

Predictability of Iron Extraction Rate Based on Rate of Phosphorus Removal and As-Beneficiated Phosphorus Content during Leaching in H₂O₂

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ABSTRACT

Iron extraction rate was predicted during leaching of iron ore in hydrogen peroxide solution based on rate of phosphorus removal and the as-beneficiated phosphorus content. A model was derived and used as a tool for the predictive analysis. The validity of the two-factorial model;

$$\alpha = -10x^2 - \gamma^2 + 168.76x + 0.09$$

was found to be rooted on the core model forming expression $\alpha - 0.09 = 10x^2 - \gamma^2 + 168.76x$ where both sides of the expression are correspondingly approximately equal. Statistical analysis of the iron extraction rate as obtained from derived model and experiment for each value of the phosphorus removal rate considered shows standard errors of 0.0004 and 0.0051 respectively. Furthermore, Iron extraction rates per unit rate of phosphorus removal as obtained from derived model-predicted and experimental results were 168.48 and 166.14 %/mins. respectively. Deviation analysis indicates that the maximum deviation of the model-predicted iron extraction rate (from experimental results) is less than 20%, implying an operational confidence level above 80%.

Keywords: Prediction, Iron Extraction Rate, Phosphorus Removal, Hydrogen Peroxide, Iron Ore Leaching.

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INTRODUCTION

Leaching out of metals from ores in various solutions has been generally accepted to be highly environment friendly. Increased intensive research has ensued, exploring optimum methods of achieving high metal yield and clean atmosphere. Alafara et al. (2005) investigated the effect of contact time, acid concentration, temperature, particle size and, the stirring speed on the dissolution of the iron ore during a quantitative leaching of iron ore in hydrochloric acid solution. The dissolution rate was found to be significantly dependent on temperature of the reaction system and the hydrogen ion concentration. The results of the investigation reveal that the mechanism of dissolution follows an exothermic pathway. The activation energy for the dissolution reaction was 13.63 kJmol⁻¹. Furthermore, about 92% of the total iron in the ore was dissolved within 120 mins. by 12M HCl solution and 800⁰C using 0.1mm particle size at an optimum stirring speed of 300rpm. Applicability of oxalic acid for the purpose ensures precipitation of dissolved iron from the leach solution as ferrous oxalate, which can be re-processed to form pure haematite by calcinations (Taxiarchour et al.,1997).

Results generated from extraction processes have been empirically analyzed and appraised using various derived models as analytical tools. A model for calculating the concentrations of dissolved iron (relative to the final solution pH and temperature) during leaching of iron oxide ore in oxalic acid solution was derived (Nwoye and Mbuka, 2011) to evaluate the correlations between dissolved iron & both final solution pH and temperature. The model

$$\%Fe = 1.1849(\gamma/T)^3 \quad (1)$$

was able to calculate the concentrations of dissolved iron being dependent on the values of the final leaching solution pH and temperature measured during the leaching process. It was observed that the validity of the model is rooted in the expression $(\%Fe/N)^{1/3} = \gamma/T$ where both sides of the expression are approximately equal to 0.2. The maximum deviation of the model-predicted concentration of dissolved iron from the corresponding experimental values was found to be less than 18% which is quite within the acceptable range of deviation limit of experimental results. Concentrations of dissolved iron per unit rise in the solution temperature as obtained from experiment and derived model were evaluated as 0.0011 and 0.0015 $\%^{\circ}C$ respectively, indicating proximate agreement.

A model for the evaluation of the concentrations of dissolved iron (relative to the final solution pH and temperature) during leaching of iron oxide ore in sulphuric acid solution has been derived (Nwoye et al., 2008). The model

$$\%Fe = 0.35(\alpha/T)^3 \quad (2)$$

depended on the values of the final pH and temperature of the leaching solution which varied with leaching time. The positive and negative deviations of the model-predicting values of %Fe (dissolved) from those of the experimental values were found to be within the range of acceptable deviation limit for experimental results.

Calculations of the concentrations of leached iron during leaching of iron oxide ore in sulphuric acid solution has been achieved through application of a model (Nwoye et al., 2009a). The model is expressed as

$$\%Fe = e^{-2.0421(\ln T)} \quad (3)$$

The predicted concentrations of leached Fe were observed to be very close to the values obtained from the experiment. The model shows that the concentrations of leached Fe were 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.

A model was successfully derived for predictive analysis of the concentrations of dissolved iron during leaching of iron oxide ore in sulphuric acid solution (Nwoye et al., 2009b). The model expressed as

$$\%Fe = 0.987(\mu/T) \quad (4)$$

was able to predict the concentrations of dissolved Fe with a high degree of precision. It was observed that the model was 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.

The aim of this work is to predict the iron extraction rate during leaching of iron ore in hydrogen peroxide solution based on the rate of phosphorus removal and as-beneficiated phosphorus content. It is generally accepted that phosphorus present in iron ore are closely locked up with the ore and so during leaching of the iron ore, as phosphorus is being oxidized by oxygen (from hydrogen peroxide) and removed with time, iron is simultaneously being extracted. And so the rate of iron extraction is affected by rate of phosphorus removal during the leaching process.

MATERIALS AND METHODS

Agbaja (Nigeria) iron ore was mined and collected from the deposit, beneficiated and the resultant concentrate used for this research work. The iron ore was crushed for the purpose of liberation size. Tyler standard was employed to produce particle size of 250 μm . The raw Agbaja iron ore was then sent for chemical analysis using X-ray fluorescence spectrometer and atomic absorption spectrophotometer.

Scrubbing process

Scrubbing was carried to remove argillaceous materials from the raw iron ore. The iron ore was poured into a head pan and water was poured to a reasonable level. The ore was washed and the water decanted. This was repeated for five times until clear water was observed. At this point 5g of sodium silicate and 25 drops of oleic acid were sprinkled and distributed uniformly throughout the ore. 20liters of distilled water was also introduced into the pan and the content mixed thoroughly. After mixing, the argillaceous materials were removed leaving behind the iron ore. The residue was washed thoroughly and was sun dried for 24 hours. Some quantities were sent for chemical analysis.

Chemical leaching process

The dried scrubbed iron was further pulverized and sieved to obtain particle sizes of 63, 90, 150, 180 and 250 μm . Analar grade of hydrogen peroxide solutions of different moles of 2, 4, 6, 8, and 10 were prepared. 50grams of particles size of 63 μm of scrubbed iron ore was poured into a beaker (reactor). 10ml of 2M of hydrogen peroxide was poured into the beaker containing the iron ore. The mixture was thoroughly mixed to ensure homogeneity. The content was allowed to leach for 20,40,60,80 and 100 minutes at 70°C. At the end of each period, the solution was cooled and filtered. The residue was collected, washed to neutrality with distilled water, air dried and oven dried at 150°C for 24hours. The experiment was repeated for different concentrations and particles sizes. The samples were analyzed using atomic absorption spectrophotometer and X-ray fluorescence diffraction spectrometer.

Model Formulation

Experimental data obtained from the highlighted research work were used for the model derivation. Computational analysis of these data shown in Table 1, gave rise to Table 3 which indicate that;

$$\alpha - K = - S_e x^2 - \gamma^2 + S x \quad (5)$$

Introducing the values of K, S_e and S into equation (5)

$$\alpha - 0.09 = - 10 x^2 - \gamma^2 + 168.76 x \quad (6)$$

$$\alpha = - 10 x^2 - \gamma^2 + 168.76 x + 0.09 \quad (7)$$

Where

(α) = Iron extraction rate (% /mins.)

(γ) = Initial conc. of phosphorus in iron ore (before leaching) (%)

x = Rate of phosphorus removal (% /mins.)

K = 0.09, S_e = 10, S = 168.76

K, S_e , and S are equalizing constant (determined using C-NIKBRAN (Nwoye, 2008))

Table 1: Variation of iron extraction rate with phosphorus removal rate

(γ) (%/ mins.)	(α) (%/ mins.)	(γ) (%)
0.0235	3.9180	0.49
0.0118	1.9830	0.49
0.0079	1.3237	0.49
0.0059	0.9930	0.49
0.0047	0.7946	0.49

Boundary and Initial Condition

Consider iron ore (in a reactor) placed with in hydrogen peroxide solution (oxidant).The reactor atmosphere is not contaminated i.e (free of unwanted gases and dusts). Initially, atmospheric levels of oxygen are assumed just before the decomposition of H_2O_2 (due to air in the reactor). **Mass of iron oxide ore: (50 g)**, range of leaching time considered: 20-100 mins., concentration of H_2O_2 : 4M, constant treatment temperature: 70°C, ore grain size; 90 μ m, were also used.

The boundary conditions are: reactor oxygen atmosphere due to decomposition of H_2O_2 (since the reactor was air-tight closed) at the bottom and top of the ore particles interacting with the gas phase. At the bottom of the particles, a zero gradient for the gas scalar are assumed and also for the gas phase at the top of the particles. The reduced iron is stationary. The sides of the particles are taken to be symmetries.

RESULTS AND DISCUSSIONS

The result of the chemical analysis carried out on the beneficiated iron ore concentrate is presented in Table 1. The table shows that the percentage of total Fe in the as-beneficiated ore is 52.67%.

Table 2: Result of chemical analysis of iron ore used

Element/Compound	Fe _T	P	SiO ₂	Al ₂ O ₃
Unit (%)	552.67	0.49	8.983	6.986

Model Validation

The validity of the model is strongly rooted in equation (6) (core model equation) where both sides of the equation are correspondingly approximately equal. Table 3 also agrees with equation (6) following the values of $\alpha - K$ and $- S_e x^2 - \gamma^2 + S x$ evaluated from the experimental results in Table 1.

Table 3: Variation of $\alpha - K$ with $- S_e x^2 - \gamma^2 + S x$

$\alpha - K$	$- S_e x^2 - \gamma^2 + S x$
3.8280	3.7203
1.8930	1.7499
1.2337	1.0925
0.9030	0.7552
0.7046	0.5529

Furthermore, the derived model was validated by comparing the extracted iron rate predicted by the model and that obtained from the experiment. This was done using various evaluative techniques such as computational, statistical, graphical and deviational analysis.

Computational Analysis

Computational analysis of the experimental and model-predicted iron extraction rate was carried out to ascertain the degree of validity of the derived model. This was done by comparing iron extraction rate per unit rate of phosphorus removal from model-predicted results with those from actual experimental results

Iron extraction rate per unit rate of phosphorus removal α_x was calculated from the equation;

$$\alpha_x = \alpha / x \quad (8)$$

Therefore, a plot of the iron extraction rate against rate of phosphorus removal as in Fig. 1 using experimental results in Table 2, gives a slope, S at points (0.0235, 3.918) and (0.0047, 0.7946) following their substitution into the mathematical expression;

$$\alpha_x = \Delta\alpha / \Delta x \quad (9)$$

Equation (9) is detailed as

$$\alpha_x = \alpha_2 - \alpha_1 / x_2 - x_1 \quad (10)$$

Where

$\Delta\alpha$ = Change in iron extraction rate α_2, α_1 at two values of the phosphorus removal rate x_2, x_1 . Considering the points (0.0235, 3.918) and (0.0047, 0.7946) for (x_1, α_1) and (x_2, α_2) respectively, and substituting them into equation (10), gives the slope as 166.14 which is the iron extraction rate per unit rate of removed phosphorus during the actual leaching process. A plot of iron extraction rate against rate of phosphorus removal (as in Fig. 2) using derived model-predicted results gives a slope: 168.48 on substituting the points (0.0235, 3.8103) and (0.0047, 0.6429) for (x_1, α_1) and (x_2, α_2) respectively into equation (10). This is the model-predicted iron extraction rate per unit rate of removed phosphorus.

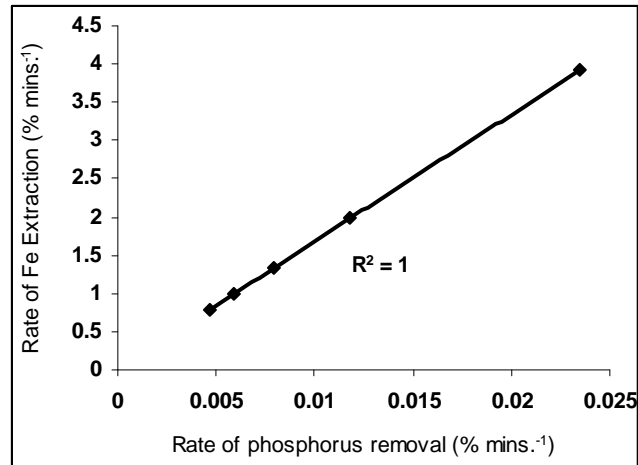


Fig. 1: Coefficient of determination between iron extraction rate iron and rate of phosphorus removal as obtained from experiment

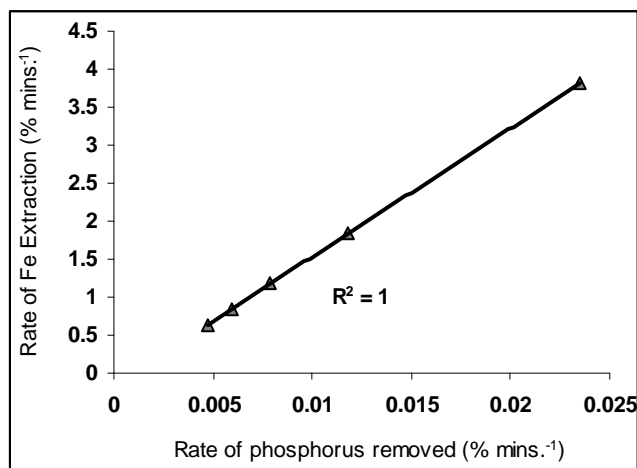


Fig. 2: Coefficient of determination between iron extraction rate iron and rate of phosphorus removal as obtained from derived model

A comparison of this set of values for iron extraction rates (per unit rate of removed phosphorus) also shows proximate agreement and a high degree of validity of the derived model.

Statistical Analysis

Standard Error (STEYX)

The standard errors (STEYX) in predicting the iron extraction rate (using results from derived model and experiment) for each value of the rate of phosphorus removal are 0.0004 and 0.0051% respectively. The standard error was evaluated using Microsoft Excel version 2003.

Correlation

The correlations between iron extraction rate and rate of phosphorus removal as obtained from experiment and derived model considering the coefficient of determination R^2 from Figs. 1 and 2 was calculated using the equation;

$$R = \sqrt{R^2} \quad (11)$$

The evaluations show correlations 1.0000 and 1.0000 respectively. These evaluated results indicate that the derived model predictions are significantly reliable and hence valid considering its proximate agreement with results from actual experiment.

Graphical Analysis

Comparative graphical analysis of Fig. 3 shows very close alignment of the curves from model-predicted iron extraction rate (MoD) and that of the experiment (ExD). The degree of alignment of these curves is indicative of the proximate agreement between both experimental and model-predicted iron extraction rate.

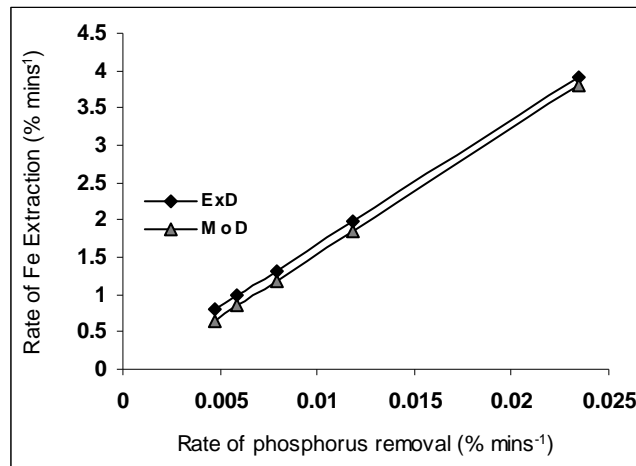


Fig. 3: Comparison of the iron extraction rates (relative to rate of phosphorus removal) as obtained from experiment and derived model

Deviational Analysis

Analysis of iron extraction rate from the experiment and derived model revealed deviations on the part of the model-predicted values relative to values obtained from the experiment. This is attributed to the fact that the surface properties of the iron ore and the physiochemical interactions between the ore and the oxidant (H_2O_2) which were found to have played vital roles during the process were not considered during the model formulation. This necessitated the introduction of correction factor, to bring the model-predicted extracted iron concentration to those of the corresponding experimental values.

Deviation (Dn) of model-predicted iron extraction rate from that of the experiment is given by

$$Dn = \left(\frac{Pv - Ev}{Ev} \right) \times 100 \quad (12)$$

Where

Pv = Iron extraction rate iron as predicted by derived model

Ev = Iron extraction rate iron as obtained from experiment

Correction factor (Cr) is the negative of the deviation i.e

$$Cr = -Dn \quad (13)$$

Therefore

$$Cr = - \left(\frac{P_v - E_v}{E_v} \right) \times 100 \quad (14)$$

Introduction of the corresponding values of Cr from equation (14) into the derived model gives exactly the iron extraction rate as obtained from experiment.

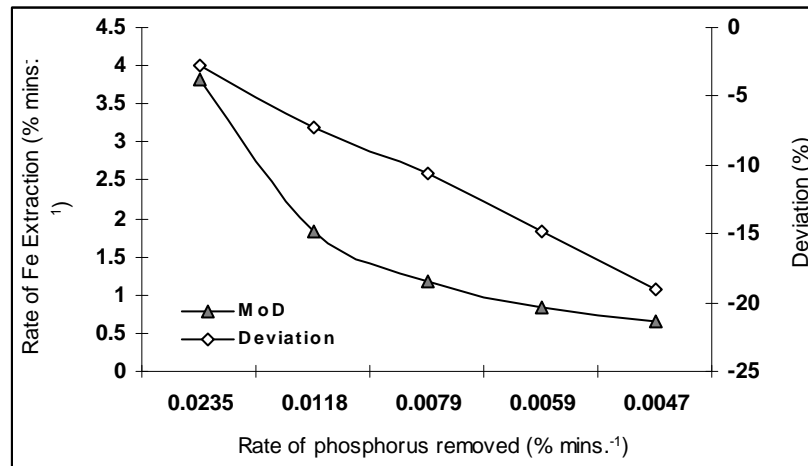


Fig. 4: Variation of model-predicted iron extraction rate with associated deviation from experimental results (relative to rate of phosphorus removal)

Fig. 4 shows that the maximum deviation of the model-predicted iron extraction rate from the corresponding experimental values is less than 20% and quite within the acceptable deviation limit of experimental results. The figure shows that the least and highest magnitudes of deviation of the model-predicted iron extraction rate (from the corresponding experimental values) are - 2.75 and - 19.09 % which corresponds to iron extraction rates: 3.8103 and 0.6429 % /mins, as well as rates of phosphorus removal: 0.0235 and 0.0047 respectively.

Comparative analysis of Fig. 4 and indicates that the orientation of the curve in Fig. 5 is opposite that of the deviation of model-predicted iron extraction rate (Fig. 4). This is because correction factor is the negative of the deviation as shown in equations (13) and (14).

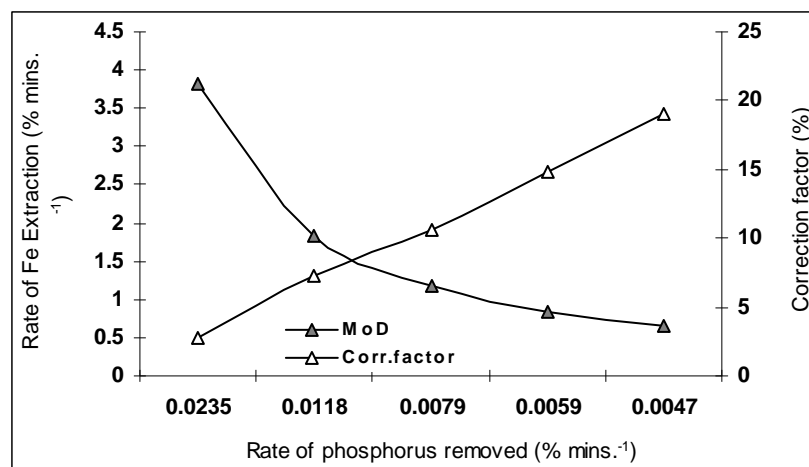


Fig. 5: Variation of model-predicted iron extraction rates with associated correction factor to model-predicted results (relative to rate of phosphorus removal)

It is believed that the correction factor takes care of the effects of surface properties of the iron ore and the physiochemical interactions between the ore and the oxidant (H_2O_2) which have played vital roles during the process, but were not considered during the model formulation. Analysis of Fig 5 indicates that the least and highest magnitudes of correction factor to the model-predicted extracted iron concentrations are + 2.75 and + 19.09 % which corresponds to iron extraction rates: 3.8103 and 0.6429 % /mins, as well as rates of phosphorus removal: 0.0235 and 0.0047 respectively.

It is important to state that the deviation of model predicted results from that of the experiment is just the magnitude of the value. The associated sign preceding the value signifies that the deviation is a deficit (negative sign) or surplus (positive sign).

CONCLUSIONS

Iron extraction rate was predicted during leaching of iron ore in hydrogen peroxide solution based on rate of phosphorus removal and the as-beneficiated phosphorus content. The validity of the two-factorial model used for the predictive analysis was rooted on the core model forming expression $\alpha - 0.09 = 10x^2 - \gamma^2 + 168.76x$ where both sides of the expression are correspondingly approximately equal. Statistical analysis of the iron extraction rate as obtained from derived model and experiment for each value of the phosphorus removal rate considered shows standard errors of 0.0004 and 0.0051 respectively. Furthermore, Iron extraction rates per unit rate of phosphorus removal as obtained from derived model-predicted and experimental results were 168.48 and 166.14 %/mins. respectively. Deviation analysis indicates that the maximum deviation of the model-predicted iron extraction rate (from experimental results) is less than 20%, implying an operational confidence level above 80%.

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