

MODEL FOR CALCULATING THE CONCENTRATION OF DISSOLVED IRON RELATIVE TO THE WEIGHT-INPUT OF IRON OXIDE ORE AND FINAL SOLUTION PH DURING NITRIC ACID LEACHING

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Abstract

Model for calculating the concentration of dissolved iron during leaching of iron oxide ore in nitric acid solution has been derived. The model was found to be dependent on the value of the weight-input of iron oxide ore and final solution pH measured during the leaching process. It was observed that the validity of the model is rooted on the expression where both sides of the relationship are correspondingly approximately almost equal. The maximum deviation of the model-predicted dissolved %Fe values from the corresponding experimental values was found to be 28% which is quite within the acceptable range of deviation limit of experimental results. Dissolved iron concentration per unit mass of iron oxide ore input evaluated from experimental and model-predicted results are 0.0010%/g and 0.0011%/g respectively, indicating proximate agreement.

Keywords: Model, Iron Dissolution, Nitric Acid, Iron Oxide Ore, Leaching.

1. Introduction

Assessment and evaluation of the prospect of several organic and inorganic acids in dissolving iron have been carried out. The dissolution of iron oxides and oxyhydroxides in hydrochloric and perchloric acids has been evaluated [1]. Usage of synthetic metal-containing goethite and haematite has also been evaluated [2]. Studies [3] has been carried out on the effect of EDTA and Fe(II) during the dissolution of magnetite. The industrial use of sulphuric acid and other inorganic acids to dissolve iron oxide has not fared too well. Researchers [4] have studied the dissolution of goethite in several inorganic acids belonging to the families of the carboxylic and diphosphoric acids in the presence of reducing agents.

Nomenclatures

| | |
|----------------------|---------------------------------------------------------------------------------------------------|
| C_t | Correction factor for the model |
| D_i | Dissolution of iron per unit mass of iron oxide ore, %/g |
| D_v | Deviation in the model |
| E_v | Experimental %Fe values |
| K_1 | Dissolution constants of Fe |
| K_2 | Dissolution constants of Fe_2O_3 |
| K_N | Haematite dissolution constant (= 4.57) |
| N | 0.0043 (Nitric acid leachability constant during leaching of iron oxide ore) |
| P_v | Model- predicted %Fe values |
| Q | Quantity of heat absorbed by oxalic acid solution, J |
| R^2 | Correlation Coefficient |
| T | Leaching temperature, °C |
| t | Time elapsed during dissolution of pre-quantified concentration of phosphorus in oxalic acid, hrs |
| <i>Greek Symbols</i> | |
| α | Final pH of the leaching solution (in presence of dissolved phosphorus) |
| γ | Final pH of the leaching solution at time t |
| ΔE | Change in the concentrations of iron dissolved |
| μ | Weight-input of iron oxide ore during the leaching process, g |

The effectiveness of several organic acids (such as acetic, formic, citric, ascorbic acids, etc.) used for dissolving iron from iron compounds has also been investigated by evaluation [5]. Oxalic acid was found to be the most promising because of its acid strength, good complexing characteristics and high reducing power, compared to other organic acids. Using oxalic acid, the dissolved iron can be precipitated from the leach solution as ferrous oxalate, which can be re-processed to form pure haematite by calcinations [6]. Several researchers [7-13] have studied the use of oxalic acid to dissolve iron oxide on a laboratory scale. Iron dissolution was found to reach 90% for a 20% slurry within 60 minutes using 0.19M oxalic for the finer fraction (<150 μm) containing 0.56% Fe_2O_3 [14]. The coarser fraction (>150 μm) containing 1.06% Fe_2O_3 achieved a lower iron removal, reaching a steady state of only 78% after 1 h of leaching. Although the pH was not measured or controlled, it was expected that the liquor pH is < pH 1 at the oxalic acid concentration range studied (0.19-0.48). It was found that the maximum iron dissolution of only 40% is within 3 h at temperatures in the range 90-100°C [6]. At 0.5M oxalate and all temperatures (25, 60 and 80°C) the dissolution of iron was faster at a lower pH in the range pH 1-5 studied. Several researchers have evaluated biological processes for iron dissolution based on the use of several micro organisms that were easily sourced and isolated. Recently, findings were presented on the study of the use of *Aspergillus niger* and their cultural filtrates for dissolving iron present in iron compounds [15].

Derivation of a model carried out for evaluating the final pH of the leaching solution during leaching of iron oxide ore in oxalic acid solution [16]. The model

evaluates the pH value as the sum of two parts, involving the % concentrations of Fe and Fe₂O₃ dissolved. The model can be expressed as

$$\gamma = 0.5 \left(\frac{K_1}{\%Fe} + \frac{K_2}{\%Fe_2O_3} \right) \quad (1)$$

where

K_1 and K_2 are dissolution constants of Fe and Fe₂O₃ respectively, and

γ is the final pH of leaching solution after time t .

It was also found that the model of [16] could predict the concentration of Fe or Fe₂O₃ dissolved in the oxalic acid solution at a particular final solution pH by taking Fe or Fe₂O₃ as the subject formulas. The prevailing process conditions under which the model works include: leaching time of 30 minutes, constant leaching temperature of 30°C, average ore grain size 150 µm and 0.1M oxalic acid.

It has been reported [17] that the heat absorbed by oxalic acid solution during leaching of iron oxide ore can be predicted using a model which works under the following process conditions; initial pH 6.9, average ore grain size 150 µm and leaching temperature 30°C. The model [17] can be stated as

$$Q = K_N \frac{\gamma}{\%Fe_2O_3} \quad (2)$$

where

Q is the quantity of heat absorbed by oxalic acid solution at the leaching process J ,

$\%Fe_2O_3$ is the concentration of haematite dissolved in oxalic acid solution, and

$K_N = 4.57$, is the haematite dissolution constant in oxalic acid solution, determined in the experiment [17].

Further work [17] carried out on the model using the same process conditions indicates that on re-arranging the model as

$$\%Fe_2O_3 = K_N \frac{\gamma}{Q} \quad (3)$$

the predicted concentrations of haematite deviated very insignificantly from the corresponding experimental values. In this case, the value of Q was calculated by considering the specific heat capacity of oxalic acid. Values of heat absorbed by the oxalic acid solution during the leaching of iron oxide ore as predicted by the model [17] agree with the experimental values that the leaching process is endothermic. This is because all the predicted values of the heat absorbed by the oxalic acid solution were positive. The model shows that the quantity of heat absorbed by oxalic acid solution during the leaching process is directly proportional to the final pH of the solution and inversely proportional to the concentration of haematite dissolved.

Calculation of the concentration of leached iron during leaching of iron oxide ore in sulphuric acid solution has been achieved through application of a model [18]. The model is expressed as

$$\%Fe = e^{-2.0421 \ln T} \quad (4)$$

It was found that the predicted concentration of leached Fe is very close to the values obtained from the experiment. The model shows that the concentration of leached Fe is dependent on the values of the final leaching solution temperature measured during the leaching process. It was observed that the validity of the model is rooted in the expression $\ln(\%Fe) = N(\ln T)$ where both sides of the expression are correspondingly approximately equal. The maximum deviation of the model-predicted values of %Fe (leached) from those of the experimental values was found to be less than 37%.

A model for predicting the final solution pH at determined initial pH and leaching time during leaching of iron oxide ore in hydrogen peroxide solution has been derived [19]. It was observed that the validity of the model is rooted in the mathematical expression; $(\ln T)^{1/2} = N(\beta^C/\alpha^C)$ where both sides of the relationship are approximately equal to 2. The model is expressed as

$$\beta = \text{Antilog} [0.2439 \log(\alpha^{4.1} (\ln T)^{1/2} / 3.6)] \quad (5)$$

The model shows that the initial solution pH is dependent on the values of the final solution pH and leaching time. The respective positive or negative deviation of the model-predicted final pH from its corresponding experimental value was found to be less than 8%, which is quite within the acceptable deviation limit of experimental results depicting the validity of the model.

Successful attempt has been made [20] to derive a model for predictive analysis of the concentration of dissolved iron during leaching of iron oxide ore in sulphuric acid solution. The model expressed as;

$$\%Fe = 0.987(\mu/T) \quad (6)$$

was found to predict the concentration of dissolved Fe with high degree of precision. It was found that the model is dependent on the values of the leaching temperature and weight of iron oxide ore added. The validity of the model was found to be rooted in the expression $\%Fe = N(\mu/T)$ where both sides of the relationship are correspondingly approximately equal. The maximum deviation of the model-predicted concentration of dissolved Fe from those of the experimental values was found to be less than 19% which is quite within the acceptable range of deviation limit for experimental results, hence depicting the usefulness of the model as a tool for predictive analysis of the dissolved iron during the process.

It has been reported [21] of a model which calculates the solution pH during hydrogen peroxide leaching of iron oxide ore. The validity of the model was found to be rooted in the expression $\ln \gamma = K_c(\%Fe_2O_3/\%Fe)^N$ where both sides of the equation are correspondingly approximately equal to 2. The model is expressed as

$$\gamma = e^{K_c \left(\frac{\%Fe_2O_3}{\%Fe} \right)^N} \quad (7)$$

The final solution pH was found to be dependent on the values of the % concentrations of dissolved iron and haematite from experiment. The respective deviation of the model-predicted pH values from the corresponding experimental values was found to be less than 20%.

The aim of this work is to derive a model for calculating the concentration of dissolved iron relative to the weight-input of iron oxide ore and final solution pH during nitric acid leaching of Agbaja (Nigeria) iron oxide ore.

2. Model

Iron ore (being in solid phase) was assumed to be stationary during the leaching process. Leaching occurred as a result of the attack on the ore by hydrogen ions from the nitric acid within the liquid phase (in the presence of oxygen).

Model formulation

Results of previous research work [22] carried out were used for this work. Statistical and computational analysis of these results [22] presented in Table 1, gave rise to Table 2 which indicate that;

$$\%Fe = N \mu / \gamma \tag{8}$$

Introducing the value of *N* into Eq. (8)

$$\%Fe = 0.0043 \mu / \gamma \tag{9}$$

where

%Fe is the concentration of dissolved iron during the leaching process,

N = 0.0043, is nitric acid leachability constant during leaching of iron oxide ore determined in the experiment [22],

μ is the weight-input of iron oxide ore during the leaching process (g), and

γ is the final pH of leaching solution at the time *t*, when %Fe is evaluated.

Table 1. Variation of Concentration of Dissolved Iron with Weight-Input of Iron Oxide Ore and Final Solution pH [22].

| %Fe | μ | γ |
|--------|-------|----------|
| 0.0025 | 2 | 4.88 |
| 0.0027 | 3 | 4.75 |
| 0.0040 | 5 | 4.44 |
| 0.0066 | 8 | 4.25 |
| 0.0084 | 9 | 4.23 |
| 0.0099 | 10 | 4.21 |

Table 2. Variation of %Fe with $N\mu/\gamma$.

| %Fe | $N\mu/\gamma$ |
|--------|---------------|
| 0.0025 | 0.0018 |
| 0.0027 | 0.0027 |
| 0.0040 | 0.0048 |
| 0.0066 | 0.0081 |
| 0.0084 | 0.0091 |
| 0.0099 | 0.0102 |

3. Boundary and Initial Conditions

In a cylindrical flask of height 30 cm, iron oxide ore was placed prior to the addition of nitric and oxalic acid which was used as leaching solutions. Initially, the flask was assumed to be free of bacteria and other micro organisms. It was assumed that atmospheric oxygen affected the process initially. Weights input of iron oxide ore considered for the work ranged from 2-10 g. Other process conditions used include: initial pH of leaching solution, 5.0, leaching time; 3 hrs, leaching temperature of 25°C, average ore grain size of 150 µm, nitric acid concentration at 0.16 mol/litre. The boundary conditions considered for the model formulation were assumption of a zero gradient for the liquid scalar and also gas phase at the top of the particles.

It was also assumed that atmospheric oxygen interacted with the non flowing leaching solution and also with the top and bottom part of the ore particles (which were in the gas and liquid phases respectively.) The sides of the particles were assumed to be symmetrical. These process conditions are presented in details in the report [22].

4. Model Validation

The validity of model was established by calculating the deviation of the model-predicted %Fe values from values obtained from the experimental work [22] carried out.

It was believed that deviations of model-predicted %Fe values from the corresponding experimental values resulted from non-consideration (during model formulation) of the surface properties of the ore and the physiochemical interactions between the ore and leaching solution which were found to have played vital roles during the leaching process [22]. Based on the foregoing, it is expected that a correction factor be added to the model-predicted values to make up for those factor neglected during the model formulation.

The deviation, D_v (%), of model-predicted %Fe values from the corresponding experimental %Fe values is expressed as

$$D_v = \left(\frac{P_v - E_v}{E_v} \right) \times 100 \quad (10)$$

where

P_v is the model- predicted %Fe values, and

E_v is the experimental %Fe values.

On the other hand, correction factor, C_t , is expressed as the negative of the deviation. Therefore

$$C_t = -D_v \quad (11)$$

Substituting Eq. (10) into Eq. (11)

$$C_t = -\left(\frac{P_v - E_v}{E_v}\right) \times 100 \quad (12)$$

Addition of C_t values obtained from Eq. (12) to the model-predicted values of %Fe gives exactly %Fe values as obtained from the experiment [22].

5. Results and Discussion

Dissolution of iron per unit mass of iron oxide ore added during the leaching process was determined following comparison of the dissolved iron per unit mass of iron oxide ore obtained by calculations involving experimental results, and that obtained directly from the model. Dissolution of iron per unit mass of iron oxide ore added, D_i (%/g) was calculated from the equation;

$$D_i = D/\mu \quad (13)$$

Therefore, a plot of concentration of dissolved iron against the mass of iron oxide ore added, Fig. 1, using experimental results in Table 1, gives a slope, S at points (3, 0.0027) and (9, 0.0084) following their substitution into the mathematical expression

$$S = \Delta D/\Delta\mu \quad (14)$$

Equation (14) is detailed as

$$S = (D_2 - D_1)/(\mu_2 - \mu_1) \quad (15)$$

where

ΔE is the change in the concentrations of iron dissolved D_2, D_1 at two different weight-input values μ_2, μ_1 . Considering the points (3, 0.0027) and (9, 0.0084) for (μ_1, D_1) and (μ_2, D_2) , respectively, and substituting them into Eq. (15), gives the slope as 0.0010%/g which is the concentration of dissolved iron per unit mass of iron oxide ore used during the actual experimental leaching process.

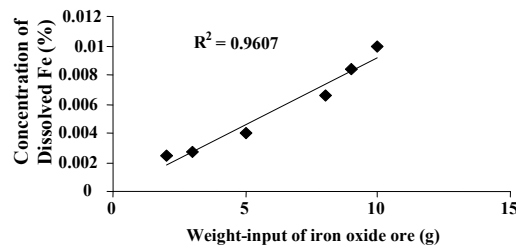


Fig. 1. Effect of Weight-Input of Iron Oxide Ore on the Concentration of Dissolved Iron as Obtained from Experiment.

Also similar plot, Fig. 2, using model-predicted results gives a slope. Considering points (3, 0.0027) and (9, 0.0091) for (μ_1, D_1) and (μ_2, D_2) , respectively and substituting them into Eq. (15) gives the value of slope, S as 0.0011%/g. This is the model-predicted concentration of dissolved iron per unit mass of iron oxide ore used for the leaching process. A comparison of these two values of dissolved iron

concentrations per unit mass of iron oxide ore used shows proximate agreement. This indicates a very high degree of validity for the model.

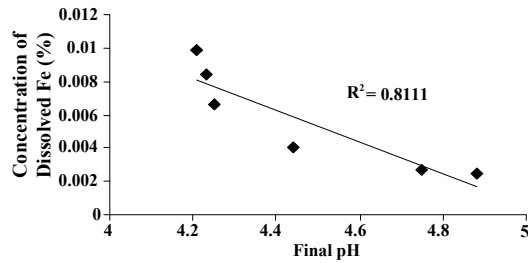


Fig. 2. Effect of Final pH on the Concentration of Dissolved Iron as Obtained from Experiment.

An ideal comparison of the concentration of dissolved iron per unit mass of iron oxide ore used 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. The values of the correlation coefficient, R^2 calculated from the equation

$$R = \sqrt{R^2} \tag{16}$$

using R^2 values (coefficient of determination) from Figs. 1-4 show a better correlation for model-predicted concentration of dissolved iron in relation to weight-input and final pH; (0.9998) and (0.9635) respectively compared to that from the experiment [22] (0.9802), (0.9006).

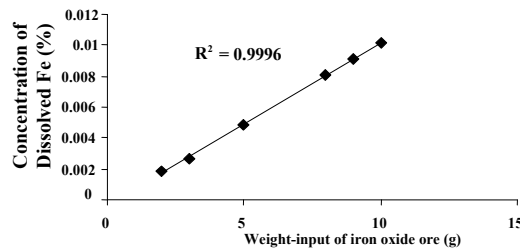


Fig. 3. Effect of Weight-Input of Iron Oxide Ore on the Concentration of Dissolved Iron as Predicted by Derived Model.

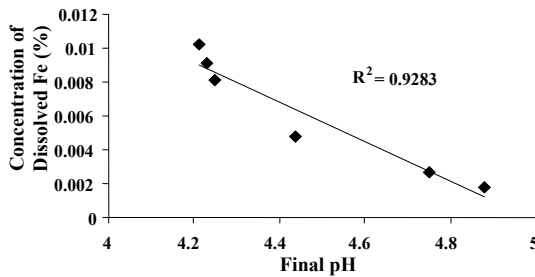


Fig. 4. Effect of Final pH on the Concentration of Dissolved Iron as Predicted by Derived Model.

This shows that the model can predict more reliable, realistic and accurate concentrations of dissolved iron for this process despite its deviation from experimental values. These correlation values also show that the model-predicted concentrations of dissolved iron are very much in proximate agreement with the corresponding dissolved iron concentration obtained from experiment [22]. Figure 3 shows that the weight-input of iron oxide ore contributed more significantly to the validity of the model compared with the final solution pH (Fig. 4). This is shown in their respective R^2 values.

Comparison of Figs. 5 and 6 show that both values of the dissolved iron concentration obtained from the experiment [22] (line *ExD*) and the derived model (line *MoD*) in relation to both the weight-input of iron oxide ore and final solution pH are generally quite close hence depicting proximate agreement and validity of the model. However, Figs. 5 and 6 show direct and inverse relationship respectively in agreement with Table 1 which is made up of data from the experiment [22].

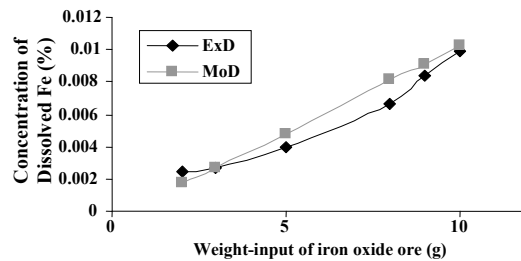


Fig. 5. Comparison of the Concentrations of Dissolved Iron in Relation to Weight of Iron Oxide Ore as Obtained from Experiment and Derived Model.

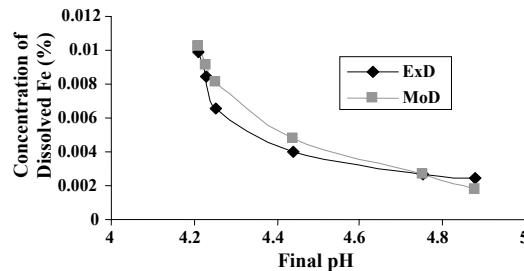


Fig. 6. Comparison of the Concentrations of Dissolved Iron in Relation to Final pH as Obtained from Experiment and Derived Model.

Effect of final solution pH and weight-input of iron oxide ore on the deviation (from experimental values) of model-predicted concentration of dissolved iron

It was found that the validity of the model is rooted in the expression (Eq. 8) where both sides of the expression are correspondingly approximately almost

equal and also the values agree with Table 2 which are evaluated from Table 1 as a result of the corresponding computational analysis.

The maximum deviation of the model-predicted concentration of dissolved iron from the corresponding experimental value is 28% which is quite within the acceptable deviation range of experimental results, hence depicting the usefulness of the model. The positive and negative deviations (of the model-predicted concentration of dissolved iron) from actual experimental values show an undulating relationship with the final solution pH, the weight-input of iron oxide ore and the actual concentration of dissolved iron (Figs. 7- 9).

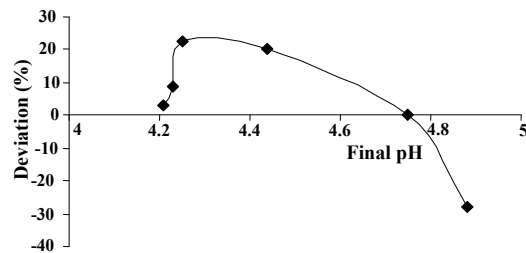


Fig.7. Variation of Deviation (from experimental values) of Model-Predicted Concentrations of Dissolved Iron with Final pH.

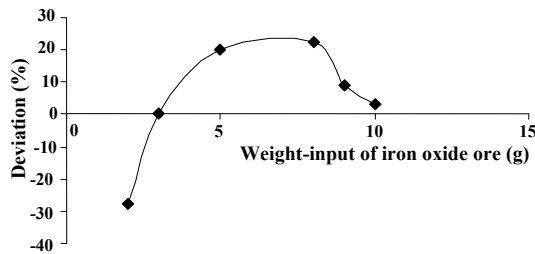


Fig.8. Variation of Deviation (from experimental values) of Model-Predicted Concentration of Dissolved Iron with Weight-Input of Iron Oxide Ore.

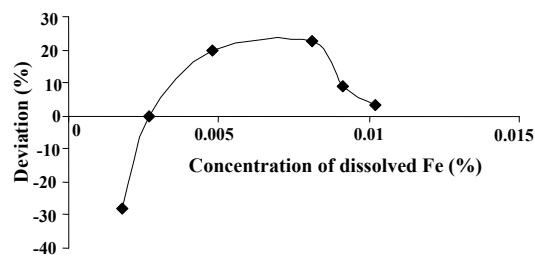


Fig.9. Variation of Deviation (from experimental values) of Model-Predicted Concentrations of Dissolved Iron with the Actual Model-Predicted Concentration of Dissolved Iron.

Figures 7-9 indicate that the highest and least deviations (-28 and 3.03%) which are same in relation to both the final solution pH obtained (at the end of the leaching process) and the weight-input of iron oxide ore corresponds to the model-predicted dissolved iron concentrations 0.0018 and 0.0102% respectively. Comparison of Figs. 7-8 shows that these percent deviations also correspond to the final solution pH; 4.88 and 4.21 and also iron oxide ore weight-input; 2 and 10 g respectively.

Effect of final solution pH and weight-input of iron oxide ore on the correction factor to the model-predicted concentration of dissolved iron

Figures 10 and 11 also show that correction factor to the model-predicted concentration of dissolved iron depict an undulating relationship with the final solution pH and weight-input of iron oxide ore. Comparison of Figs. 3, 4, 10 and 11 indicates that the highest and least correction factors (28 and -3.03%) which are same in relation to both the final solution pH obtained (at the end of the leaching process) and the weight-input of iron oxide ore also corresponds to the dissolved iron concentrations 0.0018 and 0.0102% respectively.

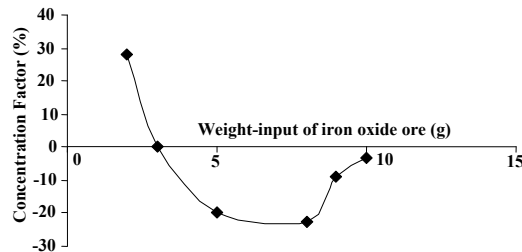


Fig. 10. Variation of Correction Factor to Model-Predicted Concentration of Dissolved Iron with Weight-Input of Iron Oxide Ore.

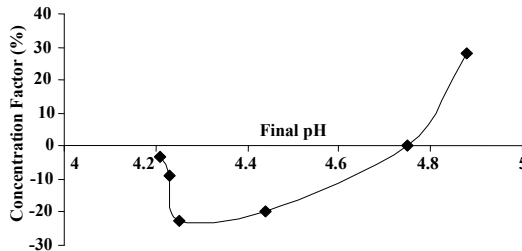


Fig. 11. Variation of Correction Factor to Model-Predicted Concentration of Dissolved Iron with Final pH.

The percent correction factors also correspond to the final solution pH; 4.88 and 4.21 as well as iron oxide ore weight-input; 2 and 10 g respectively. Comparison of Figs. 7, 8, 10 and 11 shows that the orientation of the curves of the correction factor against final pH and weight-input of iron oxide ore are opposite that of the deviation against final pH and weight-input of iron oxide ore. This is attributed to the fact that correction factor is the negative of the deviation as shown in Eqs. (11) and (12). 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.

6. Conclusions

The model predicts the concentration of iron dissolved during leaching of iron oxide ore in nitric acid solution. The validity of the model is rooted on the expression $\%Fe = N(\mu/a)$ where both sides of the expression are correspondingly approximately almost equal. The maximum deviation of the model-predicted %Fe values from the corresponding experimental %Fe values is 28% which is quite within the acceptable range of deviation limit of experimental results. The two values of dissolved iron concentrations per unit mass of iron oxide ore used as obtained from experiment and derived model show proximate agreement hence indicating a very high degree of validity for the model.

It is expected that more process parameters should be incorporated into the model in further works with the aim of reducing the deviations of the model-predicted %Fe values from those of the experiment.

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