Model for Computational Analysis of the Concentration of Phosphorus Removed during Leaching of Iron Oxide Ore in Oxalic Acid Solution

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Abstract: Model for computational analysis of the concentration of phosphorus removed (relative to the final pH of leaching solution) during leaching of iron oxide ore in oxalic acid solution has been derived. The model; $P = e^{[1.2\alpha]}$ was found to be dependent on the value of the final solution pH measured during the leaching process. It was observed that the validity of the model is rooted on the expression $\ln P = N\alpha$ where both sides of the relationship are generally approximately equal to 4. The maximum deviation of the model-predicted concentration of dissolved phosphorus from the corresponding experimental values is 22.87% which is quite within the acceptable range of deviation limit of experimental results. Dissolved phosphorus concentration per unit mass of iron oxide ore input evaluated from experimental and model-predicted results are 6.3625 mg/kg/g and 6.7188 mg/kg/g respectively, indicating proximate agreement. [New York Science Journal. 2009;2(6):13-23]. (ISSN: 1554-0200).

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

1. Introduction

Lee et al. [1] reported that the leaching of 3g/L pure haematite (98.2% purity, 105-140μm size range) using 0.048-0.48M oxalic acid at 80-100^{0}C passed through a maximum peak at pH 2.5. Dissolution of haematite was found [1] to be slower than magnetite ($FeO.Fe_2O_3$) and other hydrated iron oxide such as goethite (α-FeOOH), lapidochrosite (γ-FeOOH) and iron hydroxide ($Fe(OH)_3$).

The dissolution of iron oxide is believed to take place via a photo-electro chemical reduction process, involving a complicated mechanism of charge transfer between the predominant oxalate species, namely ferric oxalate $Fe(C_2O_4)_3^{3-}$, ferrous oxalate $Fe(C_2O_4)_2^{2-}$ acting also as an auto catalyst, and the oxalate ligand on the iron oxide surface [2].

The dissolution of iron oxides in oxalic acid was found to be very slow at temperatures within the range 25-60°C, but its rate increases rapidly above 90°C [3]. The dissolution rate also increases with increasing oxalate concentration at the constant pH values set within the optimum range of pH 2.5-3.0. At this optimum pH, the dissolution of fine pure haematite (Fe_2O_3) (105-140μm) follows a diffusion-controlled shrinking core model [3].

Taxiarchour et al [4] reported that it took close to 40h to dissolve 80% of pure haematite slurry (97% purity, 0.022% w/v or 0.21% g/L Fe_2O_3) at pH 1. He stated that even at 90^{0}C, it required close to 10h to achieve 95% dissolution of iron of the slurry at pH 1. They also dissolved iron using 0.1-0.5M oxalic acid (pH1-5) to dissolve iron from a 20% w/v slurry (83% of particle size in the range 0.18-0.35mm, containing 0.029% Fe_2O_3). The iron oxide concentration in the leach is equivalent to 0.058g/L Fe_2O_3.

The speciation of Fe(III) oxalate and Fe(II) oxalate has been found [5] to be governed by pH and total oxalate concentration. For a having pH › 2.5 and an oxalate concentration higher than 0.1M, the most predominant Fe(III) complex ion existing is $Fe(C_2O_4)_3^{3-}$. At these conditions, (pH › 2.5 and an oxalate concentration higher than 0.1M) the predominant Fe(II) complex species is $Fe(C_2O_4)_2^{2-}$.

Nwoye [6] derived a model for quantitative analysis of dissolved iron in oxalic acid solution in relation to the final pH of the solution during leaching of iron oxide ore;

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

Where

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

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

The values of the dissolution constants compared with those of % Fe and % Fe_2O_3 from the experiment [6] indicate clearly that the constants K_1 and K_2 are numerical equivalence of the chemical resistance to the dissolution of Fe and Fe_2O_3 (respectively) in oxalic acid solution. It was found that $K_1 \approx 2K_2$ indicating twice chemical resistance to the dissolution Fe compare to that of Fe_2O_3. This expression agreed with the higher percentage of Fe_2O_3 dissolved compared to that of the corresponding Fe. The model also predicted the final pH of the leaching solution when the concentrations of Fe and Fe_2O_3 dissolved (at a temperature of 30^0C and average ore grain size; 150μm) are known.

Nwoye et al [7] derived a model for predicting the concentration of dissolved iron during leaching of iron oxide ore in sulphuric acid solution. The model is stated as;

$$\%Fe = 0.35(\alpha/T)^3 \tag{2}$$

Where

T = Solution temperature at the time t, when the concentration of dissolved iron is evaluated. (^{0}C)

0.35= (pH coefficient for iron dissolution in sulphuric acid solution during the leaching process) determined in the experiment [7].

α = Final pH of the leaching solution at the time t, when the concentration of dissolved iron is evaluated.

The model (formulated at conditions; leaching temperature of 25^0C, initial solution pH 5.0 and average grain size; 150μm) is dependent of the final pH and temperature of the leaching solution. The model shows that the concentration of iron dissolved during the leaching process is directly proportional to the third power of the ratio of final leaching and temperature.

Final pH of leaching solution has been found to depend on the leaching time, initial pH for the leaching solution and the leaching temperature [8,9].

Models for computational analysis of the concentration of dissolved haematite and heat absorbed by oxalic acid solution during leaching of iron oxide ore have been derived [10]. These models are:

$$\%Fe_2O_3 = K (\gamma/\mu) \tag{3}$$
$$Q = K_C \mu \tag{4}$$

Where

$\%Fe_2O_3$ = Concentration of dissolved haematite in oxalic acid solution.

γ = Final pH of the leaching solution at time t at which $\%Fe_2O_3$ was obtained.

μ = Weight of iron oxide added into the oxalic acid leaching solution (g)

K = Constant of proportionality associated with haematite dissolution

K_C = Constant of proportionality associated with heat absorption

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

Nwoye [10] found that optimization of the weight input of iron oxide ore could be achieved using the model; ($\%Fe_2O_3 = K (\gamma/\mu)$) by comparing the concentrations of dissolved haematite at different weights input of the iron oxide ore, with the view to identifying the optimum weight input of iron oxide ore that gives the maximum dissolution of Fe_2O_3. The model also indicates that the concentration of haematite dissolved during the leaching process is directly proportional to the final pH of the leaching solution and inversely proportional to the weight input of the iron oxide ore.

It was also found [10] that values of Q obtained from both the experiment and model ($Q = K_C \mu$) agree to the fact that leaching of iron oxide ore using oxalic acid solution is an endothermic process, hence the absorbed positive heat energy by the leaching solution. The quantity of heat energy absorbed by the oxalic acid solution during the leaching process (as calculated from the model; $Q = K_C \mu$) was found to be directly proportional to the weight input of the iron oxide ore. These results were obtained at initial pH 6.9, average grain size of 150μm and leaching temperature of 30^0C. The constants of proportionality K and K_C associated with the respective derived models were evaluated to be 0.0683 and 66.88 respectively.

Several works [11-16] have been carried out to remove phosphorus from steel during steel making. All these works carried out, pointed out low treatment temperature and high oxygen activity as the only

essential and unavoidable process conditions which can enhance the rate of dephosphorization. High activity of CaO; a product of decomposition of $CaCO_3$ and a slag forming material is required for enhancement of the dephosphorization process with the phosphorus forming part of the slag. This process involves pyrometallurgy and is capital intensive.

It has been reported [17] that the removal of phosphorus from iron can be achieved only by oxidation during steel making, under a basic slag.

Nwoye [18] derived a model for predicting the time for dissolution of pre-quantified concentration of phosphorus during leaching of iron oxide ore in oxalic acid solution as:

$$\tau = \text{Log}\left(\frac{\left(\dfrac{P^{1/4}}{1.8} \right)}{\text{LogT}} \right) \qquad (5)$$

Where

T= Leaching temperature (^{0}C) in the experiment [19], taken as specified leaching
temperature (^{0}C) aiding the expected dissolution of phosphorus .

N= 1.8 (Dissolution coefficient of phosphorus in oxalic acid solution during leaching of iron
oxide ore) determined in the experiment [19].

P = Concentration of dissolved phosphorus (mg/Kg) in the experiment [19], taken as
pre-quantified concentration of phosphorus expected to dissolve after a leaching time t
(mg/Kg) in the model.

τ = Leaching time (sec.) in the experiment [19], taken as time for dissolution of the pre-
quantified concentration of phosphorus (hrs) in the model.

The model was found to depend on a range of specified leaching temperatures (45-70^{0}C) for its validity. It was found [19] that the time for dissolution of any given concentration of phosphorus decreases with increase in the leaching temperature (up to 70^{0}C), at initial pH 5.5 and average grain size of 150μm.

Nwoye et al. [20] also formulated a model for predicting the concentration of phosphorus removed during leaching of iron oxide ore in oxalic acid solution. The model is expressed as;

$$P = 150.5/\mu\alpha \qquad (6)$$

It was found to predict the removed phosphorus concentration, with utmost dependence on the final pH of the leaching solution and weight input of the iron oxide ore. The model indicates that the concentration of phosphorus removed is inversely proportional to the product of the weight input of the iron oxide ore and the final pH of the leaching solution. Process conditions considered during the formulation of the model [20] include: leaching temperature of 25^{0}C, initial solution pH 5.5 and average ore grain size; 150μm).

Nwoye [21] derived a model for the evaluation of the concentration of dissolved phosphorus (relative to the final pH of the leaching solution) during leaching of iron oxide ore in oxalic acid solution. It was observed that the validity of the model is rooted in the relationship $\ln P = N/\alpha$ where both sides of the expression are approximately equal to 4. The model expressed as;

$$P = e^{(12.25/\alpha)} \qquad (7)$$

depends on the value of the final pH of the leaching solution which varies with leaching time. In all, the positive or negative deviation of the model-predicted phosphorus concentration from its corresponding value obtained from the experiment was found to be less than 22%.

Nwoye [22] also derived a model for predicting the concentration of phosphorus removed during leaching of iron oxide ore in oxalic acid solution. The model is expressed as;

$$P = [(1.8(T)^{\tau})]^4 \qquad (8)$$

was found to be dependent on leaching temperature ranging from 45-70^{0}C and specified leaching time of 0.1381hr (497secs.) recorded during experiment, for its validity. It was found that the validity of the model is rooted in the expression $(P^{1/4})/N = (T)^{\tau}$ where both sides of the expression are correspondingly almost equal. The maximum deviation of the model-predicted values of P from the corresponding experimental values was found to be less than 29% which is quite within the range of acceptable deviation limit of experimental results.

Biological processes for phosphorus removal have also been evaluated based on the use of several types of fungi, some being oxalic acid producing. Anyakwo and Obot [23] recently presented their results of a study on the use of Aspergillus niger and their cultural filtrates for removing phosphorus from Agbaja (Nigeria)

iron oxide ore. The results of this work [23] show that phosphorus removal efficiencies at the end of the 49 days of the leaching process are 81, 63 and 68% for 5, 100 and 250 mesh grain sizes respectively.

An attempt has been made in the past [24] to leach Itakpe iron oxide ore using oxalic acid solution in order to determine the maximum concentration of phosphorus that is removable. Results of chemical analysis of the ore indicate that the percentage of phosphorus in the ore is about 1.18%, which from all indication is quite high and likely to affect adversely the mechanical properties of the steel involved; hence the need for dephosphorization. It was reported [24] that phosphorus can be removed from iron oxide ore through a process associated with hydrometallurgy. Phosphorus was removed at a temperature of 25^0C and initial solution pH 2.5, leading to the dissolution of the phosphorus oxide formed. This involved using acid leaching process to remove phosphorus from the iron oxide ore in readiness for steel making process.

The aim of this work is to derive a model for computational analysis of the concentration of phosphorus removed relative to the final leaching solution pH during leaching of Itakpe (Nigeria) iron oxide ore in oxalic acid solution. This derivation is embarked on in furtherance of the previous work [25].

2. Model

The ore is assumed to be stationary in the reaction vessel during the leaching process and contains the un-leached iron as part of reaction remnants. The ore is attacked by hydrogen ions from oxalic acid within the liquid phase, and in the presence of oxygen.

2.1 Model Formulation

Results from experimental work [25] carried out at SynchroWell Research Laboratory, Enugu were used for the model derivation. These results are as presented in Table 1.

Computational analysis of these experimental results [25] shown in Table 1, resulted to Table 2 which indicate that;

$$lnP = N\alpha \qquad \text{(approximately)} \qquad (9)$$
$$P = e^{N\alpha} \qquad (10)$$

Introducing the value of N into equation (10)

$$P = e^{[1.2\alpha]} \qquad (11)$$

Where

P = Concentration of phosphorus removed during the leaching process (mg/Kg)

N= 1.2; (Dissolution coefficient of phosphorus in oxalic acid solution) determined in the experiment[25].

α = Final pH of the leaching solution at the time t when the concentration of dissolved phosphorus is evaluated.

Equation (11) is the derived model.

Table1: Variation of weight-input of iron oxide ore and final pH of leaching solution with concentration of phosphorus removed.[25]

M (g)	(α)	P (mg/Kg)
4	3.19	59.60
6	3.20	45.60
8	3.29	44.20
10	3.45	51.31
12	3.49	60.80
14	3.84	96.50

M = Mass of iron oxide ore used for the leaching process

Table2: Variation of lnP with Nα

lnP	Nα
4.0877	3.8280
3.8200	3.8400
3.7887	3.9480
3.9379	4.1400
4.1076	4.1880
4.5695	4.6080

3. Boundary and Initial Condition

Iron oxide ore was placed in cylindrical flask 30cm high containing leaching solution of oxalic acid. 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. Range of weight of iron oxide ore used; 2-14g. Initial pH of leaching solution used; 2.5 and leaching time of 3 hrs were used for all samples. A constant leaching temperature of $25^{o}C$ was used. Oxalic acid concentration at 0.1mol/litre and average ore grain size;150μm were also used. Details of the experimental technique are as presented in the report [25].

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 concentration of phosphorus removed from the corresponding experimental 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 [25] were not considered during the model formulation. It is expected that introduction of correction factor to the predicted concentrations of P, gives exactly the experimental values of P.

Deviation (Dv) (%) of model- predicted P values from experimental P values is given by

$$Dv = \left(\frac{P_P - P_E}{P_E} \right) \times 100 \tag{12}$$

Where   Pp = Predicted P values
        P_E = Experimental P values
Since correction factor (Cr) is the negative of the deviation,
$$Cr = - Dv \tag{13}$$
Substituting equation (12) into equation (13) for Dv,

$$Cr = -100 \left(\frac{P_P - P_E}{P_E} \right) \tag{14}$$

It was observed that addition of the corresponding values of Cr from equation (14) to the model-predicted concentrations of P gave exactly the corresponding experimental P values.[25]

5. Results and Discussion

The derived model is equation (11).
Dissolution of phosphorus per unit mass of iron oxide ore added during the leaching process was determined following comparison of the dissolved phosphorus per unit mass of iron oxide ore obtained by calculations involving experimental results, and that obtained directly from the model.
Dissolution of phosphorus per unit mass of iron oxide ore added, D_i (mg/kg/g) was calculated from the equation;

$$D_i = D/\mu \tag{15}$$

Therefore, a plot of concentration of dissolved phosphorus against the mass of iron oxide ore added, as in Fig. 1 using experimental results in Table 1, gives a slope, S at points (6, 45.6) and (14, 96.5) following their substitution into the mathematical expression;

$$S = \Delta D/\Delta\mu \tag{16}$$

Eqn. (16) is detailed as

$$S = D_2 - D_1/ \mu_2 - \mu_1 \tag{17}$$

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 (6, 45.6) and (14, 96.5) for (μ_1, D_1) and (μ_2, D_2,) respectively, and substituting them into eqn. (17), gives the slope as 6.3625mg/kg/g which is the concentration of dissolved phosphorus 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 (6, 46.53) and (14, 100.28) for (μ_1, D_1) and (μ_2, D_2,) respectively and substituting them into eqn. (17) gives the value of slope, S as 6.7188mg/kg/g. This is the model-predicted concentration of dissolved phosphorus per unit mass of iron oxide ore used for the leaching process. A comparison of these two values of dissolved phosphorus 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 phosphorus 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.
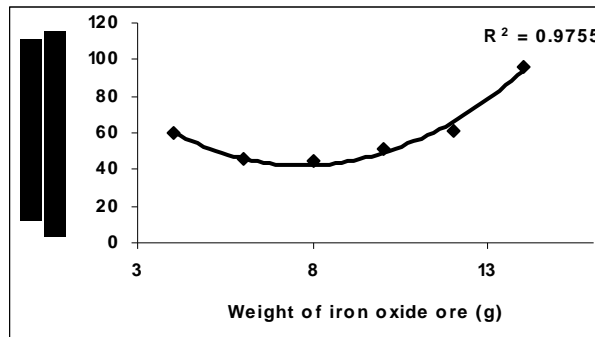
Fig.1-Effect of weight-input of iron oxide ore on the concentration of dissolved phosphorus as obtained from experiment [25]
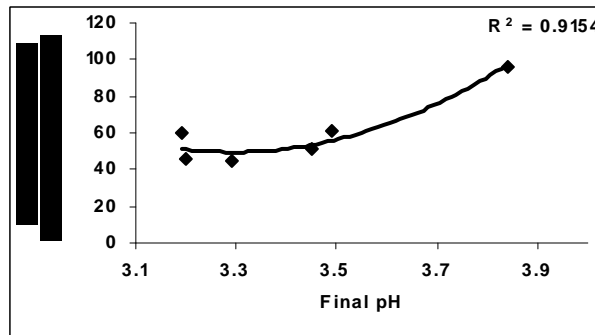

Fig.2-Effect of final pH on the concentration of dissolved phosphorus as obtained from experiment [25]

The values of the correlation coefficient, R calculated from the equation;

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

using the r-squared values (coefficient of determination) from Figs.1-4 show a very close correlation;(0.9877),(0.9568) and (0.8226),(0.9554) between values of the concentration of dissolved phosphorus obtained from experiment[25] and derived model respectively. This also shows that the model-predicted concentrations of dissolved phosphorus are very much in proximate agreement with the corresponding dissolved phosphorus concentration obtained from experiment [25]. 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.
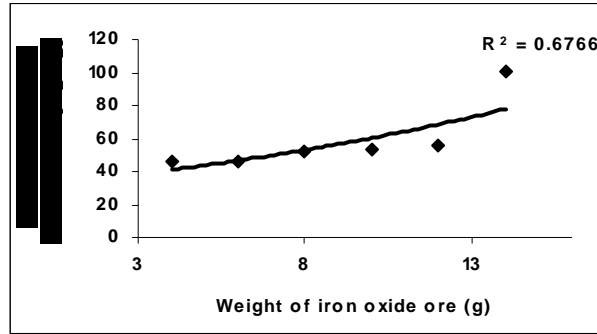
Fig.3-Effect of weight-input of iron oxide ore on the concentration of dissolved phosphorus as predicted by derived model
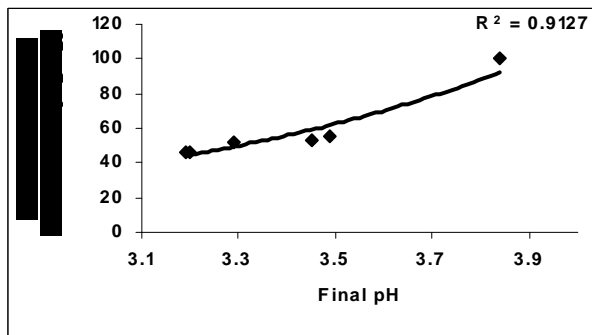

Fig.4-Effect of final pH on the concentration of dissolved phosphorus as predicted by derived model

Comparison of Figs. 5 and 6 show that both values of the dissolved phosphorus concentration obtained from the experiment [25] (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. Moreover, both Fig.5 and 6 show direct relationship in agreement with Table 1 which is made up of data from the experiment [25].
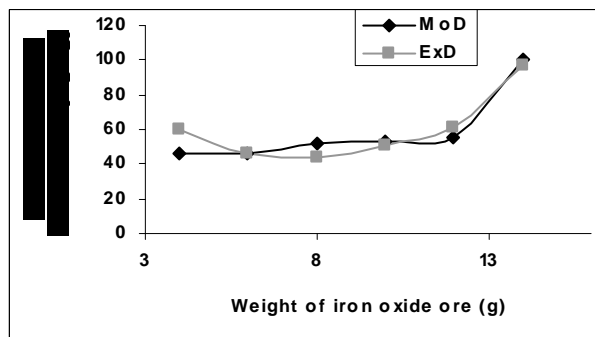
Fig.5-Comparison of the concentrations of dissolved phosphorus in relation to weight of iron oxide ore as obtained from experiment [25] and derived model
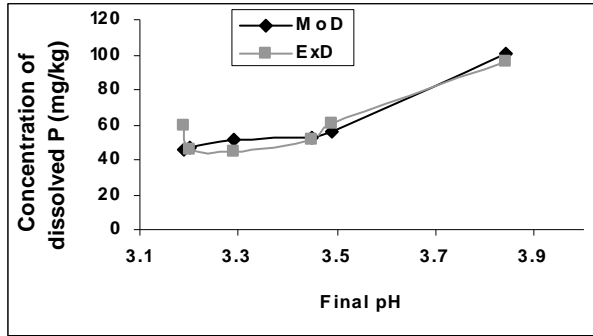
Fig.6-Comparison of the concentrations of dissolved phosphorus in relation to final pH as obtained from experiment [25] and derived model

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

It was found that the validity of the model is rooted in the expression $\ln P = N\alpha$, where both sides of the expression are correspondingly approximately equal 4. Table 2 also agrees with equation (9) following the values of $\ln P = N\alpha$ evaluated from Table 1 as a result of the corresponding computational analysis. The maximum deviation of the model-predicted concentration of dissolved phosphorus from the corresponding experimental value is 22.87% 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 phosphorus) 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 phosphorus (Figs.7- 9).
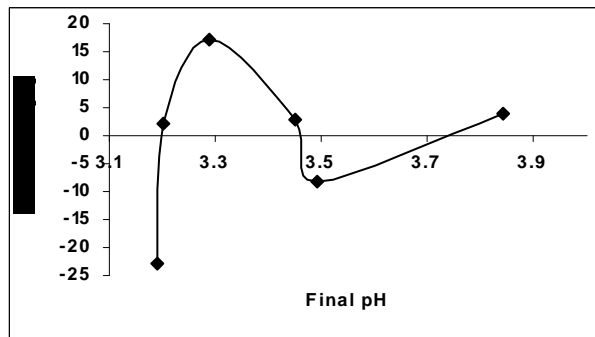

Fig.7-Variation of deviation (from experimental values [25]) of model-predicted concentrations of dissolved phosphorus with final pH
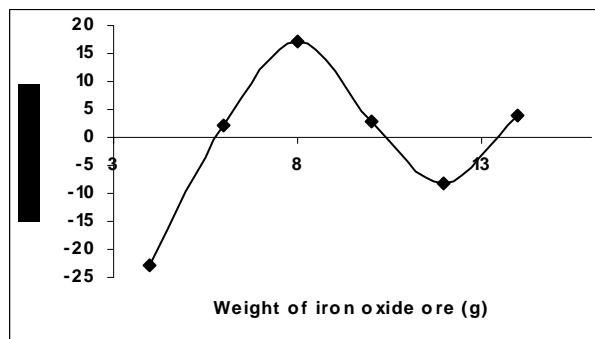

Fig.8-Variation of deviation (from experimental values [25]) of model-predicted concentration of dissolved phosphorus with weight-input of iron oxide ore

Figs.7-9 indicate that the highest and least deviations (-22.87 and 2.04%) 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 phosphorus concentrations 45.97 and 46.53mg/kg respectively. Comparison of Figs.7-8 shows that these percent deviations also correspond to the final solution pH; 3.19 and 3.20 and also iron oxide ore weight-input; 4 and 6g respectively.

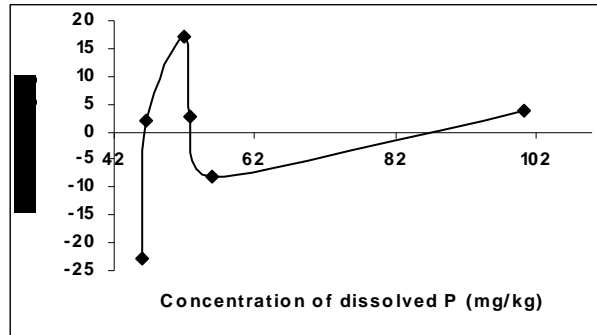
Concentration of dissolved P (mg/kg)

Fig.9-Variation of deviation (from experimental values [25]) of model-predicted concentrations of dissolved phosphorus with the actual model-predicted concentration of dissolved phosphorus

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

Figs. 10 and 11 also show that correction factor to the model-predicted concentration of dissolved phosphorus 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 (22.87 and -2.04%) 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 correspond to the model-predicted dissolved phosphorus concentrations 45.97 and 46.53 mg/kg respectively.
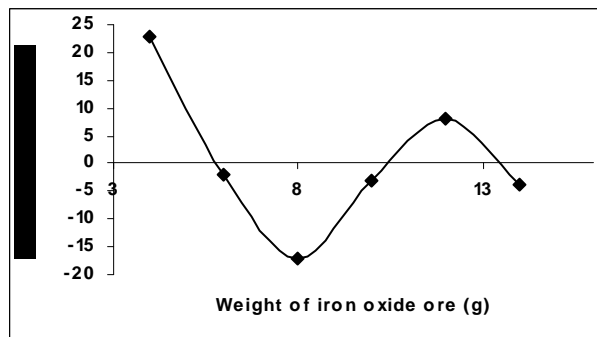

Weight of iron oxide ore (g)

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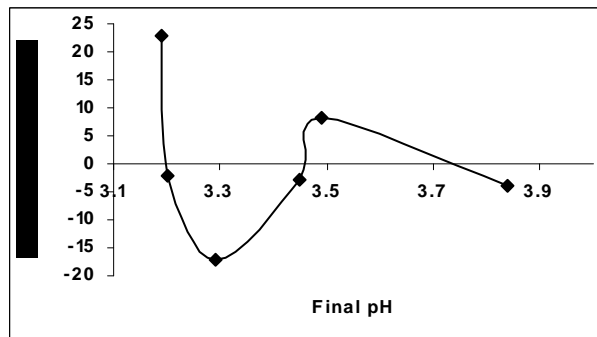

Final pH

Fig.11-Variation of correction factor to model-predicted concentration of dissolved phosphorus with final pH

The percent correction factors also correspond to the final solution pH; 3.19 and 3.20 as well as iron oxide ore weight-input; 4 and 6g 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. (13) and (14). 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 leaching of iron oxide ore in nitric acid solution. The validity of the model is rooted on the expression $\ln P = N\alpha$ where both sides of the expression are correspondingly approximately equal to 4. The maximum deviation of the model-predicted P values from the corresponding experimental P values is 22.87% which is quite within the acceptable range of deviation limit of experimental results. The two values of the dissolved phosphorus concentrations per unit mass of iron oxide ore used; 6.3625mg/kg/g and 6.7188mg/kg/g as obtained from experiment and derived model respectively, 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 P values from those of the experiment.

Acknowledgement

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