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Synergistic analysis of lead yield based on reaction time and leachant concentration during leaching of galena in ferric chloride solution

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Abstract
A synergistic analysis of lead yield was carried out based on the reaction time and ferric chloride concentration during leaching of galena in the chloride solution. An empirical model was derived, validated and used for the analysis. The validity of the model expressed as;

$$\zeta = 0.08\upsilon + 0.0013\gamma + 0.58$$

was rooted on the expression $\zeta - 0.0013\gamma = 0.08\upsilon + 0.58$ where both sides of the expression are correspondingly approximately equal. Statistical analysis of the extracted Pb concentration for each value of the reaction time as obtained from experiment and derived model-predicted results show standard errors of – 0.9772 and – 1.1011% respectively. Furthermore, Extracted Pb concentration per unit reaction time as obtained from experiment and derived model-predicted results were 0.08 and 0.08 ppm/ mins respectively. The maximum deviation of model-predicted concentration of extracted Pb (from experimental result) was less 15%. This translates into a derived model confidence level of above 85% as well as over 0.85 reliability coefficients for Pb extraction dependence on the reaction time and ferric chloride concentration.

Keywords: Evaluation, Lead yield, Galena, Reaction time, Ferric chloride.

1. Introduction
Lead is a soft metal; is easily oxidized, but it has found a lot industrial application such as in production of electronic components and different grades of alloys.
Different methods have been evaluated for extracting lead from galena. These are basically hydrometallurgical and pyrometallurgical methods. Hydrometallurgical method involves acid, salt and microbial leaching while pyrometallurgical method involves smelting.
Research [1] has revealed the kinetics and mechanisms of dissolution of the major base metal sulphide minerals, pyrite, chalcopyrite, galena and sphalerite in acidic (chloride) media. The redox potential was being monitored as minerals were ground in air, then dissolved in air-equilibrated solutions at pH 2.5. Solution samples were analysed by ICP-AES and HPLC, and surfaces of residual sulphides analysed using XPS. Results generated from the research indicated that the rates of dissolution of chalcopyrite, galena and sphalerite in the presence of pyrite were respectively as 18, 31 and 1.5 times more rapid than in single-mineral experiments. In the case of galena, the experimental data suggested extensive release of Pb ions and development of a sulphur-rich surface as galvanically-promoted dissolution progresses.
Report [2] shows the possibility of leaching galena concentrate using 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 sulphides [2]. Further studies [3] on the ferric chloride brine leaching of galena concentrate have been carried with the view to investigating the thermodynamics and kinetics of the process. It was discovered [3] that under the leaching condition of their work, the distribution of the various metal chloro complexes is relatively insensitive to the extent of PbS dissolution [3].
Appraisal [4] of the kinetics of Cl_2-O_2 leaching of galena flotation concentrate has indicated that the rate of gas transfer can be enhanced by increasing the partial pressure of the gas and by using vigorous agitation to increase the surface area of the liquid-gas interface. It has been shown [5, 6] that the final pH of the leaching solution depend on the leaching time, initial pH for the leaching solution and the leaching temperature.

The possibility of predicting the concentration of extracted lead in relation to the initial and final solution pH during leaching of galena in butanoic acid has been actualized [7]. The model expressed as:

$$Pb = \text{Antilog } \exp\left[\frac{\gamma}{\alpha}\right]^{0.7407} \quad (2)$$

Indicates that the concentration of dissolved lead during the leaching process is dependent on the values of the initial and final leaching solution pH. The validity of the model was rooted in the core expression ($(\log Pb)^N = e^{\gamma/\alpha}$ where both sides of the expression were correspondingly approximately almost equal. The maximum deviation of the model-predicted concentrations of dissolved lead from the corresponding experimental values is less than 7% which is quite within the acceptable deviation limit of experimental results.

The aim of the present work is to carry out a synergistic evaluation of lead yield based on reaction time and concentration of ferric chloride used during leaching of galena in the chloride 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, 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 chloride 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 lead yield with reaction time and ferric chloride concentration [8]

(ɤ)	(ϑ)	(ζ)
0.1	5	1.04
0.1	10	1.21
0.1	20	2.08
0.1	40	3.64
0.1	60	5.44

3.1 Model Formulation

Experimental data generated from this research work were used for the model formulation. Computational analysis of the data shown in Table 1, gave rise to Table 2 which indicate that;

$$\xi - N\text{ɤ} \approx K\vartheta + S \quad (2)$$

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

$$\zeta - 0.0013\gamma = 0.08\upsilon + 0.58 \quad (3)$$

$$\zeta = 0.08\upsilon + 0.0013\gamma + 0.58 \quad (4)$$

Where
(ζ) = Conc. of lead yield (ppm)
(ϑ) = Reaction time (mins.)
(ɤ) = Ferric chloride concentration (M)

N = 0.0013, K = 0.08, and S = 0.58. These are empirical constant (determined using C-NIKBRAN [9]

4. Boundary and Initial Condition

Galena was placed in cylindrical flask 30cm high containing leaching solution of ferric chloride. 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. Ferric chloride was added to 1.0M $NaCl_2$ and 1.0M HCl at 50°C. The range of pH used was 0.1 – 0.4. The range of reaction time used was 5-60 mins. and a constant leaching temperature of 50°C was used for all samples.

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 $\xi - 0.0013\text{ɤ}$ with $0.08\vartheta + 0.58$

$\xi - 0.0013\text{ɤ}$	$0.08\vartheta + 0.58$
1.0399	0.98
1.2099	1.38
2.0799	2.18
3.6399	3.78
5.4399	5.38

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 $\xi - 0.0013\text{ɤ} = 0.08\vartheta + 0.58$ evaluated from the experimental results in Table 1.

Furthermore, the derived model was validated by comparing the lead yield 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.

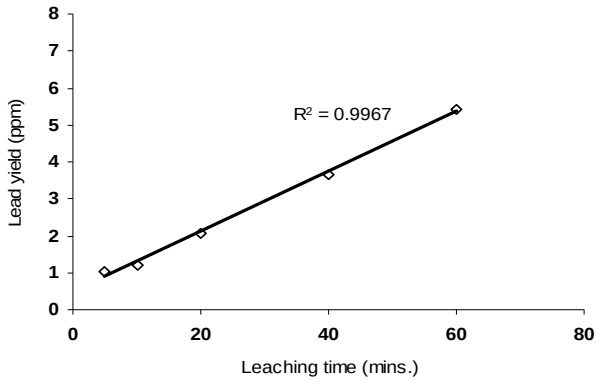

Fig 1: Coefficient of determination between lead yield concentration and reaction time as obtained from experiment

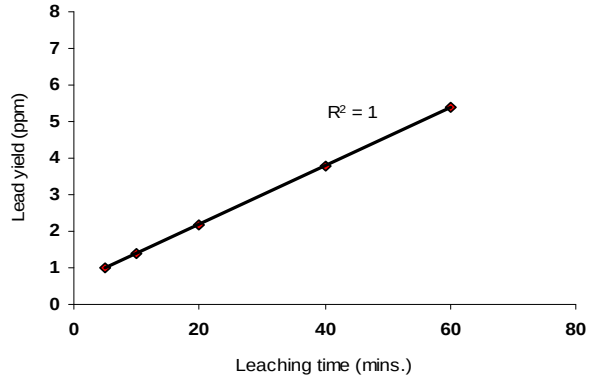

Fig 2: Coefficient of determination between lead yield concentration and reaction time as obtained from derived model

Statistical Analysis

Standard Error (STEYX)

The standard errors incurred in predicting lead yield for each value of the reaction time considered as obtained from experiment and derived model were – 0.9772 and – 1.1011 % respectively. The standard error was evaluated using Microsoft Excel version 2003.

Correlation (CORREL)

The correlation coefficient between lead yield and reaction 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.

$$R = \sqrt{R^2} \tag{5}$$

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 reaction time

Analysis	Based on reaction time	
	ExD	D-Model
CORREL	0.9983	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) lead yields.

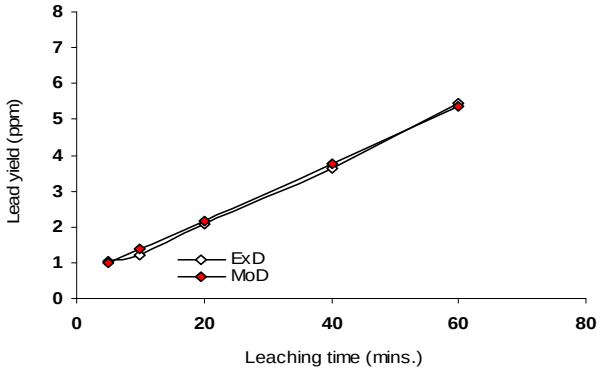

Fig 3: Comparison of lead yield concentrations (relative to reaction time) as obtained from experiment and derived model

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

Computational Analysis

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

Lead yield per unit reaction time

The lead yield per unit reaction time ζ_t was calculated from the expression;

$$\zeta_t = \Delta\zeta / \Delta\vartheta \tag{6}$$

Equation (6) is detailed as

$$\zeta_t = \zeta_2 - \zeta_1 / \vartheta_2 - \vartheta_1 \tag{7}$$

Where

$\Delta\zeta$ = Change in lead yield at two different reaction times ϑ_2, ϑ_1.

Considering the points (5, 1.04) & 60, 5.44), and (5, 0.9801) & (60, 5.3801) as shown in Figs 1 and 2, and designating them as (ξ_1, ϑ_1) & (ξ_2, ϑ_2) for experimental and derived model predicted results respectively, and then substituting them into equation (7), gives the slopes: 0.08 and 0.08 ppm/ mins. as lead yield rate respectively.

Deviational Analysis

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

$$Dv = \left[\frac{\zeta_{MoD} - \zeta_{ExD}}{\zeta_{ExD}}\right] \tag{8}$$

Where

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

Critical analysis of the lead yield 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.

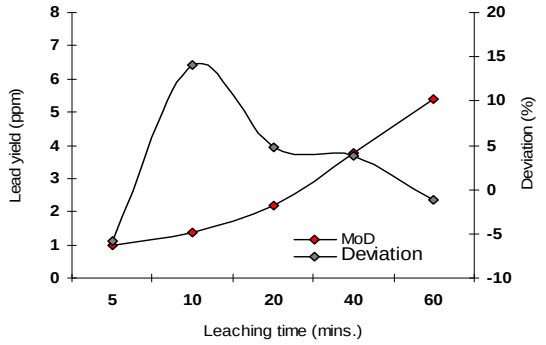

Fig 4: Variation of deviation with lead yield (relative to the reaction time)

Deviational analysis from Fig. 4 indicates that the maximum deviation of model-predicted lead yield from the experimental results is less than 15%. This translates into over 85% operational confidence and response level for the derived model as well as over 0.85 response coefficient of lead yield to the collective operational contributions of the reaction time.

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 – 1.1011 and + 14.06. Figs. 1- 4 indicate that these deviations correspond to lead yields: 5.3801 and 1.3801 ppm as well as reaction time: 60 and 10 mins. respectively.

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

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

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 lead yield.

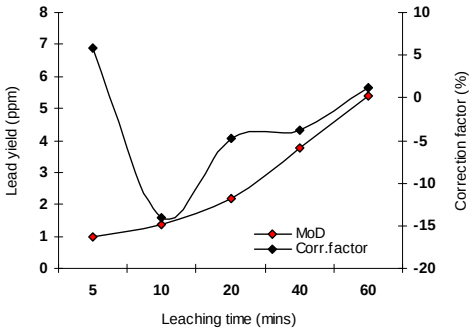

Fig 5: Variation of correction factor with lead yield concentration (relative to the reaction time)

Fig. 5 shows that the least and highest correction factor (to the model-predicted lead yield) are + 1.1011 and – 14.06 %. 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 lead yields: 5.3801 and 1.3801 ppm as well as reaction time: 60 and 10 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

A synergistic analysis of lead yield was carried out based on the reaction time and ferric chloride concentration during leaching of galena in the chloride solution. The validity of the derived empirical model was rooted on the expression $\xi - 0.0013\gamma = 0.08\vartheta + 0.58$ where both sides of the expression are correspondingly approximately equal. Statistical analysis of the extracted Pb concentration for each value of the reaction time as obtained from experiment and derived model-predicted results show standard errors of – 0.9772 and – 1.1011% respectively. Furthermore, Extracted Pb concentration per unit reaction time as obtained from experiment and derived model-predicted results were 0.08 and 0.08 ppm/ mins respectively. The maximum deviation of model-predicted concentration of extracted Pb (from experimental result) was less 15%. This translates into a derived model confidence level of above 85% as well as over 0.85 reliability coefficients for Pb extraction dependence on the reaction time and ferric chloride concentration.

7. References

1. Abraitis, P. K., Pattrick R. A. D., Kelsall, G. H and Vaughan, D. J. (2004). Acid Leaching and Dissolution of Major Sulphide Ore Minerals: Processes and Galvanic Effects in Complex Systems. Mineralogical Magazine. 68(2):343-351.DOI:10.1180/0026461046820191
2. Dutrizac, J.E. (1986). The Dissolution of Galena in Ferric Chloride Media. Metallurgical Transactions B , 17(1): 5-17.
3. Seon-Hyo, K. K., Henein, H., and Warren, G.W. (1986). An Investigation of the Thermodynamics and Kinetics of the Ferric Chloride Brine Leaching of Galena Concentrate. Metallurgical Transaction B, 17(1):29-39.
4. Dix, R. B., and Hendrix, J. L. (1986). Kinetics of Cl_2-O_2 Leaching of Lead–zinc Flotation Concentrates. University of Nevada Reno, 89557.
5. Nwoye, C.I. (2008). Bioleaching Studies of Ishiagu Galena. Ph.D Thesis, Metallurgical and Materials Engineering Department. Federal University of Technology, Owerri, Nigeria.
6. Pinches, A. (1975). Bacterial Leaching of an Arsenic Bearing Sulphide Concentrate. The Institute of Mining and Metallurgy, England, 34.
7. Nwoye, C. I. and Mbuka, I. E. (2010). Model for Predictive Analysis of the Concentration of Dissolved Lead in relation to the Initial and Final Solution pH during Leaching of Galena in Butanoic Acid. Journal of Academia Arena, 2(6), 54-61.
8. Mbah, C. N. (2012) Leaching Characteristics of Enyimgba Galena in Aqueous Ferric Salt Lixiviants. Ph.D Thesis, Enugu State University of Science & Technology, Enugu, Enugu State.
9. Nwoye, C. I. (2008). Data Analytical Memory; *C-NIKBRAN*.