corresponding experimental values of extracted lead concentration.

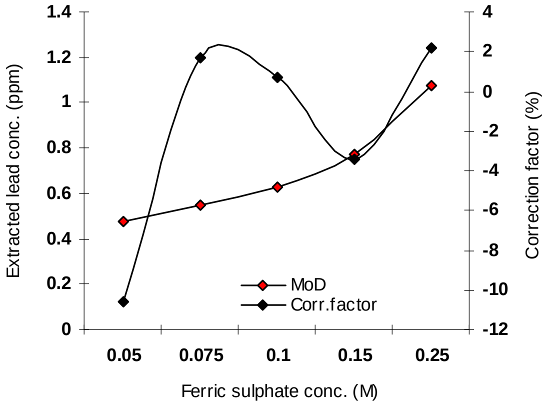

Fig.5: Variation of correction factor with extracted lead concentration (relative to the ferric sulphate conc)

Fig. 5 shows that the least and highest correction factor (to the model-predicted extracted lead concentration) are + 0.7 and – 10.6%. 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 extracted lead concentrations: 0.6256 and 0.4756 ppm as well as ferric sulphate concentrations: 0.1 and 0.05M 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).

V. CONCLUSION

An implicit assessment of lead extraction was carried out based on its as-mined content in galena and ferric sulphate concentration during leaching in the sulphate solution. A two-factorial empirical model derived and validated, following computational analysis of experimental results was used for the assessment. The validity of the model was rooted on the expression $\xi - 0.0015\gamma = 3\upsilon + 0.3$ where both sides of the expression are correspondingly approximately equal. Evaluation of generated results indicates that for each value of the ferric sulphate concentration, the statistical analysis of the extracted concentration as obtained from experiment and derived model-predicted results show standard errors of 0.0266 and 3.04 x 10^{-9} % respectively. Extracted lead concentration per unit ferric sulphate concentration as obtained from experiment and derived model-predicted results were 3.35 and 3.0 ppm/M respectively. Deviational analysis of model-predicted extracted lead concentration (from experimental result) was less than 11%. This invariably implies a derived model confidence level of above 89% as well as over 0.89 response coefficients for lead extraction dependence on its original content in the galena and ferric sulphate concentration.

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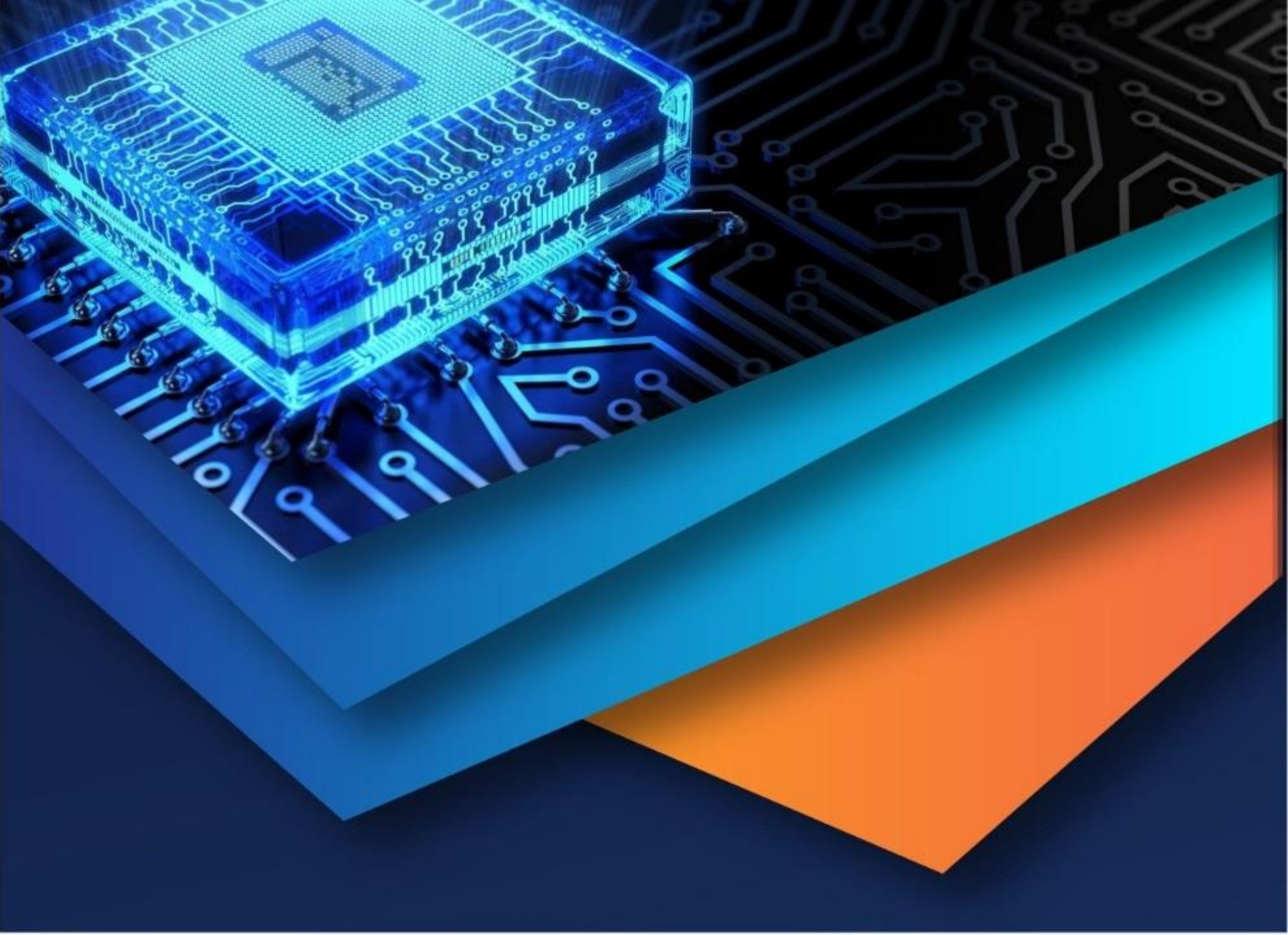

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