

## MODEL FOR PREDICTING THE INITIAL SOLUTION pH AT PRE-ASSUMED FINAL pH AND CONCENTRATION OF DISSOLVED LEAD DURING LEACHING OF GALENA IN BUTANOIC ACID SOLUTION

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### Abstract

Model for predicting the initial solution pH at pre-assumed final pH and concentration of dissolved lead, during leaching of galena in butanoic acid solution has been derived. The model shows that the initial pH of the leaching is dependent on the values of the pre-assumed final solution pH and concentration of dissolved lead. The validity of the model was rooted in the expression  $e^{Ny/\alpha} = \sqrt[3]{Pb}$  where both sides of the expression were approximately equal to 5. The maximum deviation of the model-predicted initial solution pH value from that of the corresponding experimental value was less than 3% which is quite within the acceptable deviation limit of experimental results.

Keywords: Model, Prediction, pH, Butanoic acid, Galena, Leaching.

### 1. Introduction

Pyrometallurgical route has for long been widely applied as a conventional means of lead extraction from galena. There are two main processes which have account for world's production of lead; the hearth smelting and the blast furnace smelting of roasted ores. In as much as these routes have effectively recovered both lead and other associated precious metals, they have not really been widely accepted due to emission of poisonous gases during the associated operations. Based on the foregoing, it is clear that the need for environmental-friendly industrial operations has posed a threat to the future well being of lead producing industries. Application of hydrometallurgy in lead extraction prevents  $SO_2$  formation by recovering elemental sulphur [1, 2].

**Nomenclatures**

$C$	Bacterial leaching index
$C_r$	Correction factor for the model
$D_n$	Deviation in the model
$E_l$	Experimental initial pH values
$ExD$	Experimental model value
$MoD$	Model predicted value
$N$	Dissolution coefficient of lead in butanoic acid (=1.61)
$P_l$	Predicted initial pH value
$Pb$	Concentration of dissolved lead during the leaching process (mg/kg)
$R^2$	Correlation coefficient
$t$	Leaching time, s

*Greek Symbols*

$\alpha$	Initial pH of the leaching solution at time $t$
$\gamma$	Final pH of the leaching solution at time $t$
$\Delta G$	Leaching free energy change
$\phi$	Leaching rate of lead from galena (g/dm <sup>3</sup> .hr)
$\theta$	Concentration of Pb leached out

Sedimentation analysis has been carried out on Ishiagu galena concentrate to determine its particle size and size distribution [3]. The result of the investigation shows that the average grain size of the ore is approximately 100  $\mu\text{m}$ . The researcher [3] further studied the mechanism of bioleaching of Ishiagu galena concentrates and found that it is indirect mechanism. This was sequel to the dominance of  $\text{Fe}^{3+}$  ions over  $\text{H}^+$  during the leaching process. The best operating conditions for the highest yield of Pb were found to be; leaching temperature of not less than 32°C, starting pH of leaching solution in the range 1.8 – 2.0, concentration of  $\text{Fe}^{2+}$  in the starting leaching solution; 2g/dm<sup>3</sup> (0.007M), grain size of ore to be leached; 0.063  $\mu\text{m}$  and mixed culture of *Acidithiobacillus Ferrooxidans* (ATF), *Acidithiobacillus Thiooxidans* (ATT) and a newly discovered bacteria (CBT) [3]. A model has been derived for predicting the leaching rate of lead during bioleaching of galena using different strains of bacteria such ATF, ATT and the newly discovered bacteria (CBT and CTT) [3]. The model referred to as pH-model, calculates the leaching rate when the pH of the leaching solution is known at any instant during the leaching process

$$\phi = e^{-(\gamma + \ln \gamma)} \quad (1)$$

where

$\gamma$  is the pH of the leaching solution at any instant during the leaching process,

$\phi$  is the leaching rate of lead from galena (g/dm<sup>3</sup>hr<sup>-1</sup>).

On multiplying both sides of the model by leaching time  $t$ , the model then calculates the concentration of Pb leached out as  $\theta = e^{-(\gamma + \ln \gamma)t}$ .

Derivation of a model has been carried out [3] (known as  $\Delta G$  – model) for predicting the leaching rate of lead relative to the bacterial leaching index and the free energy change associated with the bioleaching process involved.

The model

$$\phi = 10^{\Delta G/C} \quad (2)$$

calculates the leaching rate when the values of the free energy change  $\Delta G$ , associated with the leaching reaction as well as bacterial leaching index  $C$ , are known. This model indicates that the value of the leaching rate and concentration of leached Pb depends largely on the nature and leaching ability of bacteria or bacteria consortium used. It was observed that the greater the value of  $C$ , the higher the bacterial leaching ability and tendency. It was also found that the bacterial leaching index of *Bacillus spp.* is within the range 2-2.2 while mixed cultures of *Bacillus spp.* gave greater value (close to 3) of  $C$  than the case of single *Bacillus spp.* [3]. Based on the foregoing, given the values of the leaching rate and the associated free energy change, the species of the bacteria used can be identified by calculating the value of  $C$ , just by re-arranging the model as  $C = \Delta G / \log \phi$ . It was observed that *Pseudomonas spp.* of bacteria have a value of  $C$ , less than 1. This value was found to be associated with very poor yield of Pb and leaching rate. Furthermore, on multiplying both sides of the model by the leaching time  $t$ , the model then calculates the concentration of leached Pb as  $\theta = (10^{\Delta G/C})t$ . A more comprehensive and precision-enhanced model was also derived by jointly associating the pH-model and  $\Delta G$ -model. The resultant model;

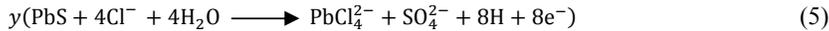
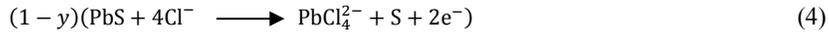
$$\Delta G = \log e^{-(\gamma + \ln \gamma)C} \quad (3)$$

not only calculates both the leaching rate and concentration of leached Pb (though indirectly), but also calculates directly the free energy change associated with the leaching process as well as the bacterial leaching index, as the case may be providing that two of the process parameters are known. The pH of the leaching solution during the leaching process can also be calculated using this model.

The leaching rates of single sulphide minerals have been found [4] to decreased in the order pyrite > sphalerite > galena > chalcopyrite, with the rate of pyrite dissolution being of a similar magnitude to the highest values reported previously [4]. The leaching rates of galena, chalcopyrite and sphalerite increased by factors of 31, 18 and 1.5, respectively, in the presence of pyrite, due to its superior catalytic properties [4]. In the galena+pyrite experiment, the concentration of Fe did not increase appreciably between the first and final sampling times, whilst the Pb concentration did increase significantly. Therefore, dissolution of galena+pyrite promoted galvanically decreases the pyrite electrode potential and its dissolution rate. Also in the galena+pyrite experiment, 75% of the total S in solution as measured by ICP–AES was detectable by HPLC, which detects only anionic species; this could be due to the presence of colloidal elemental S [4]. Acid leaching of lead sulphide has been investigated [5]. The results of the investigation indicate that prior to mineral addition, the redox potential of the acid solutions was  $360 \pm 10$  mV (SHE). On addition of the mineral powders, the value changed rapidly. In most cases, the redox potential then decreased by a few tens to a hundred or more mV to reach a stable value, except for the single-phase galena+pyrite mixture, for which the redox potential rose continuously throughout the experiment. The ranges of redox potential at pH 2.5 recorded for each single, mineral sample is plotted onto the potential pH diagram for the S–H<sub>2</sub>O system. Although the pH of the leach solution was kept constant, the generation or consumption of protons could be determined by monitoring the volume of acid needed to maintain a pH of 2.5. These quantities (in cm<sup>3</sup>) were for

galena: 0.19, galena+pyrite: 1.90 and other elements. Oxidation products formed during grinding of galena dissolves resulting in the production of dissolved Pb(II) and sulphur concentrations significantly higher than in the case of sphalerite leaching. However, concentrations of both species decreased over the first hour of the experiment, probably due to restricted solubility of  $\text{PbCl}_2$  and  $\text{PbSO}_4$ , the latter phase having a particularly low solubility product  $K_{sp}(\text{PbSO}_4) = 10^{-7.86}$ .

The equations of the reactions involved [5] are as follows.



Though the dissolved metals to sulphur ratios were not as high as in the case of sphalerite leaching, they increased from 1.3 ( $y = 0.7$ ) after 19 minutes to 2.5 ( $y = 0.4$ ) after 182 min. Based on the XPS data, no significant changes in S speciation occur at the surfaces of these minerals as a result of atmospheric oxidation and acid leaching. Elemental S has been reported [6] at the surfaces of both air oxidized and acid-leached galena but no evidence for the presence of elemental S was obtained here possibly due to the sulphur desorption in UHV chamber of the instrument, which had no low – temperature range [5].

Lead sulphide is rapidly attacked by ferric ion over a wide range of conditions and during this reaction, soluble lead chlorocomplexes as well as ferric and ferrous chlorocomplexes are formed [7]. It is to be expected after all, that the various metal chlorocomplexes would play an important role in the leaching reaction mechanism. In addition, it is clear from the result of previous researchers that the effect of  $\text{Fe}^{3+}$ ,  $\text{Cl}^-$  and  $\text{H}^+$  are coupled, and that the reaction kinetics and the mechanism of ferric chloride leaching of PbS have not been clearly established [7]. Rapid parabolic kinetics was observed in this study under all conditions and it was shown that the parabolic rate constant was directly proportional to the area of galena being leached. In the presence of ferric ion, the rate was insensitive to HCl concentrations  $< 3.0\text{M}$ , but increased rapidly at higher acidities because of direct acid attack of the sulphide. In the absence of ferric ion, the rate increased steadily with increasing HCl concentrations, and linear kinetics was observed. The galena leaching rate increased as  $(\text{FeCl}_3)$  for  $\text{FeCl}_3$  concentrations in the range 0.01-0.1M, but decreased slightly with increasing  $\text{FeCl}_3$  concentrations in the range 0.1M to 2.0M  $\text{FeCl}_3$ . The rate was virtually independent of the concentration of the  $\text{FeCl}_2$  reaction product. The presence of significant amount of the  $\text{PbCl}_2$  reaction product, however, caused the galena leaching rate to decrease rapidly. A minimum leaching rate was realized in saturated  $\text{PbCl}_2$  solutions [7].

Studies on the ferric chloride brine leaching of galena concentrate have been carried out [7]. 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 [7]. Further studies [8] 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 [8] 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 [8].

Investigations on the  $\text{Cl}_2\text{-O}_2$  leaching of galena flotation concentrate have been carried out with the view to evaluating the kinetics of the process [9]. The results of this investigation indicate 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. Studies [3, 10] have shown that the final pH of the leaching solution depend on the leaching time, initial pH for the leaching solution and the leaching temperature.

Research has shown that galena contains some quantities of zinc and copper [3]. Past report [11] has also indicated that sulphides of both zinc and copper contain lead. Therefore during dissolution of lead in an acid solution, it is expected that some of the zinc and copper present in the sulphide dissolves and some lead present in the sulphides of zinc and copper also dissolves. It was found [11] during the leaching of zinc and copper out from their respective sulphide ore that the concentrations of zinc and copper formed reduced as particle size decreased while silica, sulphur, iron and lead contents increased. Also leaching rate of copper was found to be lower than zinc.

A model has been derived for predicting the initial solution pH at pre-assumed final pH and concentration of dissolved zinc, during leaching of galena in butanoic acid solution [12]. The model

$$\alpha = \frac{1.4 \gamma}{\ln \text{Zn}^{1/3}} \quad (7)$$

shows that the initial pH of the leaching is dependent on the values of the pre-assumed final solution pH and concentration of dissolved zinc. The validity of the model was rooted in the expression  $e^{N\gamma/\alpha} = \sqrt[3]{\text{Zn}}$ , where both sides of the expression were approximately equal to 4. The respective deviation of the model-predicted initial solution pH value from that of the corresponding experimental value was less than 2% which is quite within the acceptable deviation limit of experimental results.

Derivation of model has been carried out [13] for assessment and computational analysis of the concentration of zinc dissolved during leaching of sphalerite in butanoic acid. The model,

$$\text{Zn} = \text{Antilog}[\exp(\gamma/\alpha)^{0.6173}] \quad (8)$$

shows that the concentration of dissolved zinc is dependent on the initial and final pH of the leaching solution. The validity of the model was found to be rooted in the expression  $(\log \text{Zn})^{1.62} = \exp(\gamma/\alpha)$  where both sides of the expression are approximately equal to 3. The maximum deviation of the model-predicted concentrations of dissolved zinc from that of the corresponding experimental values is less than 14% which is quite within the acceptable deviation limit of experimental results. This derivation was carried out under the process conditions: initial pH range of leaching solutions used: 3.80-4.83, leaching time:

2 hrs. Leaching temperature: 25°C, butanoic acid concentration: 0.27 mol/litre and average ore grain size 150 µm were used.

A model was derived 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 [14]. The model shows

$$Pb = \text{Antilog}[\exp(\gamma/\alpha)^{0.7407}] \quad (9)$$

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 found to be rooted in the 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 derived models [12, 14] agree with past studies [3,11] that galena contains zinc which also dissolves simultaneously with lead. Further more, preliminary investigations carried out on the sphalerite before embarking on the actual experiment [15] leading to derivation of model [13] indicate that little quantities of lead and copper also dissolved during dissolution of zinc dissolved.

The aim of this work is to derive a model for predicting the initial solution pH at pre-assumed final pH and concentration of dissolved lead during butanoic acid leaching of Ishiagu (Nigeria) galena. The proposed work resulted from the need to be informed about the range of initial solution pH at which a feasible butanoic acid leaching of galena should commence having pre-assumed the final pH and concentration of dissolved lead expected at the end of the leaching process. It is expected that the model would guide extractive metallurgists in achieving maximum yield through the application of the optimum initial pH of the leaching solution. This derivation is in furtherance of the previous work [16].

## 2. Model

During the leaching process, the ore was assumed to be stationary in the reaction vessel and contains the un-leached lead and zinc as part of reaction remnants. The ore was attacked by hydrogen ions from butanoic acid within the liquid phase, and in the presence of oxygen.

### Model formulation

Results from experimental work [16] carried out at Synchro Well Research Laboratory, Enugu were used for the model derivation. These results are as presented in Table 1.

Computational analysis of these experimental results [16] shown in Table 1, resulted to Table 2 which indicate that;

$$e^{Ny/\alpha} = \sqrt[3]{Pb} \text{ (approximately), or} \\ e^{Ny/\alpha} = (Pb)^{1/3} \quad (10)$$

**Table 1. Variation of the Initial and Final pH of the Butanoic Acid Leaching Solution with the Concentration of Dissolved Lead [16].**

$\gamma$	$\alpha$	Pb
3.98	3.80	140.22
4.25	4.08	138.42
4.33	4.24	136.22
4.41	4.36	136.02
4.50	4.46	135.86
4.61	4.55	135.04
4.63	4.60	133.96
4.72	4.67	133.34
4.84	4.81	132.82
4.86	4.83	132.60

**Table 2. Variation of  $e^{N\gamma/\alpha}$  with  $\sqrt[3]{\text{Pb}}$ .**

$\gamma/\alpha$	$N\gamma/\alpha$	$e^{N\gamma/\alpha}$	$\sqrt[3]{\text{Pb}}$
1.0474	1.6863	5.3995	5.1952
1.0417	1.6771	5.3500	5.1729
1.0212	1.6441	5.1763	5.1453
1.0115	1.6285	5.0962	5.1428
1.0090	1.6245	5.0759	5.1408
1.0132	1.6313	5.1105	5.1304
1.0065	1.6205	5.0556	5.1167
1.0107	1.6272	5.0896	5.1088
1.0062	1.6200	5.0531	5.1022
1.0062	1.6200	5.0531	5.0993

Taking the natural log of both sides of Eq. (10)

$$N\gamma/\alpha = \ln[(\text{Pb})^{1/3}] \quad (11)$$

$$\gamma/\alpha = \frac{\ln\left[(\text{Pb})^{\frac{1}{3}}\right]}{N} \quad (12)$$

$$\alpha/\gamma = \frac{N}{\ln\left[(\text{Pb})^{\frac{1}{3}}\right]} \quad (13)$$

Introducing the value of  $N$  into Eq. (13) reduces it to;

$$\alpha = \frac{1.61\gamma}{\ln\left[(\text{Pb})^{\frac{1}{3}}\right]} \quad (13)$$

where,  $N = 1.61$ , dissolution coefficient of lead in butanoic acid, determined in the experiment [16].

$\alpha$  is initial pH of the butanoic acid leaching solution just before the leaching process started.

$\gamma$  is the final pH of the butanoic acid leaching solution at time  $t$ .

Pb is the concentration of dissolved lead during the leaching process (mg/kg). Equation (13) is the derived model.

### 3. Initial and Boundary Conditions

Iron oxide ore was placed in cylindrical flask 30 cm high containing leaching solution of hydrogen peroxide. 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 iron oxide ore used was 5 g. The initial pH range of leaching solutions used; 3.80 - 4.83 and leaching time of 2 hrs (120 minutes) were used for all samples. A constant leaching temperature of 25°C was used. Hydrogen peroxide concentration at 0.27 mol/litre and average ore grain size; 150  $\mu\text{m}$  were also used. Details of the experimental technique are as presented in the report [16].

The leaching process boundary conditions include: atmospheric levels 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

The formulated model was validated by calculating the deviation of the model-predicted initial pH from the corresponding experimental pH values.

The deviation recorded is believed to be due to the fact that the surface properties of the ore and the physiochemical interactions between the ore and leaching solution which were found to play vital roles during the leaching process [16] were not considered during the model formulation. It is expected that introduction of correction factor to the predicted initial pH, gives exactly the experimental initial pH values.

Deviation,  $D_n(\%)$ , of model-predicted initial pH values from those of the experiment is given by

$$D_n = \frac{P_I - E_I}{E_I} \times 100 \quad (15)$$

where

$P_I$  = Predicted initial pH values

$E_I$  = Experimental initial pH values

Since correction factor ( $C_r$ ) is the negative of the deviation,

$$C_r = -D_n \quad (16)$$

Substituting Eq. (15) into Eq. (16) for  $D_n$ ,

$$C_r = -\frac{P_I - E_I}{E_I} \times 100 \quad (17)$$

It was observed that addition of the corresponding values of  $C_r$  from Eq. (17) to the model-predicted initial pH gave exactly the corresponding experimental initial pH values [16].

## 5. Results and Discussion

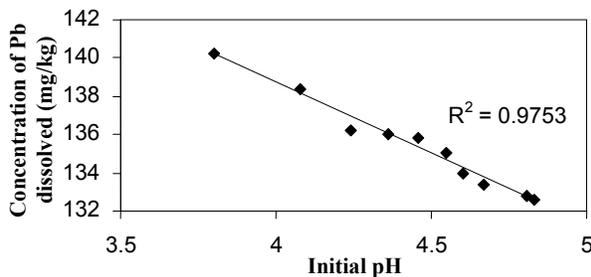
The derived model is Eq. (14). An ideal comparison of the initial pH as obtained from experiment and as predicted by the model for the purpose of testing the validity of the model is achieved by considering the  $R^2$  values (coefficient of determination). The values of the correlation coefficient,  $R^2$  calculated from the equation;

$$R = \sqrt{R^2} \quad (18)$$

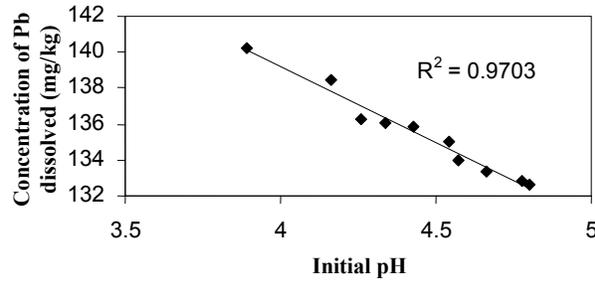
Using the r-squared values (coefficient of determination) from Figs. 1 and 2 show a very close correlation (0.9703) and (0.9753) between model-predicted initial solution pH and that obtained from the experiment [16] respectively. This suggests that the model predicts accurate and reliable initial solution pH which is in proximate agreement with values from the actual experiment.

Figure 3 shows very close alignment of the curves from model-predicted values of the initial solution pH ( $MoD$ ) and that from the corresponding experimental values ( $ExD$ ). The degree of alignment of these curves is indicative of the proximate agreement between both experimental and model-predicted initial solution pH.

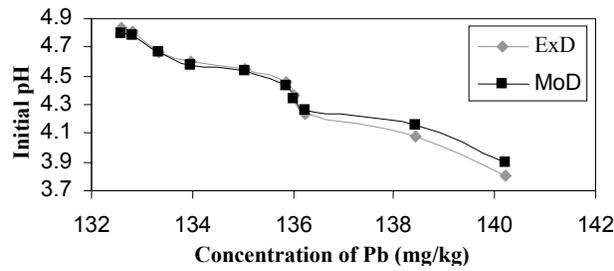
The validity of the model is believed to be rooted on Eq. (10) where both sides of the equation are approximately equal to 5. Table 2 also agrees with Eq. (10) following the values of  $e^{N\gamma/\alpha}$  and  $\sqrt[3]{Pb}$  evaluated following statistical and computational analysis carried out on the experimental results in Table 1.



**Fig. 1. Effect of Initial Solution pH (As Obtained from the Experiment [16]) on the Concentration of Lead Dissolved in Butanoic Acid Solution.**

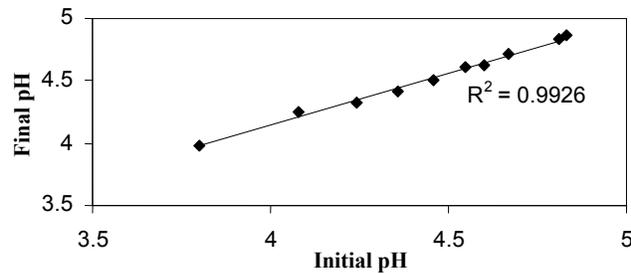


**Fig. 2. Effect of Initial Solution pH (As Predicted by Derived Model) on the Concentration of Lead Dissolved in Butanoic Acid Solution.**

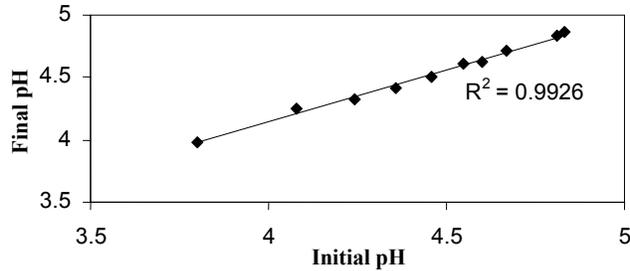


**Fig. 3. Comparison of Initial Solution pH as Obtained from the Experiment [16] and Predicted by Derived Model.**

Comparison between Figs. 4 and 5 show the final solution pH from experiment gives very close correlations; (0.9997) and (0.9926) with the initial pH predicted by the model and that from the experiment [16] respectively. These two correlation values also show proximate agreement between model-predicted and experimental values of the initial solution pH.

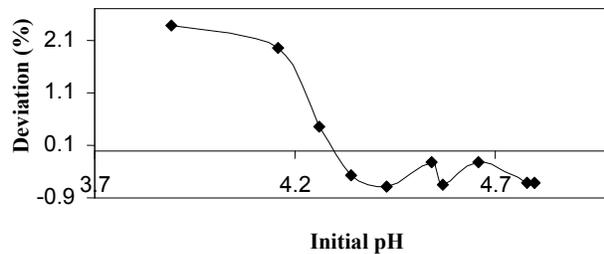


**Fig. 4. Effect of Initial Solution pH on the Final pH (Both As Obtained from the Experiment [16]).**



**Fig. 5. Effect of Initial Solution pH (From Model) on the Final pH (Obtained from the Experiment [16]).**

Based on the foregoing, the model is believed to be very valid as a predictive tool. Furthermore, Fig. 6 shows insignificant positive and negative deviations of the model-predicted initial solution pH from the corresponding experimental values. Figure 6 also shows that the maximum deviation of the model-predicted initial solution pH values from those of the experiment is less than 3% which is quite within the acceptable deviation limit of experimental results. The positive and negative deviations (of the model-predicted initial pH) from the actual experimental values show undulating relationship (Fig. 6) with the predicted initial pH.

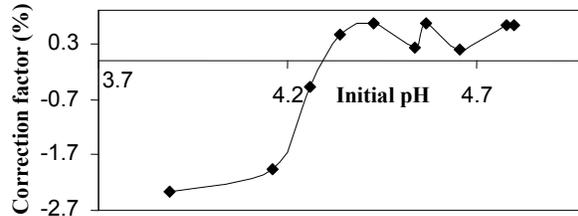


**Fig. 6. Variation of Deviation (From Experimental Values [16]) of Model – Predicted Initial Solution pH with Model – Predicted Initial Solution pH.**

The least and highest magnitude of deviation of the model-predicted initial solution pH (from the corresponding experimental values) are -0.22% and 2.37% which correspond to initial solution pH 4.66 and 3.89 respectively.

Correction factor for the model-predicted initial solution pH (Fig. 7) similarly shows an undulating relationship with model-predicted initial pH. However, the orientation of this curve is opposite that of the deviation values of model-predicted initial pH (Fig. 6). This is because correction factor is the negative of the deviation as shown in Eqs. (16) and (17). It is believed that the correction factor takes care of the effects of the surface properties of the ore and the physiochemical interaction between the ore and the leaching solution which (affected experimental results) were not considered during the model formulation.

Based on the foregoing, Fig. 7 indicates that correction factors of 0.21 and -2.37% make up for the least and highest deviation of -0.21 and 2.37% resulting from application of initial solution pH 4.66 and 3.89 respectively. It is pertinent to state that the actual deviations are just the modulus of the values. The role of the sign attached to the values is just to show when the deviation is surplus or deficit.



**Fig. 7. Variation of Correction Factor (To Model Predicted Initial Solution pH) with the Actual Initial Solution pH.**

## 6. Conclusion

The model predicts the initial solution pH at pre-assumed final solution pH and concentration of dissolved lead during leaching of Ishiagu (Nigeria) galena in butanoic acid solution. This prediction could be done during the leaching process providing the expectant final pH of the solution and concentration of dissolved lead are known. The validity of the model is rooted in the expression  $e^{Ny/\alpha} = \sqrt[3]{PB}$  where both sides of the expression are approximately equal to 5. The maximum deviation of the model-predicted initial solution pH value from that of the corresponding experimental value is less than 3% which is quite within the acceptable deviation limit of experimental results.

The justification for applicability of the presently derived model stems on the fact that some output process parameters such as pH of leaching solutions during leaching of galena can be determined directly by substituting the appropriate input process parameters into the associated models without using any equipment. Past derivations involving leaching of iron ore [17-19] complement the fact that the pH of leaching solutions can also be determined without actually using any recordable meters.

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