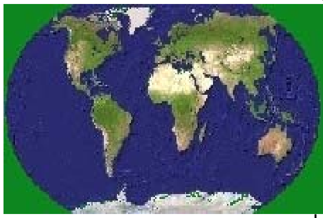

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Empirical analysis of lead extraction based on reaction temperature and time during leaching of galena in ferric sulphate solution

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Abstract

This paper presents an empirical analysis of lead extraction based on the reaction temperature and time during leaching of galena in ferric sulphate solution. A model was derived, validated and used for the analysis. The validity of the model expressed as;

$$\zeta = 0.128 \ln \upsilon + 0.0001\gamma + 0.22$$

was rooted on the expression $\zeta - 0.0001\gamma = 0.128 \ln \upsilon + 0.22$ 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 0.0346 and 0.0285% respectively. Furthermore, Extracted Pb concentration per unit reaction time as obtained from experiment and derived model-predicted results were 0.0136 and 0.0124 ppm/ hr respectively. The maximum deviation of model-predicted concentration of extracted Pb (from experimental result) was less 6%. This translates into a derived model confidence level of above 94% as well as over 0.94 reliability coefficients for Pb extraction dependence on the reaction temperature and time.

Keywords: Analysis, Lead extraction, Reaction temperature & time, Galena, Ferric sulphate.

1. Introduction

The overwhelming usefulness of lead for production of electronic components and different grades of industrial alloys has raised the need for intensive research and development geared towards improving methods of extracting lead from its natural ores.

Different methods of extracting lead from its natural ores have been evaluated to underscore the possibility of increasing yield at reduced input variables. The possibilities of achieving bacterial assisted extraction of lead from its natural ores have been evaluated and found amply rewarding.

Since the last two decades, hydrometallurgical process and conventional roasting has been the basic method of lead extraction from galena. The hydrometallurgical route has received wide application because the method significantly eliminates atmospheric pollution due to production of SO_2. Environment friendliness of this process is based on the dissolution of produced gases (in the leaching solution) as the process progresses.

Research [1] has revealed the possibility of leaching galena by non-oxidative method using hydrochloric acid in the presence of a metallic chloride. The research shows that a succession of metallic chlorides with cations of different valencies could be used in an attempt to generalize the leaching behavior of these solutions. The reaction order for leaching galena, in terms of the mean ionic activity of HC1 was 3/2 over a wide range of concentration. Results generated from the research clearly revealed that the addition of soluble chlorides to a HC1 solution increased the leaching rate of the galena by augmenting the mean ionic activity of the acid. It was observed that the only activity that must feature in the kinetic equation is that of the HC1. The activation energy (58.5 kJ/mole) was found to be independent of the chloride used to increase the activity of the hydrochloric acid; in line with the Arrhenius prefactor.

Production of sufficiently soluble lead salts, from which very pure metal can be obtained by electrowinning is an alternative method for treating galena [1].

Leaching of galena through non-oxidative method using HCl in the presence of chlorides has been given much consideration, since it permits the direct conversion of lead sulfide into chloride as shown in the equation [1]:

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

Studies [2-7] have shown that the accelerating effect of chlorides in the HCl solutions used for leaching galena is well known and has found application in processes on a pilot-plant scale.

The aim of the present work is to carry out an empirical analysis of lead extraction based on reaction temperature and time during leaching of galena in ferric sulphate solution.

2. 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 reaction temperature and time [8]

(γ)	(ϑ)	(ζ)
50	2	0.34
50	4	0.40
50	6	0.43
50	10	0.52
50	14	0.56
50	16	0.61
50	24	0.64

2.1 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;

$$\zeta - N\gamma = K \ln \upsilon + S \tag{2}$$

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

$$\zeta - 0.0001\gamma = 0.128 \ln \upsilon + 0.22 \tag{3}$$

$$\zeta = 0.128 \ln \upsilon + 0.0001\gamma + 0.22 \tag{4}$$

Where

(ζ) = Conc. of extracted lead (ppm)

(υ) = Reaction time (hr)

(γ) = Reaction temperature (^{0}C)

S = 0.22, K = 0.128, and N = 0.0001. These are empirical constant (determined using C-NIKBRAN [9]

3. 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 pH range used is 0.3 – 1.0.

The range of reaction time used was 2-24 hrs and a constant leaching temperature of 50oC 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.

4. Model Validation

Table 2: Variation of ζ - 0.0001 γ with 0.128 ln υ + 0.22

$\xi - 0.0001\gamma$	$0.128 \ln \upsilon + 0.22$
0.3399	0.3087
0.3999	0.3974
0.4299	0.4493
0.5199	0.5147
0.5599	0.5578
0.6099	0.5749
0.6399	0.6268

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 $\zeta - 0.0001\gamma = 0.128 \ln \upsilon + 0.22$ 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.

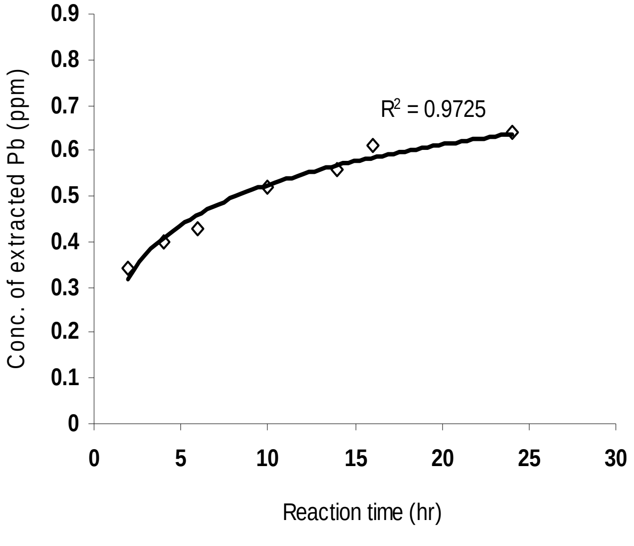

Fig 1: Coefficient of determination between lead yield concentration and reaction time as obtained from experiment [8]

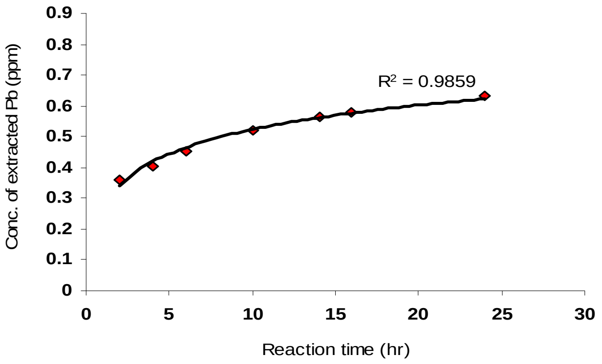

Fig 2: Coefficient of determination between lead yield concentration and reaction time as obtained from derived model

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 0.0346 and 0.0285 % 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.

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

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 reaction time

Analysis	Based on reaction time	
	ExD	**D-Model**
CORREL	0.9862	0.9929

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.

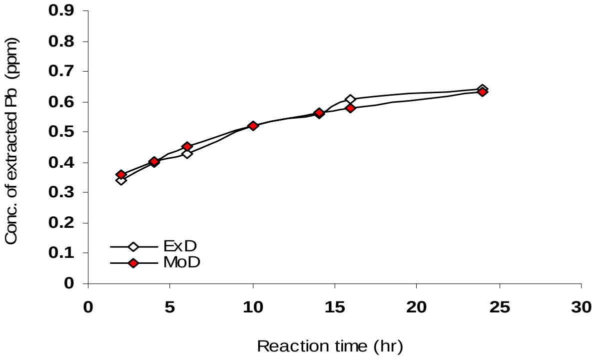

Fig 3: Comparison of lead yield concentrations (relative to reaction time) as obtained from experiment and derived model Furthermore, the degree of alignment of these curves is indicative of the proximate agreement between both experimental and model-predicted lead yields.

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;

$$\zeta_t = \Delta \zeta / \Delta \vartheta \qquad (6)$$

Equation (6) is detailed as

$$\zeta_t = \frac{\zeta_2 - \zeta_1}{\upsilon_2 - \upsilon_1} \qquad (7)$$

Where

$\Delta\zeta$ = Change in lead yield at two different reaction times ϑ_2, ϑ_1. Considering the points (2, 0.34) & (24, 0.64), and (2, 0.3587) & (24, 0.6318) 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: 0.0136 and 0.0124 ppm/hr as lead yield rate respectively.

Deviational Analysis

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

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

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.

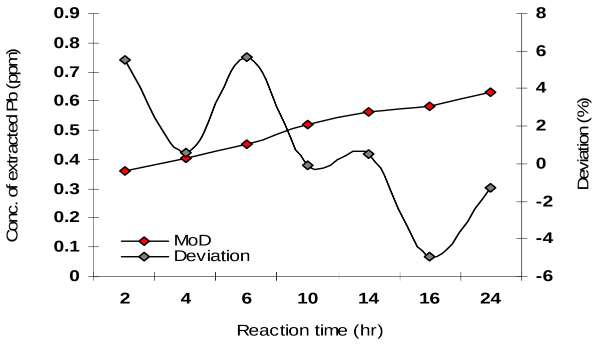

Fig 4: Variation of deviation with lead yield (relative to the reaction time)

Deviational analysis from Fig. 4 indicates that the precise maximum deviation of model-predicted lead yield from the experimental results was less than 6%. This translates into over 94% operational confidence and response level for the derived model as well as over 0.94 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 - 0.06 and + 5.65. Figs. 1- 4 indicate that these deviations correspond to lead yields: 0.4024 and 0.4543 ppm as well as reaction time: 10 and 6 hrs respectively.

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

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

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.

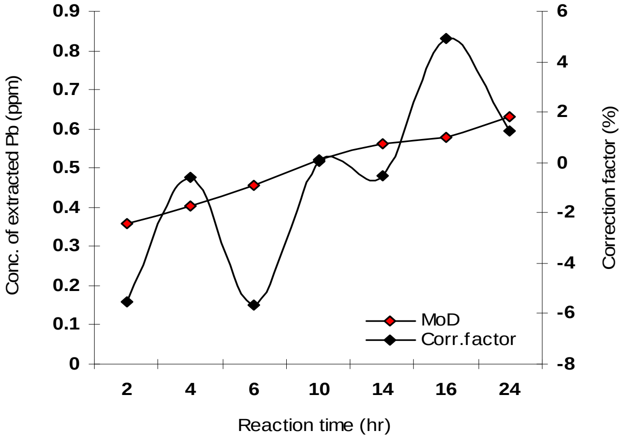

Fig 5: Variation of correction factor with lead yield concentration (relative to the reaction time)

Fig. 5 shows that the least and highest correction factor (to the model-predicted lead yield) are + 0.06 and – 5.65 %. 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: 0.4024 and 0.4543 ppm as well as reaction time: 10 and 6 hrs 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).

5. Conclusion

An empirical analysis of lead extraction has been carried out based on the reaction temperature and time during leaching of galena in ferric sulphate solution. A model was derived, validated and used for the analysis. The validity of the model was rooted on the expression $\xi - 0.0001\gamma = 0.128 \ln\vartheta + 0.22$ 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 0.0346 and 0.0285% respectively. Furthermore, Extracted Pb concentration per unit reaction time as obtained from experiment and derived model-predicted results were 0.0136 and 0.0124 ppm/ hr respectively. The maximum deviation of model-predicted concentration of extracted Pb (from experimental result) was less 6%. This translates into a derived model confidence level of above 94% as well as over 0.94 reliability coefficients for Pb extraction dependence on the reaction temperature and time.

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