

Empirical Analysis of Extracted Iron Concentration Based on Its As-Beneficiated Content and Mass-Input of Powdered Potassium Chlorate

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Abstract—Empirical analysis of extracted iron concentration was carried out based on its as-beneficiated content and mass-input of powdered potassium chlorate used as oxidant. A model was derived, validated and used as a tool for the analysis. The model is expressed as;

$$\zeta = 6 \times 10^{-5} \vartheta^2 - 0.0018 \vartheta - 0.00001\tau + 0.015$$

The validity of the two-factorial model was found to be rooted on the expression

$\zeta + 0.00001\tau - 0.015 = 6 \times 10^{-5} \vartheta^2 - 0.0018 \vartheta$ where both sides of the expression are correspondingly approximately equal. Statistical analysis of the extracted iron concentration as obtained from experiment and derived model for each value of the mass-input of KClO_3 shows standard errors of 2.1×10^{-4} and 2.3×10^{-4} % respectively. Furthermore, extracted iron concentration per unit mass-input of KClO_3 as obtained from experiment and derived model-predicted results were 1.13×10^{-4} and 1.67×10^{-4} % g^{-1} respectively.

Keywords—Analysis, Extracted Iron, Potassium Chlorate Addition, Iron Ore.

INTRODUCTION

Extraction of metals through leaching of ores in various solutions has been generally accepted to highly environmental friendly. There have been also increased research interests in exploring optimum methods of achieving balance between high yield and clean atmosphere. A comparative assessment evaluation carried out on the solubility of iron in several organic and inorganic acids has shown that iron oxides and oxyhydroxides can dissolve in hydrochloric and perchloric acids [1]. Studies have been conducted on the dissolution of goethite in several inorganic acids belonging to the families of the carboxylic and diphosphoric acids in the presence of reducing agents [2]. Investigations have been carried out 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 [3]. The dissolution rate was found to depend on the hydrogen ion concentration and temperature of the reaction system. The mechanism of dissolution appears to follow an exothermic pathway. The activation energy for the dissolution reaction was 13.63 kJmol^{-1} . About 92% of the total iron in the ore was dissolved within 120 min. by 12M HCl solution and 800°C using 0.1mm particle size at an optimum stirring speed of 300rpm.

Appraisal of results generated from these extraction processes has been carried using various derived models as analytical tools. 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 [4]. The model

$$\% \text{Fe} = 0.35(\alpha/T)^3 \quad (1)$$

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 [5]. The model is expressed as

$$\% \text{Fe} = e^{-2.0421(\ln T)} \quad (2)$$

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 $\ln(\% \text{Fe}) = N(\ln T)$ where both sides of the expression are correspondingly approximately equal.

A model for calculating the concentrations of dissolved iron during leaching of iron oxide ore in nitric acid solution was also derived [6]. It was observed that the validity of the model is rooted on the expression $\%Fe = N(\mu/\alpha)$ 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%. The model

$$\%Fe = \left(\frac{0.0043}{\alpha} \mu \right) \quad (3)$$

was found to be dependent on the value of the mass-input of iron oxide ore and final solution pH measured during the leaching process. Dissolved iron concentration per unit mass of iron oxide ore input evaluated from experimental and model-predicted results were 0.0010%/g and 0.0011%/g respectively, indicating proximate agreement.

A model was successfully derived for predictive analysis of the concentrations of dissolved iron during leaching of iron oxide ore in sulphuric acid solution [7]. 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.

A model for predicting the concentration of iron dissolved during nitric acid leaching of iron oxide ore in oxalic acid solution has been derived [8] to assess how the final solution pH affects the extraction of iron. The model

$$\%Fe = \left(\frac{0.0133}{\mu} \right) \alpha \quad (5)$$

was found to depend on the value of the final solution pH and mass-input of iron oxide ore during the experiment. It was observed that the validity of the model is rooted in the expression $\%Fe = N(\alpha/\mu)$, where both sides of the relationship are correspondingly approximately almost equal. Dissolved iron concentrations per unit mass of iron oxide ore input evaluated from experimental and model-predicted results were 0.0058%/g and 0.006%/g respectively, indicating proximate agreement.

Evaluation of the prospect and effectiveness of dissolving iron (from iron compounds) in organic acids such as acetic, oxalic formic, citric and ascorbic acids

has shown that oxalic acid is most effective and promising because of its acid strength, good complexing characteristics and high reducing power, compared to other organic acids [9].

Applicability of oxalic acid ensures precipitation of dissolved iron from the leach solution as ferrous oxalate, which can be re-processed to form pure haematite by calcinations [10].

Also 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 [11] to evaluate the correlations between dissolved iron & both final solution pH and temperature. The model

$$\%Fe = 1.1849(\gamma/T)^3 \quad (6)$$

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 %/°C respectively, indicating proximate agreement.

The aim of this work is to take an empirical analysis of extracted iron concentration was carried out based on its as-beneficiated content and mass-input of powdered potassium chlorate used as oxidant. A model will be derived, validated and used for the predictive analysis.

MATERIALS AND METHODS

Agbaja (Nigeria) iron ore concentrate used for this work was obtained from Nigeria Metallurgical Development Centre (NMDC) Jos. This concentrate was dried in air (under atmospheric condition) and used in the as-received condition with particle size; 150 μ m. A weighed quantity of the dried iron ore concentrate was mixed with 11g of powdered KClO₃ (obtained from Fisher Scientific Company Fair Lawn, New Jersey, USA). These mixtures were provided on five different iron crucibles. The mixtures were heated to a temperature of 400°C in a Gallenkamp Hot pot electric furnace at NMDC Laboratory for 600 secs. and thereafter were emptied on white steel pans for observation. The experiment was repeated using varying mass-inputs of KClO₃ i.e 12, 13, 15, 16g and constant mass-inputs of iron ore. Weighed quantities of the sample mixtures for each experiment set were taken (after heating) for chemical analysis (to determine the extracted Fe concentration) using wet analysis method. The average of the extracted Fe

concentration determined in each experiment set was taken as the precise result [12].

Model Formulation

Experimental data [12] 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;

$$\zeta + K\tau - S = N\vartheta^2 - S_e \vartheta \quad (7)$$

Introducing the values of K, S, N and S_e into equation (7)

$$\zeta + 0.00001\tau - 0.015 = 6 \times 10^{-5} \vartheta^2 - 0.0018 \vartheta \quad (8)$$

$$\zeta = 6 \times 10^{-5} \vartheta^2 - 0.0018 \vartheta - 0.00001\tau + 0.015 \quad (9)$$

Where (ζ) = Conc. of extracted iron (%)

(τ) = As- beneficiated Fe content of the iron ore (%)

(ϑ) = Mass-input of $KClO_3$ (g)

$K = 0.00001$, $S = 0.015$, $N = 6 \times 10^{-5}$, $S_e = 0.0018$; K, S, N and S_e are equalizing constant (determined using C-NIKBRAN [13])

Table 1: Variation of iron extracted concentration with mass-input of $KClO_3$ [12]

(τ)	(ϑ)	(ζ)
0.00183	11	45.9
0.00166	12	45.9
0.00138	13	45.9
0.00132	15	45.9
0.00164	16	45.9

Boundary and Initial Condition

Consider iron ore (in a furnace) mixed with potassium chlorate (oxidant). The furnace atmosphere is not contaminated i.e (free of unwanted gases and dusts). Initially, atmospheric levels of oxygen are assumed just before the decomposition of $KClO_3$ (due to air in the furnace). Mass of iron oxide ore: (50g), treatment time: 600 secs., treatment temperature range: 400°C, ore grain size; 150 μ m and range of $KClO_3$ mass-input: 11-16g.

The boundary conditions are: furnace oxygen atmosphere due to decomposition of $KClO_3$ (since the furnace was air-tight closed) at the top and bottom 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 2. The table shows that the percentage of total Fe in the as-beneficiated ore is 45.9%.

Table 2: Result of chemical analysis of iron ore used [12]

Element/Compound	Fe τ	P	SiO $_2$	Al $_2$ O $_3$
Unit (%)	78.6	0.9	5.3	11.3

Model Validation

The validity of the model is strongly rooted in equation (8) (core model equation) where both sides of the equation are correspondingly approximately equal. Table 3 also agrees with equation (8) following the values of $\zeta + 0.00001\tau - 0.015$ and $6 \times 10^{-5} \vartheta^2 - 0.0018 \vartheta$ evaluated from the experimental results in Table 1.

Table 3: Variation of $\zeta + 0.00001\tau - 0.015$ with $6 \times 10^{-5} \vartheta^2 - 0.0018 \vartheta$

$\zeta + 0.00001\tau - 0.015$	$6 \times 10^{-5} \vartheta^2 - 0.0018 \vartheta$
-0.0127	-0.0125
-0.0128	-0.0130
-0.0131	-0.0133
-0.0132	-0.0153
-0.0129	-0.0134

Furthermore, the derived model was validated by comparing the extracted iron concentration 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 extracted iron concentration was carried out to ascertain the degree of validity of the derived model. This was done by comparing extracted iron per unit leaching time as well as extracted iron per unit mass-input of $KClO_3$ evaluated from model-predicted results with those from actual experimental results

Extracted iron concentration per unit mass-input of $KClO_3$ ζ_m (%/ g) was calculated from the equation;

$$\zeta_m = \zeta / m \quad (10)$$

Therefore, a plot of the extracted iron concentration against mass-input of $KClO_3$ as in Fig. 1 using experimental results in Table 1, gives a slope, S at points (12, 0.00166) and (15, 0.00132) following their substitution into the mathematical expression;

$$\zeta_m = \Delta\zeta / \Delta m \quad (11)$$

Equation (11) is detailed as

$$\zeta_m = \zeta_2 - \zeta_1 / m_2 - m_1 \quad (12)$$

Where

$\Delta\zeta$ = Change in extracted iron concentrations of ζ_2 , ζ_1 at two mass-input values m_2 , m_1 . Considering the points (12, 0.00166) and (15, 0.00132) for (m_1 , ζ_1) and (m_2 , ζ_2) respectively, and substituting them into equation (12),

gives the slope as $-1.13 \times 10^{-4} \% g^{-1}$ which is the extracted iron concentration per unit mass-input of $KClO_3$ during the actual extraction process [12].

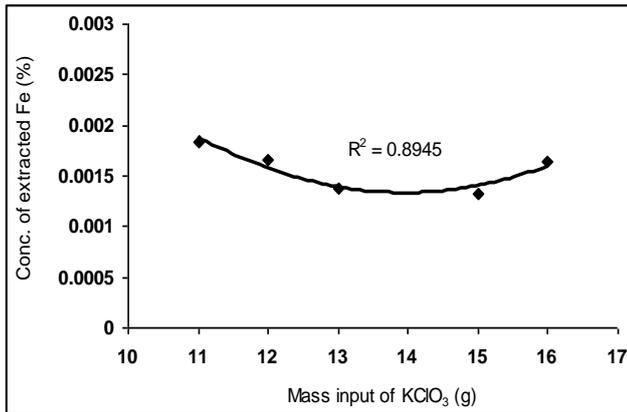


Fig. 1: Coefficient of determination between extracted iron concentration and mass-input of $KClO_3$ as obtained from experiment [12]

A plot of the concentration of extracted iron against mass-input of $KClO_3$ (as in Fig. 2) using derived model-predicted results gives a slope: $-1.67 \times 10^{-4} \% g^{-1}$ on substituting the points (12, 0.0015) and (15, 0.001) for (m_1, ζ_1) and (m_2, ζ_2) respectively into equation (12). This is the model-predicted extracted iron concentration per unit mass-input of $KClO_3$.

A comparison of this set of values for extracted Fe concentration (per unit mass-input of $KClO_3$) also shows proximate agreement and a high degree of validity of the derived model.

It is very pertinent to state that the actual extracted Fe concentration per unit mass-input of $KClO_3$ (as obtained from experiment and derived model) was just the magnitude of the signed value. The associated sign preceding these values signifies that the associated slope tilted to negative plane. Based on the foregoing, extracted Fe concentration per unit mass-input of $KClO_3$ as obtained from experiment and derived model were $1.13 \times 10^{-4} \% g^{-1}$ and $1.67 \times 10^{-4} \% g^{-1}$ respectively.

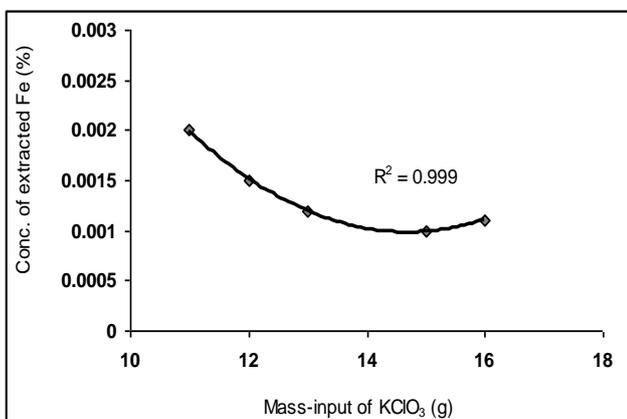


Fig. 2: Coefficient of determination between extracted iron concentration and mass-input of $KClO_3$ as obtained from derived model

Statistical Analysis

The standard errors (STEYX) in predicting the extracted iron concentration (using results from experiment [12] and derived model) for each value of the mass-input of $KClO_3$ are 2.1×10^{-4} and 2.3×10^{-4} respectively. The standard error was evaluated using Microsoft Excel version 2003.

Also the correlations between extracted iron concentration and mass-input of $KClO_3$ as obtained from experiment and derived model considering the coefficient of determination R^2 from Figs. 1 and 2 was calculated using the equation;

$$R = \sqrt{R^2} \quad (13)$$

The evaluations show correlations 0.9458 and 0.9995 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 [12].

Graphical Analysis

Comparative graphical analysis of Fig. 3 shows very close alignment of the curves from model-predicted extracted iron concentration (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 extracted iron concentration.

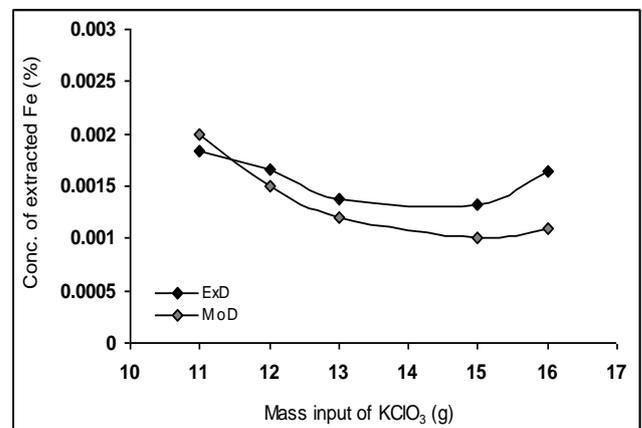


Fig. 3: Comparison of the extracted iron concentrations (relative to mass-input of $KClO_3$) as obtained from experiment [12] and derived model

Deviational Analysis

Analysis of extracted Fe concentrations 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 ($KClO_3$) 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 Fe concentration to those of the corresponding experimental values.

Deviation (D_n) of model-predicted extracted Fe concentration from that of the experiment is given by

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

Where

P_v = Extracted iron concentration as predicted by derived model

E_v = Extracted iron concentration as obtained from experiment [12]

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

$$C = -D_n \quad (15)$$

Therefore

$$C_r = - \left(\frac{P_v - E_v}{E_v} \right) \times 100 \quad (16)$$

Introduction of the corresponding values of C_r from equation (16) into the derived model gives exactly the extracted iron concentration as obtained from experiment.

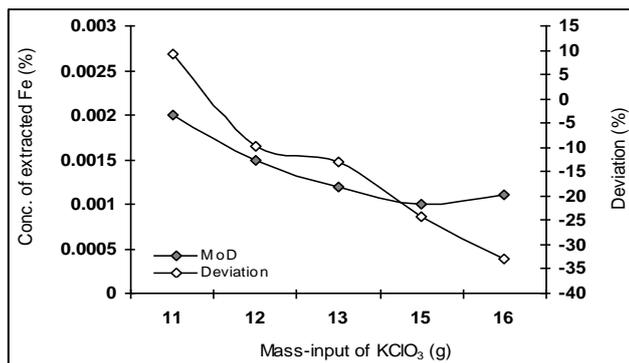


Fig. 4: Variation of model-predicted extracted iron concentration with associated deviation from experimental results (relative to mass-input of $KClO_3$)

Fig. 4 show that the maximum deviation of the model-predicted extracted iron concentration from the corresponding experimental values is less than 33%. The figure show that the least and highest magnitudes of deviation of the model-predicted extracted iron concentration (from the corresponding experimental values) are + 9.29 and - 32.93 % which corresponds to extracted iron concentrations: 0.002 and 0.0011 %, as well as mass-input of $KClO_3$: 11 and 16 g respectively.

Comparative analysis of Figs. 4 and 5 indicate that the orientation of the curve in Fig. 5 is opposite that of the deviation of model-predicted extracted iron concentration (Fig. 4). This is because correction factor is the negative of the deviation as shown in equations (15) and (16).

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 ($KClO_3$) which were found to have played vital roles during the process were not considered during the model formulation. Figs 8 indicate that the least and highest magnitudes of correction factor to the model-predicted extracted iron concentrations are - 9.29 and + 32.93 % which corresponds to extracted iron concentrations: 0.002 and 0.0011 %, as well as mass-input of $KClO_3$: 11 and 16 g respectively.

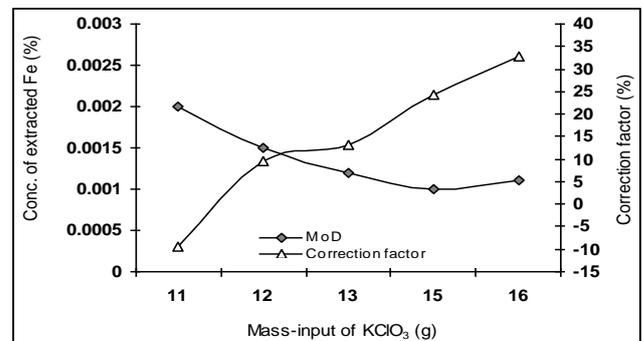


Fig. 5: Variation of model-predicted extracted iron concentration with associated correction factor from experimental results (relative to mass-input of $KClO_3$)

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

Empirical analysis of extracted iron concentration was carried out based on its as-beneficiated content and mass-input of powdered potassium chlorate used as oxidant. A model was derived, validated and used as a tool for the analysis. The validity of the two-factorial model was found to be rooted on the expression $\zeta + 0.00001\tau - 0.015 = 6 \times 10^{-5}\vartheta^2 - 0.0018\vartheta$ where both sides of the expression are correspondingly approximately equal. Statistical analysis of the extracted iron concentration as obtained from experiment and derived model for each value of the mass-input of $KClO_3$ shows standard errors of 2.1×10^{-4} and 2.3×10^{-4} % respectively. Furthermore, extracted iron concentration per unit mass-input of $KClO_3$ as obtained from experiment and derived model-predicted results were 1.13×10^{-4} and 1.67×10^{-4} % g^{-1} respectively.

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