Model for Predicting the Concentration of Iron Dissolved during Nitric Acid Leaching of Iron Oxide Ore in Oxalic Acid Solution

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Abstract: Model for predicting the concentration of iron dissolved during nitric acid leaching of iron oxide ore in oxalic acid solution has been derived. The model;

$$\%Fe = 0.0133\left(\frac{\alpha}{\mu}\right)$$

was found to depend on the value of the final solution pH and weight-input of iron oxide ore during the experiment. It was observed that the validity of the model is rooted on the expression $\%Fe = N(\alpha/\mu)$ 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 30% 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 0.0058%/g and 0.006%/g respectively, indicating proximate agreement. [New York Science Journal. 2009;2(6):1-12]. (ISSN: 1554-0200).

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

1. Introduction

The prospect of several organic and inorganic acids in dissolving iron has been evaluated in several studies. Sidhu et al.[1] evaluated the dissolution of iron oxides and oxyhydroxides in hydrochloric and perchloric acids. Lim-Nunez and Gilkes [2] used synthetic metal-containing goethite and haematite in their evaluation while Borghi et al. [3] studied 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. Chiarizia and Hotwitz [4] 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. Ambikadevi and Lalithambika [5] evaluated the effectiveness of several organic acids (such as acetic, formic, citric, ascorbic acids etc.) used for dissolving iron from iron compounds. Oxalic acid was found to be the most promising because of its acid strength, good comlexing 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]. Many researchers have studied the use of oxalic acid to dissolve iron oxide on a laboratory scale [7-13]. Lee et al [14] used 0.19-0.48M oxalic acid to dissolve hydrated iron oxide. Iron dissolution was found [14] to reach 90% for a 20% slurry within 60mins. using 0.19M oxalic for the finer fraction (< 150μm) containing 0.56% Fe_2O_3. The coarser fraction (>150μm) 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 < pH1 at the oxalic acid concentration range studied (0.19-0.48). Taxiarchou et al.[6] found that the maximum iron dissolution of only 40% is within 3 h at temperatures in the range 90-100^{0}C. At 0.5M oxalate and all temperatures (25, 60 and 80^{0}C) the dissolution of iron was faster at a lower pH in the range pH 1-5 studied. Biological processes for iron dissolution have been evaluated by several researchers based on the use of several micro organisms that were easily sourced and isolated. Mandal and Banerjee [15] recently presented their findings on the study of the use of Aspergillus niger and their cultural filtrates for dissolving iron present in iron compounds.

Nwoye [16] derived a model for evaluating the final pH of the leaching solution during leaching of iron oxide ore in oxalic acid solution. The model evaluates the pH value as the sum of two parts, involving the % concentrations of Fe and Fe_2O_3 dissolved. The model can be expressed as;

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

Where

K_1 and K_2 = dissolution constants of Fe and Fe_2O_3 respectively.

γ = final pH of leaching solution (after time t).

It was also found that the model [16] could predict the concentration of Fe or Fe_2O_3 dissolved in the oxalic acid solution at a particular final solution pH by taking Fe or Fe_2O_3 as the subject formular. The prevailing process conditions under which the model works include: leaching time of 30mins., constant leaching temperature of 30°C, average ore grain size; 150μm and 0.1M oxalic acid.

Nwoye [17] has reported that the heat absorbed by oxalic acid solution during leaching of iron oxide ore can be predicted using the model he derived which works under the process condition; initial pH 6.9, average ore grain size; 150μm and leaching temperature; 30^{0}C. The model [17] can be stated as

$$Q = K_N \left(\frac{\gamma}{\%Fe_2O_3} \right) \tag{2}$$

Where

Q = Quantity of heat absorbed by oxalic acid solution during the leaching process. (J)

γ = Final pH of the leaching solution (at time t).

%Fe_2O_3 = Concentration of haematite dissolved in oxalic acid solution during the leaching process.

K_N = 4.57(Haematite dissolution constant in oxalic acid solution) determined in the experiment [17].

Nwoye [17] carried out further work on the model using the same process conditions and observed that on re-arranging the model as;

$$\%Fe_2O_3 = K_N\left(\frac{\gamma}{Q}\right) \tag{3}$$

the concentrations of haematite predicted 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.

Nwoye et al.[18] derived a model for calculating the concentration of leached iron during leaching of iron oxide ore in sulphuric acid solution. The model is expressed as;

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

The model was found to predict %Fe (leached) very close to the values obtained from the experiment, being 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 positive or negative deviation of each of the model-predicted values of %Fe (leached) from those of the experimental values was found to be less than 37%.

Nwoye et al.[19] derived 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. 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;

$$\beta = Antilog[0.2439Log(\alpha^{4.1}(\ln t)^{1/2}/3.6)] \tag{5}$$

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.

Model for predictive analysis of the concentration of dissolved iron during leaching of iron oxide ore in sulphuric acid solution was derived by Nwoye et al.[20]. The model expressed as;

$$\%Fe = 0.987(\mu/T) \tag{6}$$

was found to predict %Fe dissolved with high degree of precision being dependent on the values of the leaching temperature and weight of iron oxide ore added. It was observed that the validity of the model is rooted in the expression $\%Fe = N(\mu/T)$ where both sides of the relationship are correspondingly approximately equal. The positive or negative deviation of each of the model-predicted values of %Fe

(dissolved) 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.

Model for calculating the solution pH during hydrogen peroxide leaching of iron oxide ore has also been derived by Nwoye et al [21]. It was observed that the validity of the model is rooted in the expression $In\gamma = K_C[(\%Fe_2O_3/\%Fe)^N]$ where both sides of the equation are correspondingly approximately equal to 2. The model expressed as;

$$\gamma = \exp\left[K_C[(\%Fe_2O_3/\%Fe)^N]\right] \qquad (7)$$

The final solution pH was found to depend 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% which is quite within the acceptable range of deviation limit of experimental results.

The aim of this work is to derive a model for predicting the concentration of iron dissolved during nitric acid leaching of Agbaja (Nigeria) iron oxide ore in oxalic acid solution.

2. Model

During the leaching process, the iron ore (being in solid phase) was assumed to be stationary. The leaching occurs as a result of the attack on the ore by hydrogen ions from the nitric and oxalic acid within the liquid phase (in the presence of oxygen).

2.1 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;

$$\%Fe = N\left(\frac{\alpha}{\mu}\right) \qquad \text{(approximately)} \qquad (8)$$

Introducing the value of N into equation (8)

$$\%Fe = 0.0133\left(\frac{\alpha}{\mu}\right) \qquad (9)$$

Where

%Fe = Concentration of dissolved iron during the leaching process.

N = 0.0133 (Oxalic-nitric acid leachibility ratio during leaching of iron oxide ore)

determined in the experiment [22].

(μ) = Weight-input of iron oxide ore during the leaching process.(g)

(α) = Final pH of leaching solution at the time t, when %Fe is evaluated.

Equation (9) is the derived model.

Table 1: Variation of concentration of dissolved iron with weight-input of iron oxide ore and final solution pH.[22]

%Fe	(µ)	(α)
0.0377	2	6.10
0.0304	3	5.82
0.0193	4	5.80
0.0185	5	5.76
0.0098	6	5.71
0.0087	7	5.64

Table 2: Variation of %Fe with N(α/µ)

%Fe	N(α/µ)
0.0377	0.0406
0.0304	0.0258
0.0193	0.0193
0.0185	0.0153
0.0098	0.0127
0.0087	0.0107

3. Boundary and Initial Condition

In a cylindrical flask of height; 30cm, iron oxide ore was placed prior to the addition of nitric and oxalic acid which were 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 2-7g. Other process conditions used include: initial pH of leaching solution; 6.0, leaching time; 30 minutes, leaching temperature of 25°C, average ore grain size; 150µm, nitric and oxalic acid concentrations at 0.08 and 0.05mol/litre respectively.

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 (Dv) (%) of model-predicted %Fe values from the corresponding experimental %Fe values is expressed as;

$$Dv = \frac{Pv - Ev}{Ev} \times 100 \qquad (10)$$

Where   Pv = Model- predicted %Fe values
        Ev = Experimental %Fe values

On the other hand, correction factor (Ct) is expressed as the negative of the deviation. Therefore

$$Ct = -Dv \qquad (11)$$

Substituting equation (10) into equation (11)

$$Ct = -100 \left(\frac{Pv - Ev}{Ev} \right) \qquad (12)$$

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

5. Results and Discussion

The derived model is equation (9).

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;

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

Therefore, a plot of concentration of dissolved iron against the mass of iron oxide ore added, as in Fig. 1 using experimental results in Table 1, gives a slope, S at points (2, 0.0377) and (7, 0.0087) following their substitution into the mathematical expression;

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

Eqn. (14) is detailed as

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

Where

ΔE = Change in the concentrations of iron dissolved D_2, D_1 at two different weight-input values μ_2, μ_1. Considering the points (2, 0.0377) and (7, 0.0087) for (μ_1, D_1) and (μ_2, D_2,) respectively, and substituting them into eqn. (15), gives the slope as -0.0058%/g which is the concentration of dissolved iron per unit mass of iron oxide ore used during the actual experimental leaching process. Also similar plot (as in Fig. 2) using model-predicted results gives a slope. Considering points (2, 0.0406) and (7, 0.0107) for (μ_1, D_1) and (μ_2, D_2,) respectively and substituting them into eqn. (15) gives the value of slope, S as -0.0060%/g. This is the model-predicted concentration of dissolved iron per unit mass of iron oxide ore used for the leaching process. The negative sign preceding both 0.0058 and 0.0060 is not part of the values of the concentrations of dissolved iron per unit mass of iron oxide ore
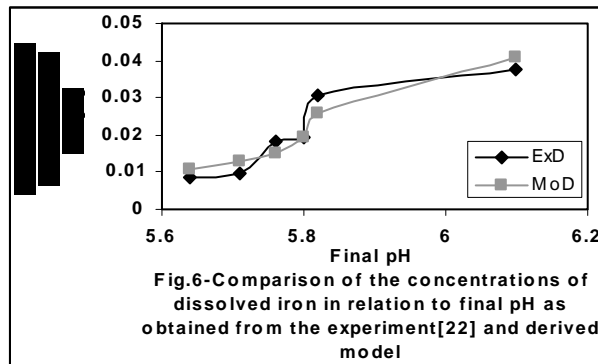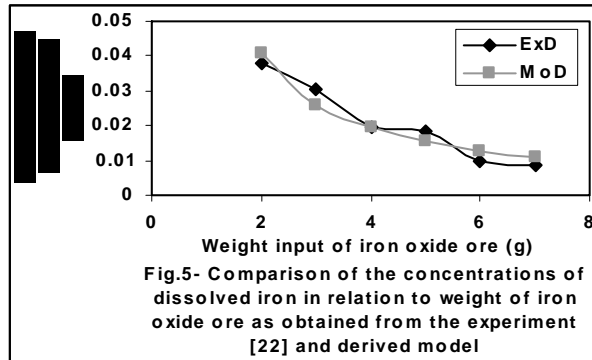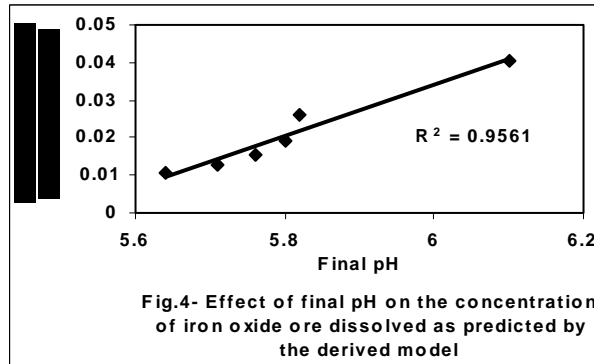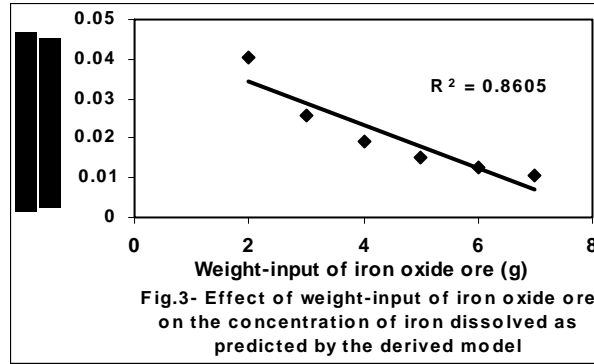
used as obtained from the experiment [22] and derived model but indicative of the inverse relationship between the concentrations of dissolved iron and the weight–input of iron oxide ore as obtained in the experiment [22] and the derived model which resulted to negative slopes. A comparison of these 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.

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 calculated from the equation;

Fig.1- Effect of weight-input of iron oxide ore on the concentration of iron dissolved as obtained from experiment [22]

Fig.2- Effect of final pH on the concentration of iron dissolved as obtained from experiment [22]
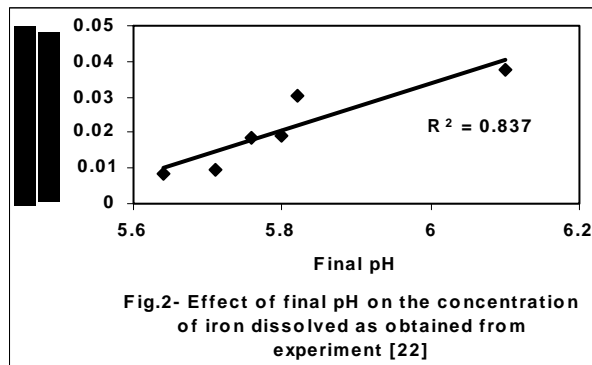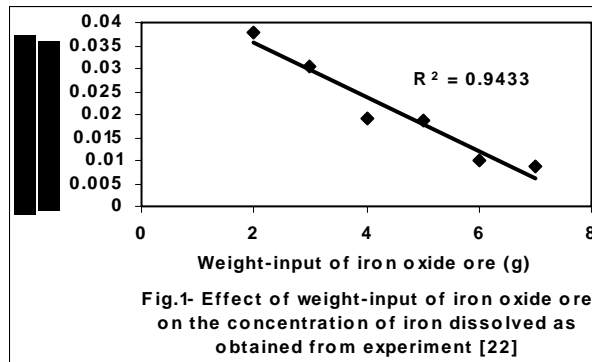
$$R = \sqrt{R^2} \hspace{3cm} (16)$$

using the r-squared values (coefficient of determination) from Figs.1-4 show a very close correlation;(0.9712),(0.9149) and (0.9276),(0.9778) between values of the concentration of dissolved iron obtained from experiment and derived model respectively. This also shows that the model- predicted concentrations of dissolved iron are very much in proximate agreement with the corresponding dissolved iron concentration obtained from experiment [22]. Fig.4 shows that final pH contributed more significantly to the validity of the model compared with the weight-input of iron oxide ore (Fig.3). This is shown in their respective R^2 values.

Fig.3- Effect of weight-input of iron oxide ore on the concentration of iron dissolved as predicted by the derived model

Fig.4- Effect of final pH on the concentration of iron oxide ore dissolved as predicted by the derived model

Fig.5- Comparison of the concentrations of dissolved iron in relation to weight of iron oxide ore as obtained from the experiment [22] and derived model

Fig.6-Comparison of the concentrations of dissolved iron in relation to final pH as obtained from the experiment[22] 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

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 the reliability and validity of the model. However, Fig.5 and 6 show inverse and direct relationship respectively in agreement with Table 1 which is made up of data from the experiment [22].

It was found that the validity of the model is rooted in the expression %Fe = N(α/μ) where both sides of the expression are correspondingly approximately almost equal. Table 2 also agrees with equation (8) following the values of %Fe = N(α/μ) 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 30% 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 distinct 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[22]) of model-predicted concentration of dissolved iron with the final pH

Fig.8- Variation of deviation (from experimental values[22]) of model-predicted concentration of dissolved iron with weight-input of iron oxide ore
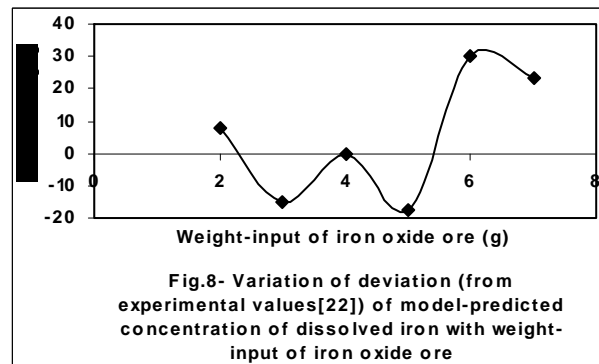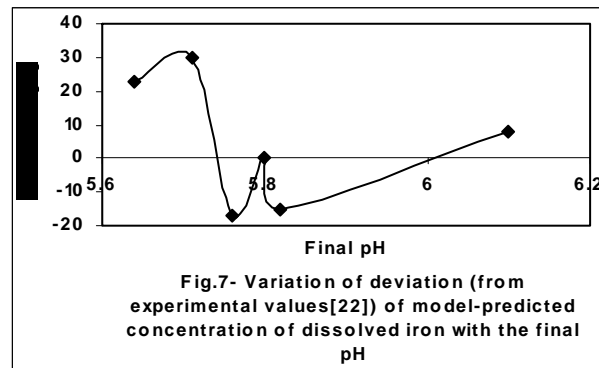
Figs.7-9 indicate that the highest and least deviations (30 and 7.7%) which are same in relation to both the final solution pH obtained (at the end of the leaching process) and the weight-input of iron oxide ore corresponds to the model-predicted dissolved iron concentrations 0.0127 and 0.0406% respectively.

Comparison of Figs. 7-8 shows that these percent deviations also correspond to the final solution pH; 5.71 and 6.1 and also iron oxide ore weight-input; 6 and 2g respectively.

Fig.9- Variation of deviation (from experimental values [22]) of model-predicted concentration of dissolved iron with the actual model-predicted concentration of dissolved iron
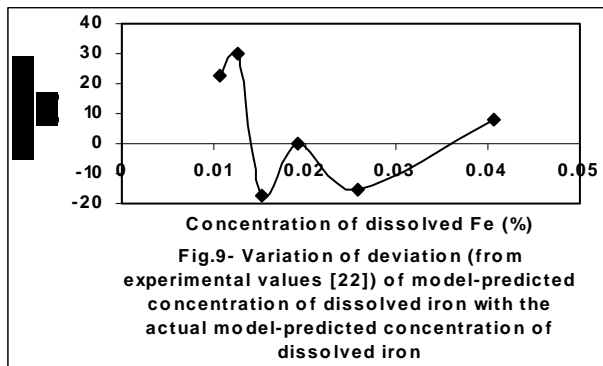
Effect of final solution pH and weight-input of iron oxide ore on the correction factor to the model-predicted concentration of dissolved iron

Figs. 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. Figs.3, 4, 10 and 11 indicate that the highest and least correction factors (-30 and -7.7%) 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 0.0127 and 0.0406% respectively.

Fig.10- Variation of correction factor to model-predicted concentration of dissolved iron with the weight-input of iron oxide ore

Fig.11-Variation of correction factor to model-predicted concentration of dissolved iron with the final pH
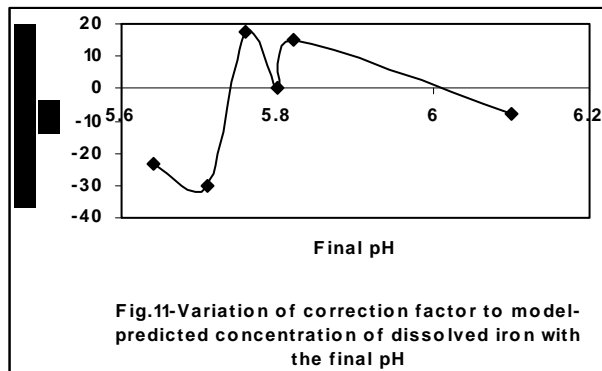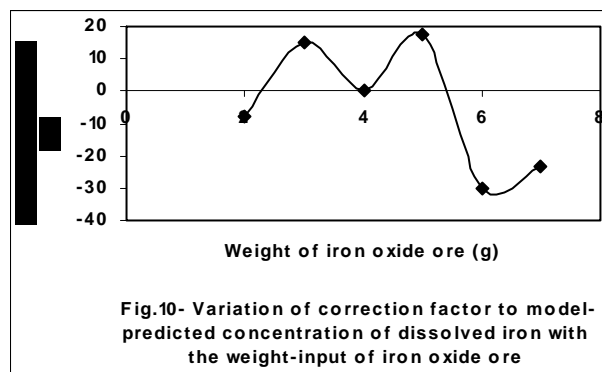
The percent correction factors also correspond to the final solution pH; 5.71 and 6.1 as well as iron oxide ore weight-input; 6 and 2g respectively.

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 eqns. (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. Conclusion

The model predicts the concentration of iron dissolved during nitric acid leaching of iron oxide ore in oxalic acid solution. The validity of the model is rooted on the expression %Fe = N(α/μ) 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 30% 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.

Acknowledgement

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