Assessment Evaluation of Removed Phosphorus Concentration Based on Its As-Beneficiated Content and Treatment Temperature

E. M. Ameh[1], S. I. Neife[2], I. D. Adiele[3], C. A. Mgbachi[4], A. O. Agbo[1], S. E. Ede[1] and C.N. Eze[5]

[1]Department of Metallurgical and Materials Engineering, Enugu State University of Science & Technology, Enugu, Nigeria
[2] Department of Metallurgical and Materials Engineering, University of Nigeria, Nsukka, Nigeria
[3] Projects Development Institute (PRODA), Enugu, Nigeria
[4]Department of Computer Science Engineering, Enugu State University of Science & Technology, Enugu, Nigeria
[5]Department of Microbiology, University of Nigeria, Nsukka, Nigeria

ABSTRACT

An assessment evaluation of removed phosphorus concentration was carried out based on its as-beneficiated concentration and treatment temperature. Powdered potassium chlorate ($KClO_3$) was mixed with iron ore concentrate and the mixture heated for 600 secs.

$$\zeta = 0.0969 \ln \vartheta - 0.531 + 0.01\gamma$$

was found to be rooted on the expression $\zeta - 0.01\gamma = 0.0969 \ln \vartheta - 0.531$ where both sides of the expression are correspondingly approximately equal. Statistical analysis of the experimental and derived model-predicted removed phosphorus concentrations for each value of the treatment temperature considered shows standard errors of 0.0093 and 0.0011% respectively. Furthermore, removed phosphorus concentration per unit rise in the treatment

in a laboratory furnace at within temperature range: 400-600^{0}C. A model was derived from the generated experimental results, validated and used as a tool for the analysis. The validity of the two-factorial model expressed as;

temperature as obtained from experiment and derived model-predicted results were 2.31×10^{-4} and 2.05×10^{-4} % / ^{0}C respectively. Deviational analysis indicates that the derived model gives best-fit process analysis at deviations 4.63, 4.68 and 5.8% from experimental results and invariably, operational confidence level range: 94.2 - 95.37%. The deviations corresponded to treatment temperature range: 450-550^{0}C.

Keywords: Assessment, Removed phosphorus concentration, $KClO_3$, Agbaja Iron Ore.

INTRODUCTION

Embrittlement resulting from presence of phosphorus in produced steel materials, above the admissible quantity has caused series of structural failures in the oil industry. This awakens the need for intensive researches geared towards reducing the phosphorus content of pig iron below the admissible level.

Some researches [1-5] have evaluated ways of achieving successful dephosphorization of iron oxide ore and also predicting removed phosphorus concentrations using empirical models. A model was derived for predictive analysis of the concentration of phosphorus removed during leaching of iron oxide ore in sulphuric acid solution [6]. The work indicated that phosphorus removal from the iron oxide ore as obtained from experiment and derived model is dependent on the initial and final pH of the

leaching solution. This is because the final pH of the leaching solution is greatly determined by the initial pH which is function of hydrogen ion concentration.

A similar work [7] shows that phosphorus removal is dependent on leaching temperature. The model derived using experimental results indicated that at a leaching temperature range 45-70^{0}C, the maximum deviation of the model-predicted removed phosphorus concentration (from the corresponding experimental values) was less than 29%.

Empirical analysis of phosphorus removal was carried out [8] based on its as-beneficiated concentration and mass-input of $KClO_3$. A model was derived and used as a tool for the analysis. The model is expressed as: P_R = -

$0.0005 \; \alpha_2 \; + \; 0.01\alpha \; + \; 0.0024 \; \gamma_2 \; + \; 0.0181$ (1)

The validity of the two-factorial model was found to be rooted on the expression $100 \, P_R + 1.81 = -0.05 \, \alpha_2 + \alpha + 0.24 \, \gamma_2$ where both sides of the expression are correspondingly approximately equal. Statistical analysis of the derived model-predicted, regression model-predicted and experimental removed phosphorus concentrations for each value of the mass-inputs of $KClO_3$ considered shows standard errors of 0.0015, 3.45×10^{-5} and 0.0025% respectively. Furthermore, removed phosphorus concentration per unit mass-input of $KClO_3$ as obtained from derived model-predicted, regression model-predicted and experimental results were 3.0×10^{-3}, 2.975×10^{-3} and 2.475×10^{-3} % g^{-1} respectively. Deviational analysis indicates that the derived model gives best-fit process analysis

with a deviation range of just 1.35 – 12.29%, from experimental results and invariably an operational confidence level range 87.71-98.65%. The deviation range corresponded to the removed phosphorus concentration range: 0.0213-0.0333 % and $KClO_3$ mass-input range: 11-15g. This implies that the derived model can exclusively be significantly useful and viably operational within these process conditions.

The aim of this work is to assess the removed phosphorus concentration based on its as-beneficiated concentration and treatment temperature. The essence of this work is to ascertain the level of dephosphorization achievable at different treatment temperatures where all other input process parameters are kept constant.

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; 150µm. Weighed quantities of the dried iron ore concentrate and powdered $KClO_3$ (obtained from Fisher Scientific Company Fair Lawn, New Jerry, USA) were mixed on five different iron crucibles. The mixtures were poured into the crucibles and then heated to a temperature of 400^0C 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 varied treatment temperatures i.e 450, 500, 550, 600^0C and constant mass-inputs of iron ore and $KClO_3$. Weighed quantities of the sample mixtures for each experiment set were taken (after heating) for chemical analysis (to determine percentage phosphorus removal) using wet analysis method. The average of the removed phosphorus concentration determined in each experiment set was taken as the precise result.

Model Formulation

Experimental data [9] 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;

$\zeta - K\gamma \approx S \ln \vartheta - N$ (2)

Introducing the values of K, S and N into equation (2)

$\zeta - 0.01\gamma = 0.0969 \ln \vartheta - 0.531$ (3)

$\zeta = 0.0969 \ln \vartheta - 0.531 + 0.01\gamma$ (4)

Where

(ζ) = Conc. of removed phosphorus (%)

(γ) = As- beneficiated phosphorus content of the ore (%)

(ϑ) = Treatment temperature (^0C)

K = 0.01, S = 0.0969 and N = 0.531; Equalizing constant

(determined using C-NIKBRAN [10])

Table 1: variation of removed phosphorus concentration with treatment temperature [9]

($ɤ$)	($ϑ$)	($ζ$)
0.9	400	0.0507
0.9	450	0.0734
0.9	500	0.0758
0.9	550	0.0854
0.9	600	0.0769

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: 400-600°C, ore grain size; 150μm and mass of $KClO_3$ used: 15g.

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 1. The table shows that the percentage of phosphorus present in the as-beneficiated ore is 0.9%.

Table 2: Result of chemical analysis of iron ore used [9]

Element/Compound	Fe	P	SiO_2	Al_2O_3
Unit (%)	78.6	0.90	5.30	11.0

Model Validation

The validity of the model is strongly rooted in equation (3) (core model equation) where both sides of the equation are correspondingly approximately equal. Table 3 also agrees with equation (3) following the values of $ζ$ - 0.01$ɤ$ and 0.0969 ln $ϑ$ - 0.531 evaluated from the experimental results in Table 1.

Table 3: Variation of $ζ$ - 0.01$ɤ$ with 0.0969 ln $ϑ$ - 0.531

$ζ$ - 0.01$ɤ$	0.0969 ln $ϑ$ - 0.531
0.0417	0.0496
0.0644	0.0610
0.0668	0.0712
0.0764	0.0804
0.0679	0.0889

Furthermore, the derived model was validated by comparing the removed phosphorus 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 removed phosphorus concentration was carried out to ascertain the degree of validity of the derived model. This was done by comparing phosphorus removal per unit mass-input of $KClO_3$ evaluated from

model-predicted results with those from actual experimental results

$$\zeta^T = \zeta / T \qquad (5)$$

Therefore, a plot of the concentration of phosphorus removed against treatment temperature as in Fig. 1 using experimental results in Table 2, gives a slope, S at points (400, 0.0507) and (550, 0.0854) following their substitution into the mathematical expression;

$$\zeta^T = \Delta\zeta / \Delta T \qquad (6)$$

Equation (6) is detailed a

$$\zeta^T = \zeta_2 - \zeta_1 / T_2 - T_1 \qquad (7)$$

Removed phosphorus concentration per unit rise in treatment temperature P_R^M (%/ g) was calculated from the equation;

Where

$\Delta\zeta$ = Change in removed phosphorus concentrations of ζ_2, ζ_1 at two treatment temperatures T_2, T_1. Considering the points (400, 0.0507) and (550, 0.0854) for (T_1, ζ_1) and (T_2, ζ_2) respectively, and substituting them into equation (7), gives the slope as 2.31×10^{-4} %/ ^{0}C which is the removed phosphorus concentration per unit rise in treatment temperature during the actual dephosphorization process [9].
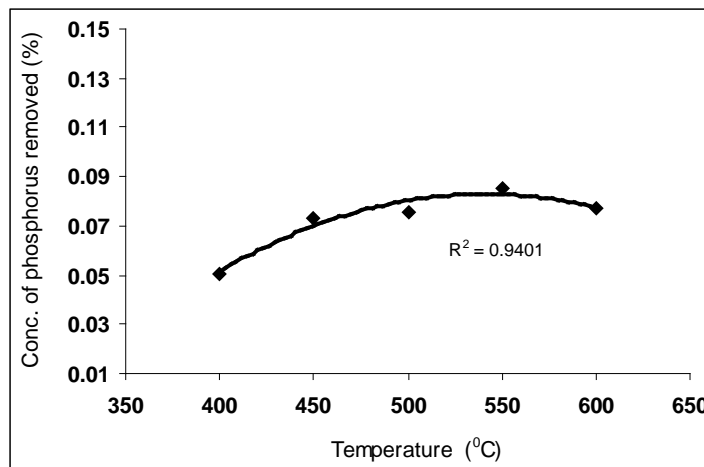

Fig. 1: Coefficient of determination between concentrations of removed phosphorus and treatment temperature as obtained from the experiment [9]

Similarly, a plot of the concentration of removed phosphorus against mass-input of treatment temperature (as in Fig. 2) using derived model-predicted results gives a slope: 2.05×10^{-4} %/ ^{0}C on substituting the points (400, 0.0586) and (550, 0.0984) for (T_1, ζ_1) and (T_2, ζ_2) respectively into equation (7). This is the model-predicted removed

phosphorus concentration per unit rise in treatment temperature.

A comparison of this set of values for removed phosphorus concentration (per unit rise in treatment temperature) also shows proximate agreement and a high degree of validity of the derived model.
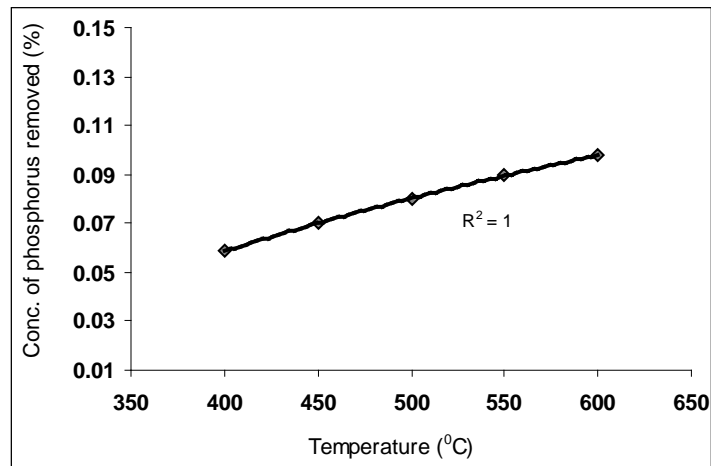
Fig. 2: Coefficient of determination between concentration of removed phosphorus and treatment temperature as obtained from derived model

Statistical Analysis

The standard errors (STEYX) in predicting the removed phosphorus concentration (using results from experiment [9] and derived model) for each value of the treatment temperature were 0.0093 and 0.0011 % respectively. The standard error was evaluated using Microsoft Excel version 2003.

Also the correlations between removed phosphorus concentration and treatment temperature as obtained from experiment and derived model was calculated considering the coefficient of determination R^2 from Figs. 1-3. The calculation was done using the equation

$$R = \sqrt{R^2} \qquad (8)$$

The calculated correlations were 0.9696 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 removed phosphorus 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 phosphorus removed concentration.
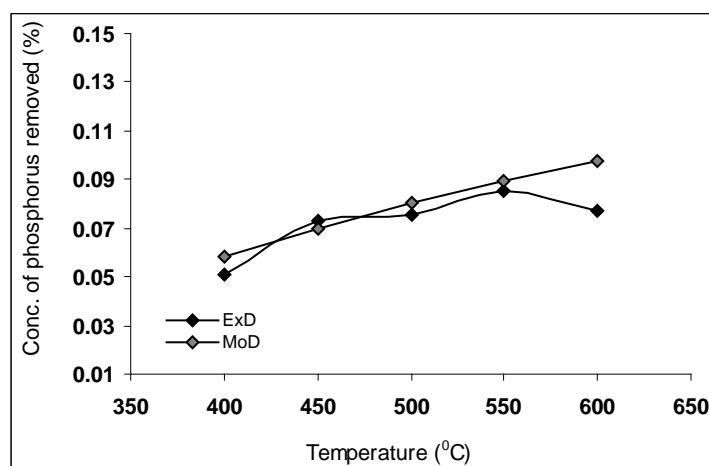

Fig. 3: Comparison of the concentrations of removed phosphorus (relative to treatment temperature) as obtained from experiment [9] and derived model

Deviational Analysis

Analysis of removed phosphorus 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

$$Dn = \left(\frac{Pv - Ev}{Ev}\right) \times 100 \qquad (9)$$

Where

Pv = Removed phosphorus concentration as predicted by derived model

Ev = Removed phosphorus concentration as obtained from experiment [9]

$$Cr = -Dn \qquad (10)$$

Therefore

$$Cr = -\left(\frac{Pv - Ev}{Ev}\right) \times 100 \qquad (11)$$

Introduction of the corresponding values of Cr from equation (11) into the derived model gives

vital roles during the process were not considered during the model formulation. This necessitated the introduction of correction factor, to bring the model-predicted removed phosphorus concentration to those of the corresponding experimental values.

Deviation (Dn) of model-predicted removed phosphorus concentration from that of the experiment is given by

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

exactly the removed phosphorus concentration as obtained from experiment.
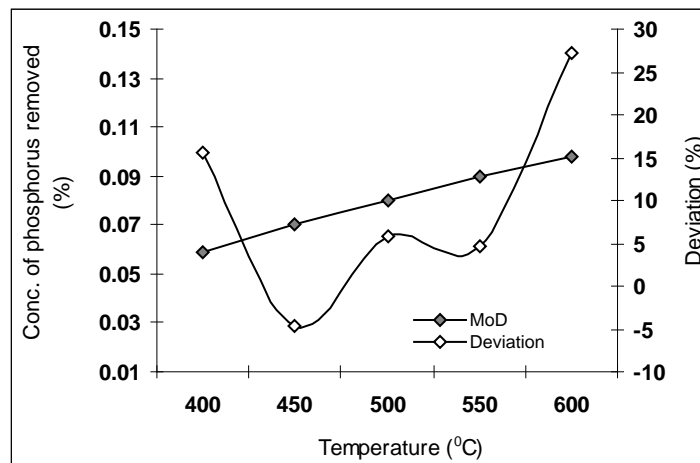

Fig. 4: Variation of model-predicted removed phosphorus concentration with associated deviation from experimental results [9] (relative to treatment temperature)

Fig. 4 shows that the maximum deviation of the model-predicted removed phosphorus concentration from the corresponding experimental values is less than 28%. The figure shows that the least and highest magnitudes of deviation of the model-predicted removed phosphorus concentration (from the corresponding experimental values) are - 4.63 and + 27.31 % which corresponds to removed

phosphorus concentrations: 0.07 and 0.0979 %, as well as treatment temperature: 450 and 600^0C respectively. Deviational analysis also indicates that the derived model gives best-fit process analysis at deviations 4.63, 4.68 and 5.8% from experimental results and invariably, operational confidence level range: 94.2 - 95.37%. These deviations corresponded to treatment temperature range: 450-550^{0}C.
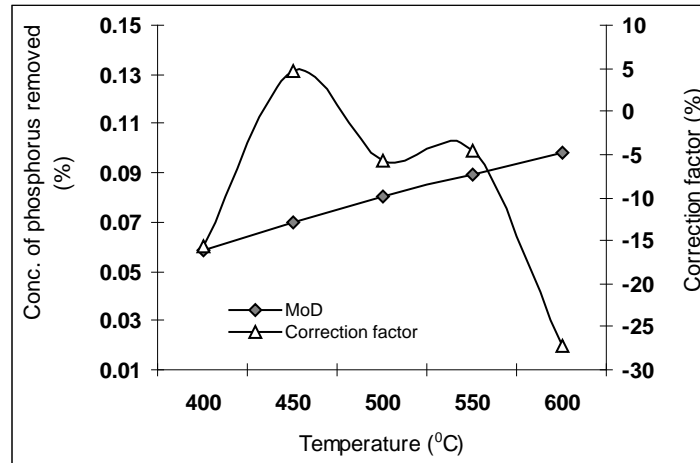
Fig. 5: Variation of model-predicted removed phosphorus concentration with associated correction factor (relative to treatment temperature)

Comparative analysis of Figs. 4 and 5 indicates that the orientation of the curve in Fig. 5 is opposite that of the deviation of model-predicted removed phosphorus concentration (Fig. 4). This is because correction factor is the negative of the deviation as shown in equations (10) and (11).

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 have played vital roles during the

process, but were not considered during the model formulation. Fig. 5 indicates that the least and highest magnitudes of correction factor to the model-predicted removed phosphorus concentration are + 4.63 and - 27.31 % which corresponds to removed phosphorus concentrations: 0.07 and 0.0979 %, as well as treatment temperature: 450 and 600^0C 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

An assessment evaluation of removed phosphorus concentration was carried out based on its as-beneficiated concentration and treatment temperature. A model was derived from the generated experimental results, validated and used as a tool for the analysis. The validity of the two-factorial model was found to be rooted on the expression $\zeta - 0.01\tau = 0.0969 \ln \vartheta - 0.531$ where both sides of the expression are correspondingly approximately equal. Statistical analysis of the experimental and derived model-predicted removed phosphorus concentrations for each value of the treatment temperature considered shows

standard errors of 0.0093 and 0.0011% respectively. Furthermore, removed phosphorus concentration per unit rise in the treatment temperature as obtained from experiment and derived model-predicted results were 2.31 x 10^{-4} and 2.05 x10^{-4} % / ^{0}C respectively. Deviational analysis indicates that the derived model gives best-fit process analysis at deviations 4.63, 4.68 and 5.8% from experimental results and invariably, operational confidence level range: 94.2 - 95.37%. The deviations corresponded to treatment temperature range: 450-550^0C.

REFERENCES

[1] Patent Publication No. 101037724, 2007

[2] Anyakwo, C. N., and Obot, O.W., (2008). Phosphorus Removal from Nigeria`s Agbaja Iron Ore by Aspergillus niger, Inter. Res. J. Eng. Sc. Tech. 5(1):54-58.

[3] Nwoye, C. I., C. N. Mbah., C. C. Nwakwuo., and A. I. Ogbonna. (2010). Model for Quantitative
Analysis of Phosphorus Removed during Leaching of Iron Oxide Ore in Oxalic Acid Solution. J. Min. Mater. Charact. Eng. 9(4):331-341.

[4] Nwoye, C. I. (2009).Model for Evaluation of the Concentration of Dissolved Phosphorus during Leaching of Iron Oxide Ore in Oxalic Acid Solution. J. Min. Mater. Charact. Eng. 8(3):181-188

[5] Nwoye, C. I., Agu, P. C., Mark, U., Ikele, U. S., Mbuka, I. E., and Anyakwo, C. N. (2008) Model for Predicting Phosphorus Removal in Relation to Weight of Iron Oxide Ore and pH during Leaching with Oxalic Acid. Inter. J. Nat. Appl. Sc. 4(3):292-298

[6] Nwoye, C. I. and Ndlu, S. (2009). Model for Predictive Analysis of the Concentration of Phosphorus Removed during Leaching of Iron Oxide Ore in Sulphuric Acid Solution J. Min. Mater. Charact. Eng. 8(4):261-270.

[7] Nwoye, C. I. (2010). Model for Predicting the Concentration of Phosphorus Removed during
Leaching of Iron Oxide Ore in Oxalic Acid Solution. J. Eng. Appl. Sc. 6(1,2):23-29.

[8]E. E. Nnuka, E. M. Ameh and C. I. Nwoye.(2013). Empirical analysis of phosphorus removal based on its as-beneficiated concentration and mass-Input of $KClO_3$. American Journal of Research Communication, 1(8): 32-42. www.usa-journals.com,

[9] Ameh, E. M. (2013). Dephosphorization of Agbaja Iron Ore By Sintering And Bioleaching Processes. Ph.D Thesis, Nnamdi Azikiwe University Awka, Anambra State, Nigeria.

[10] Nwoye, C. I. (2008). C-NIKBRAN; Data Analytical Memory.