

## Beneficiation Processing of Agbaja Iron Ore By Chemical Leaching Technique

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**ABSTRACT:** The effects of dwell time, dilution ratio, leachant concentration and variables interaction on the dephosphorization of Agbaja iron ore have been investigated by chemical leaching [ $H_2SO_4$ ] technique and  $2^3$ -factorial surface response methodology. Results indicate 96%, 95% and 96% of phosphorus removal at 36 min leaching time, 0.3M leachant concentration and 1:2 dilution ratio, respectively. Iron loss during the leaching was less than 0.15%. It can be concluded that acid leaching of Agbaja iron ore is feasible at the conditions of the experiment. [S.M.O. Obiorah, M.C.Menkiti, E.E.Nnuka. **Beneficiation Processing of Agbaja Iron Ore By Chemical Leaching Technique**. New York Science Journal 2011;4(5):22-27]. (ISSN: 1554-0200). <http://www.sciencepub.net/newyork>.

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### INTRODUCTION

Nigeria is one of the richest countries of the world in terms of mineral deposits. Among these deposits is iron ore, located at Agbaja, Kogi State, Nigeria. Agbaja iron ore deposit, the largest in Nigeria is about 1.3 billion tones. Agbaja iron ore is of low silicon modulus ( $SiO_2/Al_2O_3$ ), fine texture and contains about 1.4-2.0% phosphorus. The prevalence of this amount of phosphorus is a major setback to its utilization in the blast furnace or direct reduction process (Alafara *et al*, 2005).

The removal of phosphorus from iron and steel presents problems because of similarity of the standard free energies of formation of iron oxide and phosphorus pentoxide (Alafara *et al*, 2005). Consequently, in the reducing conditions of the blast furnace to recover some 99.5% of the iron charged, near complete reduction of phosphorus pentoxide from the acid blast furnace occurs. As the phosphorus in the ore impregnates the pig iron, there occurs two distinct processes of tackling the problem: pyrometallurgical route and hydrometallurgical route. The first route employs basic slag during the conversion to steel. This technique covers the activity coefficient of phosphorus pentoxide in the slag (Alafara *et al*, 2005; Alafara *et al*, 2003; Li and Wen, 2004; Whiteley, 1923). The second route delves into ways of reducing phosphorus in the iron at relatively low temperatures.

Leaching of lean ores or complex ore in different acids has proved successful for several years. However, the leaching of phosphorus contaminated iron ore has made a very limited progress. This underscores the ongoing intense research in the area for

several decades. Depending upon the degree of association of phosphorus with the minerals in the iron body, iron ore can be beneficiated in either of the three ways: smelting process, physical separation and chemical leaching (Kokal, 1990).

As is well known, smelting process is effective for dispersion but with very high cost, and it is still under fundamental research. For physical separation, comminution followed by wet magnetic separation or froth flotation is generally employed when the phosphatic gangue mineral appears as discrete inclusion in the iron body matrix (primary mineralization) (Kokal, 1990; Kokal *et al*, 2003; Fonesca *et al*, 1994). Low phosphorus extraction, high grinding cost and iron loss are the major disadvantages of the method. However, when phosphorus is disseminated in the iron structure, possibly forming cryptocrystalline phosphates or solids solutions with the iron oxide phases (secondary mineralization), the beneficiation can only proceed by chemical routes (Kokal, 1990; Kokal *et al*, 2003; Forsberg and Asolfesson, 1981).

Forsberg and Asolfesson (1981); Cuj and Fang (1998); Hang *et al*, (1994); He and Zhou, (2000); Balajiva *et al*, (1946); investigated dephosphorization with acid leaching. In their studies, the acid concentrations were very high and low phosphorus extraction were obtained. In this study, the feasibility of dephosphorization of Agbaja iron ore by acid leaching at various dilution ratio, dwell time and particle size were investigated. Also, the Design of Experiment for the evaluation of interactive factorial effects on the phosphorus removal was also investigated.

## MATERIALS AND METHOD

### Sample Collection

Iron Ore samples of 0.40mm particle size of Agbaja iron ore were collected from National Metallurgical Development Centre( N M D C), Jos, Plateau State, Nigeria. The samples were stored in a dry environment for analysis after being sieved to produce particle size fraction of 0.2-0.4mm.

### Determination Of Iron And Phosphorus Phase Content Of Untreated Ore

The phases in which iron and phosphorus phases were present within the iron ore were determined by x-ray diffraction(XRD) analysis and atomic absorption spectrophotometry.

### Chemical Preparation Of Leaching Solution

All leaching chemicals used were of laboratory grade, procured from Bridge Head Chemical Market, Onitsha, Nigeria. 1M leaching solutions of sulphuric acid were prepared by diluting the concentrated form of the acid in distilled water. The dilution ratios employed were 1:0,1:1,1:2,1:3,1:4,1:5,1:6.

### Chemical Leaching Procedure

Cotton wool was rammed into the bottom part of chromatographic column followed by the introduction of fine granules of crushed ceramics. Thereafter, 0.4mm of 20g of Agbaja iron ore was introduced into the tube and the tube clamped. 100ml of 1M H<sub>2</sub>SO<sub>4</sub> was introduced into the clamped column containing the 20g iron ore. A conical flask is placed beneath the column to receive the leach liquor. The leaching time and leachant dilution were varied at 31°C ,0.4mm particle size. At the end of each leaching treatment, the residue was removed from the column and washed for 3 times with distilled water. The treated ore samples were dried in oven at 150°C for 24 hours. The respective leached liquor were stored in flask for phosphorus content analysis.

### Design Of Experiment For The Leaching

Statistically designed experiment was conducted for the leaching treatment based on 2<sup>3</sup> full factorial design. The design matrix for the three variables, two levels and eight experimental runs and responses are shown below:

**Table 2.1: Design matrix of DOE and responses.**

S/NO	X0	X1	X2	X3	Y <sub>ave</sub> (mg/g)
1	+1	-1	-1	-1	7.75
2	+1	+1	-1	-1	15.0
3	+1	-1	+1	-1	15.50
4	+1	+1	+1	-1	15.00
5	+1	-1	-1	+1	7.75
6	+1	+1	-1	+1	6.50
7	+1	-1	+1	+1	5.55
8	+1	+1	+1	+1	4.35

Also, shown below is the experimental range and levels of independent process variables.

**Table 2.2: Experimental range and levels of Independent process variables.**

Independent Variable	Lower limit (-1)	Base level (0)	Upper limit (+1)
Dilution ratio	0.20	0.60	1.00
Particle size	0.20	0.40	0.60
Dwell time	20.00	30.00	40.00

## RESULTS AND DISCUSSION

X-Ray diffraction (XRD) analysis of the untreated ore revealed the major mineral phases present as hematite (hematite), quartz (Si<sub>2</sub>O<sub>3</sub>), alumina(Al<sub>2</sub>O<sub>3</sub>) and phosphorus pentoxide( PO<sub>5</sub>) while the X-ray fluorescence spectroscopy (XRF) and atomic absorption spectroscopy (AAS) revealed the chemical composition as shown in table 3.1. Since phosphorus contained within the iron ore is non-sulphidic phase, acid leaching presents the best option for its removal.

### Effect Of Leaching time

The summarized effects of acid leaching time on dephosphorization of the ore samples are shown in table 3.2. From the table, it could be seen that the phosphorus removal from the ore reached maximum values of about 96% at 36 minutes during the leach process. The results indicate that as the leaching time decreases, there is corresponding decrease in the amount of phosphorus removed.

### Effect of Leachant Concentration

The effects of leachant concentration is presented in table 3.3 for samples A-B. There was increment in percentage of leached liquor as the leachant concentration increased from 0.13-0.3M. Above 0.3M, the % phosphorus decreased from 92% to 47%. The maximum phosphorus removal of 95% was achieved at 0.3M leachant concentration. The initial low phosphorus removal using the concentrated acid could be due to cations present(Forsberg and Asolfesson,1981).

### Effect Of Acid Dilution Ratio

The effect of acid dilution ratio is shown in 3.4. From the table, it can be shown that phosphorus extraction increases as the dilution ratio increases and reaches a maximum at 1:2 dilution of the H<sub>2</sub>SO<sub>4</sub> corresponding to maximum of 96% phosphorus removal. As the dilution ratio increased further, there was a progressive decrease in phosphorus extraction.

### Iron Loss During Leaching

Iron loss recorded after the leaching process is shown in table 3.5. It can be seen that iron content in the leachate (leached liquor) decreases as the dilution ratio increases. Quantitatively, it can be shown that iron loss is less than 0.1%, an amount that can be practically considered negligible.

### DOE Interaction Effects And Responses

A  $2^3$  full factorial experimental design with two replications generated 16 experiments employed in this study. The design matrix is shown in table 2.1. The parameters: dilution ratio ( $X_1$ ), particle size ( $X_2$ ) and dwell time ( $X_3$ ) were chosen as independent variables at two levels while phosphorus removal is the output response. The analysis is focused on how the phosphorus removal (dependent output variable) is influenced by the independent variables, i.e.  $X_1$ ,  $X_2$  and  $X_3$ . The dilution ratio range studied was between 0.2 and 1, particle size varied between 0.2 and 0.6mm and dwell time in the range of 20 to 40min as shown in table 2.2. In order to study the combined effect of these factors, experiments were performed at different combinations of the physical parameters using statistically designed experiments. Upon the determination of polynomial coefficients, statistical analysis (CSI, G-Test, F-Test, T-Test) were performed to develop model that is adequate, significant and homogeneous (variance wise) (Menkiti, 2010). The main effects of the parameters and response behavior of the system are explained by equation shown below:

$$Y_u = 9.675 + 0.493X_1 + 0.425X_2 - 3.637X_3 - 0.9625X_1X_2 - 1.150X_1X_3 - 1.512X_2X_3$$

The corresponding interactive surface response plots are presented in figures 1-3. Figure 1 shows the interaction effect of dwell time and particle size on phosphorus removal. In respect of figure 2, the interaction effect of dilution ratio and particle size is presented while figure 3 shows the interaction effect of dilution ratio and dwell time. It is important to note that the value of output responses are tied to the intensity of the colour of surface plots. The plots provide avenue to observe the surface area of the curve within which the process can perform at optimal level based on the effects of the interaction of the variables under consideration. The significance of these interaction effect between the variables would have been lost if the experiments was conducted by traditional method.

**Table 3.1: Major Elemental Composition of Agbaja Iron Ore Deposit**

Major Elements	Mass (%)
Fe	47.40
SiO <sub>2</sub>	10.25
Al <sub>2</sub> O <sub>3</sub>	11.35
CaO	0.88
MgO	0.22
TiO <sub>2</sub>	0.31
MnO	0.15
P <sub>2</sub> O <sub>5</sub>	1.60
S	0.07

**Table 3.2 : Effect of leaching time**

S/NO	Sample	Leaching time (min)	Residue Wt(g)	% P in leached Liquour
1	A	60.00	17.30	48.00
2	B	38.00	19.50	93.00
3	C	36.00	19.80	96.00
4	D	24.00	20.00	93.00
5	E	23.00	20.00	48.00
6	F	22.00	20.00	41.00
7	G	21.00	20.00	35.00
8	H	20.00	20.00	29.00

**Table 3.3 : Effect of leachant concentration**

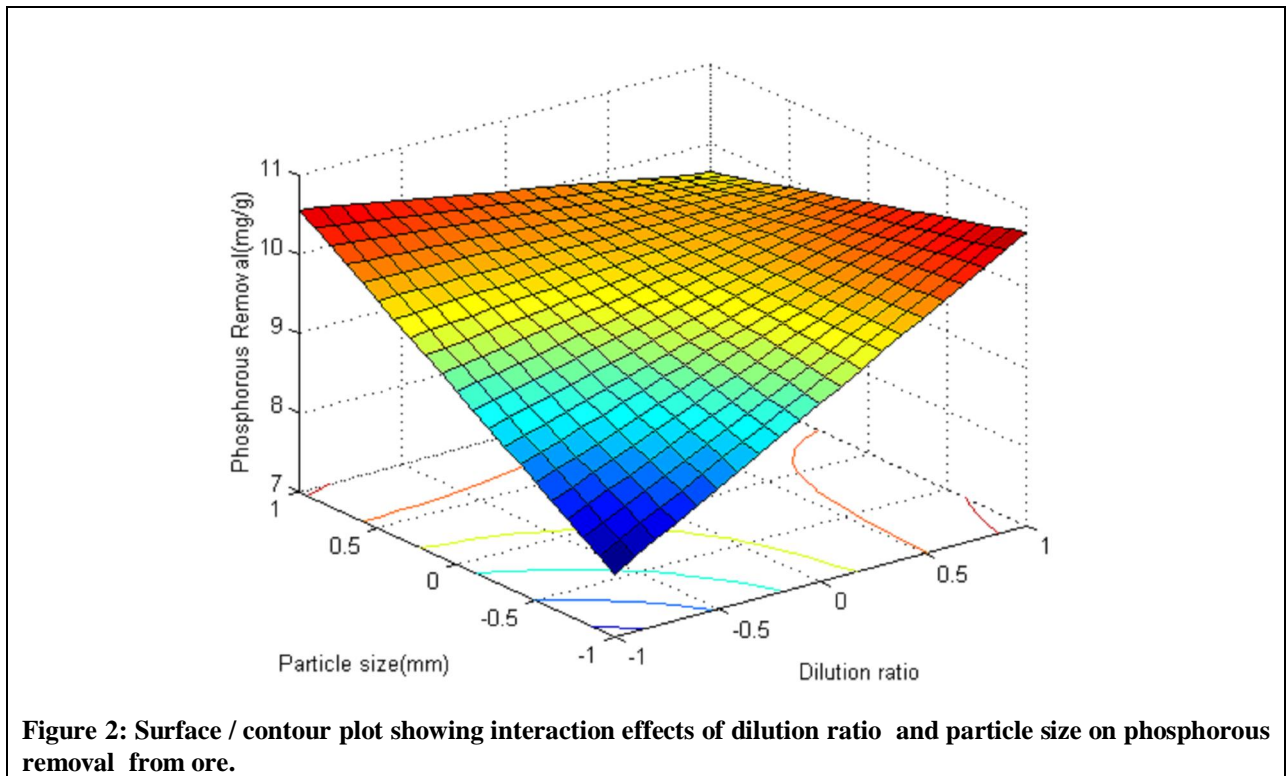
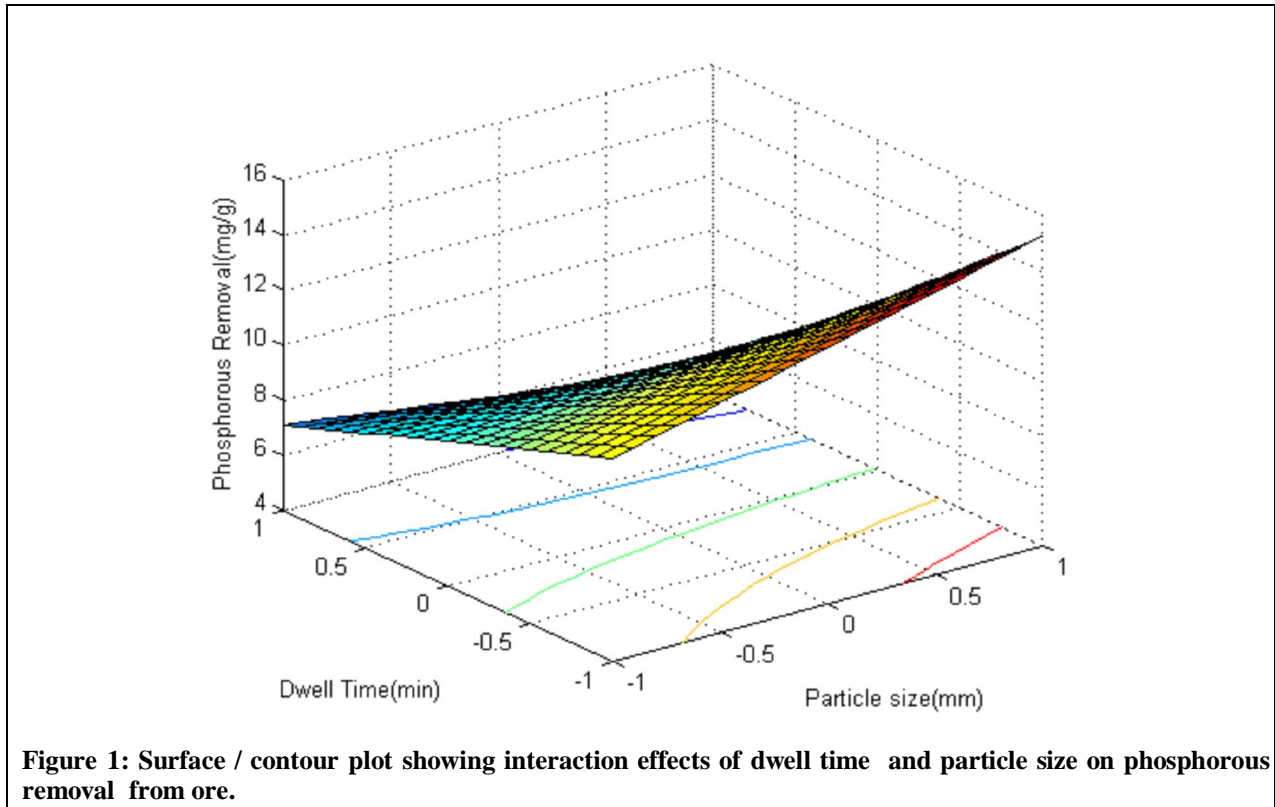
S/NO	Sample	Leachant Conc (M)	Residue Wt(g)	% P in leached Liquour
1	A	1.00	16.47	47.00
2	B	0.50	18.58	92.00
3	C	0.30	18.88	95.00
4	D	0.25	19.05	92.00
5	E	0.20	19.02	47.00
6	F	0.17	19.02	43.00
7	G	0.14	19.02	40.00
8	H	0.13	19.02	31.00

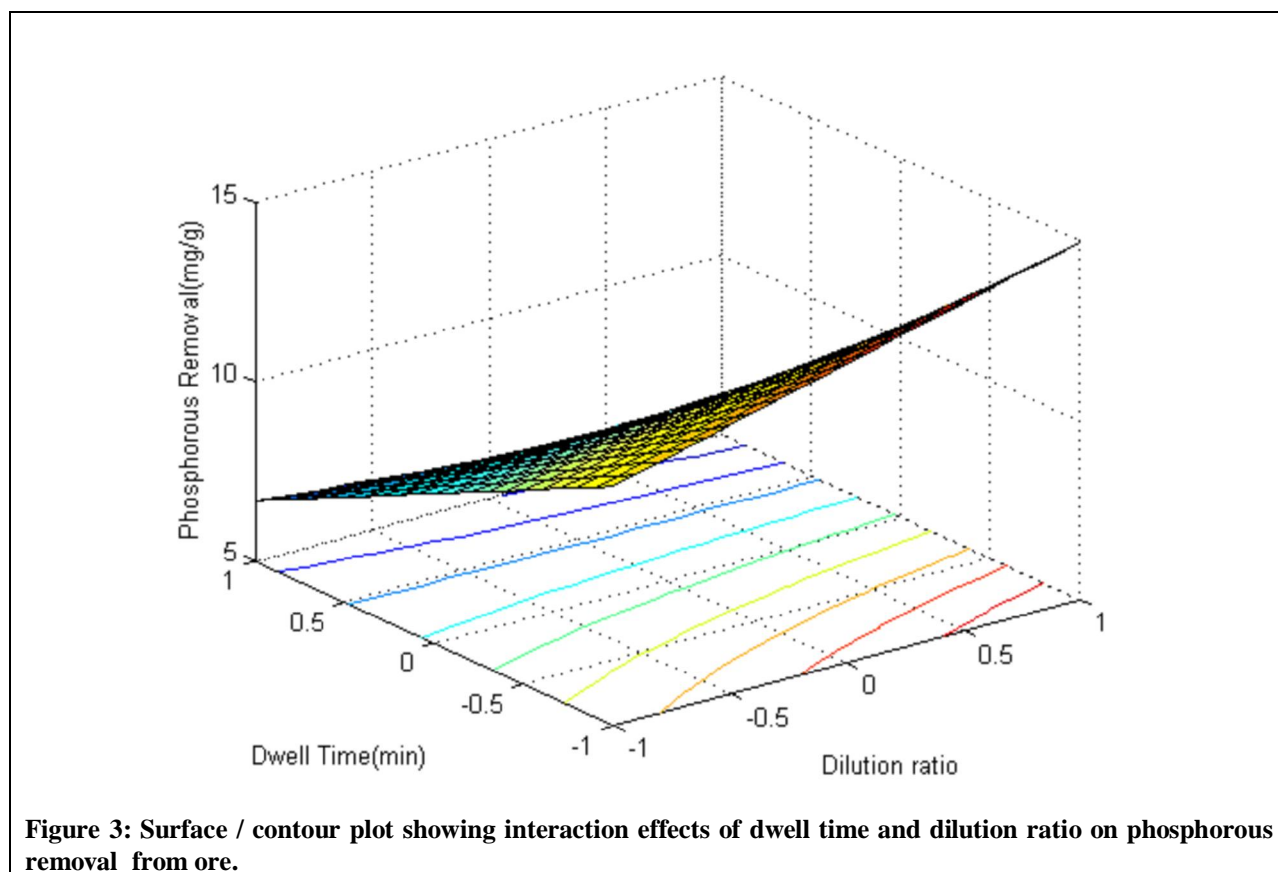
**Table 3.4 : Effect of dilution ratio on dephosphorization**

Specimen sample	Dilution ratio	% Conc of Phosphorous in the tailing
A	1:0	48.00
B	1:1	93.00
C	1:2	96.00
D	1:3	93.00
E	1:4	48.00
F	1:5	41.00
G	1:6	35.00
H	1:7	29.00

**Table 3.5 : Iron loss from the leached liquor**

Sample	ppm
A	2.14
B	2.12
C	1.23
D	1.04
E	1.04
F	0.60
G	0.50
H	0.36





**Figure 3: Surface / contour plot showing interaction effects of dwell time and dilution ratio on phosphorous removal from ore.**

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