DEPHOSPHORIZATION OF KOTON-KARFE IRON ORE BY LEACHING WITH   SULPHURIC ACID

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ABSTRACT.
Dephosphorization of Koton-karfe iron ore Kogi State, Nigeria has been studied. Sulphuric acid was used to leach -355 +250µm and -180 +125µm size fractions, the analysis was carried out using X–Ray fluorescence (XRF) Spectrometer andinductively-coupledplasma optical emission spectroscopy(ICP-OES). The leaching was conducted under varying time, concentration, agitation and temperature. The optimum leaching conditionswere attained at a time of 80minutes, concentration of 0.9M, agitation of 500rpm and temperature of 30°C. leaching of -355 +250µm and -180 +125µm particle sizes at optimum condition gave 41.41 and 45.40% dephosphorization respectively. This indicates that mostly phosphorus associated with $AlPO_4$is leached. ICP-OES results reveal that the iron that dissolved in the leach liquor is minimal, and the pH measurements reveal minimal amount of acid is used, therefore acid can be reclaimed and recycled.

1.0 INTRODUCTION

The technological advancement and growth of industrialization from the eighteenth century upward has led to the increase in the demand of most mineral commodities, particularly metals. In the last ten years, the situation of iron ore markets has changed dramatically due to an increase in the world steel consumption, pushed up mainly by the economic growth of China and other Asian emerging markets. Currently, prices of iron ores are increasing in real terms and the production of iron and steel is increasing as well (Poveromo, 2005). This new scenario for the iron mining industry has revived theinterest in upgrading the quality of phosphorus bearing iron ores

Nigeria is one of the countries in the world blessed with large reserves of iron ores some of which have been declared suitable for use in the production of iron and steel after beneficiation, but the largest deposit in Nigeria the Agbaja iron ore containing 47.80%Fe; estimated at two (2) billion tonnes and the second largest deposit the Koton-karfe iron ore containing48.125%Fe; estimated at four hundred and twenty eight (428) million tonnes have high phosphorus content(2.08%$P_2O_5$for Agbaja iron

Ore and 2.14%$P_2O_5$for Koton-karfe iron ore) and fine grain texture are not suitable for use in iron and steel making in Nigeria (Baba *et. al*, 2005).

Elements such as phosphorus (P) and sulphur (S) when contained in large amount in iron ores have detrimental effects on the steel produced .Phosphorus impacts to steel cold shortness. Large quantity of phosphorous reduces the ductility of steel, thereby increasing the tendency of the steel to crack when cold worked (Serdar,1999).The depletion of the richer iron ore deposits as a result of advancing global technologies and civilization necessitated the processing of lower quality iron ores (Jian and Sharma 2004).

In this research, an attempt is made to dephosphorize the Koton-karfe iron ore by leaching with sulphuric acid. Various attempts have been made by researchers to make possible the utilization of the high phosphorus iron ore deposits. Among such attempts is the use of Hydrometallurgy.

Asuke *et. al*., (2018a) studied the dephosphorization of Koton-karfe iron ore by leaching with citric and hydrochloric acids. They varied leaching time,

temperature, acid concentration and agitation and developed the optimum leaching condition. They reported that hydrochloric acid was a better leaching agent than citric acid, dephosphorizing 35.13% at -180 +125µm size fraction. With both acids, only phosphorus in $AlPO_4$ was leached while phosphorus in solid solution with goethite could not be leached.

Williams and Cloete 2008 reported an attempt to investigate the most effective acids to remove phosphorus and potassium from Sishen iron ore concentrates by chemical leaching using a range of acids and oxidative chemicals, the acids used are, acetic, citric, hydrochloric, lactic, oxalic, propanoic, sulphuric acids and ferric sulphate. One molar concentrations of all leaching acids were used for five days at room temperature. They reported that citric acid proved to be best leaching agent for removing potassium from the ore, while sulphuric acid was detected to be the most effective to remove phosphorus and most corrosive on iron.

The feasibility of dephosphorization of Changde (China) phosphorus containing iron ore (1.12%P) was investigated using alkali and acid leaching. Sulphuric, hydrochloric, nitric acids and sodium hydroxide were used for the leaching. They reported that Alkali-leaching is ineffective to remove phosphorus from the iron ore. With 0.2 mol/L and 1.0 mol/L sodium hydroxide, only 24.41% and 27.96% phosphorus was removed respectively. A prolonged alkali-leaching would go against dephosphorization because of the precipitation of Phosphorus with other metal ions released during the leaching. Acid-leaching is an effective method to remove phosphorus from Changde iron ore. Sulfuric acid is more effective for dephosphorization than hydrochloric acid and nitric acid. 91.61% phosphorus is extracted with 1.0% sulfuric acid after leaching for 20min at room temperature. The optimum stirring speed is 620 revolution per minute. Iron loss during theacid-leaching can be negligible, which is less than 0.25%, making the process quite economical Jin Yong-shi *et. al*., (2006).Rawling (2007),

worked on the chemical leaching of Iron ore using a range of oxidants, and concluded that citric acid proved to be the best leaching agent for the removal of potassium from the iron ore, while sulphuric acid reduced the amount of phosphorus from the ore.

Matsuo *et al*., (1980) investigated the dephosphorization of iron ore using acid leaching. The iron ore contained phosphorus in the form of apatite, $Ca_5(PO_4)_3F$. The acids tested were hydrochloric, nitric and sulphuric acids. The optimum acid concentration was determined to be between 2 – 7%. The optimum leaching time was found to exceed 10 hours. Without the use of heat treatment, a 20% reduction in the phosphorous content was achieved. With heat treatment, a reduction of phosphorus of up to 40% was obtained.

Based on these backgrounds, little or no work has been done on the leaching of Koton-karfe iron ore with sulphuric acid. It is for this reason that a chemical process of reducing the phosphorus content was considered.

2.0 MATERIALS AND METHODS.
2.1 Materials and Equipment
The materials used include ;Koton-karfe Iron ore sample, sulphuric acid and filter papers. The equipment used include: Denver pulverizing machine, Denver jaw crushing machine, Denver sieve shaking machine, X – Ray fluorescence Spectrometer (XRF), Inductively Coupled plasma optical emission spectroscopy(ICP-OES), Conical Flasks and Beakers, Digital Weight Scale, Drier, magnetic stirrer and a pH meter

2.2 Material Collection and Preparation:
The sample of the iron ore used for this research work was obtained from the Koton-karfe Iron ore deposit in Kogi State, Nigeria. 80kg of the Samples were collected from different points on the deposit by simple random sampling. The ore was crushed and ground using the Denver crusher (JC - 300) and ball milling machine (GMO85) respectively. The ground ore was sieved to produce particle size fractions of

-355 +250µm, and -180 +125µm as earlier reported in Asuke *et al*. (2018b).

2.3 Determination of the chemical composition of the head Sample.

The XRF analysis was done in the Faculty of Natural and Agricultural Science, Geology Department, University of Pretoria, Pretoria South Africa. Thermo fisher ARL9400 XP+ Sequential XRF and uniquant software was used for the XRF analysis. Before the analysis, the ore was first milled to -75µm, the samples were prepared as pressed powder briquettes as required by the machine standard for proper mineral count and analysis. Each sample was placed in the machine for one hour thirty minutes (thirty minutes for major elements and one hour for trace elements). The software used analyzed for all elements in the periodic table between Sodium (Na) and Uranium (U), but only elements found above the detection limits were reported. All major elements were expressed as oxides and analyzed in percentage (%) composition.

2.4.1 Leaching reagents used.

Sulphuric acid (H_2SO_4) used for the research was prepared from analytical grade by dissolution of calculated amount in deionized water. Sulphuric acid (H_2SO_4) used had 98% concentration and density 1.84g/cm^3 (batch no 350313)

2.4.2 Leaching Procedures:

The leaching was carried out in four stages:
Stage I:40g of-355 +250µm particle size was weighed, and then transferred into 500ml conical flask containing 200ml of0.3M sulphuric acid at temperature of 30^0. The mixture was covered with glass lid and agitated with the aid of magnetic stirrer at an initial rate of 100rpm. The mixture was leached for a constant time of 1hr after which the solution was filtered, the residue washed thoroughly and taken for analyses. The process was repeated varying agitation speed from 200 – 500rpm at an interval of 100rpm.
Stage II: From stage I the best agitation was determined and used in this stage. The leaching was carried out at constant time (1hr), acid concentration (0.3M), agitation of 500rpm and an initial temperature of 30^0Cafter which the solution was filtered, the residue washed thoroughly and taken for analyses. The stage II process was repeated with varying temperature of 40 - 80^0Cat an interval of 10^0C.
Stage III: With the optimum parameters developed from stages one and two for agitation and temperature respectively, the effect of the variation of concentration and time were studied at this stage. An initial concentration of 0.15M was used with varying time from 0 – 120minutes at an interval of 20minutes (at 500rpm and 30^0C), after every 20minutes of leaching, samples were taken, filtered and the residue washed thoroughly with distilled water, dried and analyzed and the leach liquor kept for further analysis. This process was repeated but varying concentration from 0.3 – 1.2M at an interval of 0.3M; at each concentration varying time as stated above.
From the above procedures, the best leaching parameters were attained.
Stage IV: In this stage, the effect of the variation of particle size was investigated. The optimum conditions developed from stages I - III above were used to leach -180 +125µm size fraction.

2.5 Determination of phosphorus content before and after leaching of the ore.

The phosphorus content of the ore before and after leaching was determined using XRF analysis. The samples were prepared using the same method as earlier described.

2.6 Determination of the Amounts of Phosphorus removed during Leaching with Acids.

Reduction in the contents of phosphorus as a result of the acid-leaching process was calculated using the results from the XRF analysis in equation (2.1);

$$R_P = \frac{\%P_B - \%P_A}{\%P_B} \; X \; 100 \; (2.1)$$

Where R_P = Reduction in Phosphorus Content (%), P_A, P_B= phosphorus content after and before leaching (%) respectively.

2.7Analysisof the acids before leaching using inductively coupled plasma optical emission spectrometry (ICP-OES).

The acid before leaching and the leach liquor were analyzed using inductively coupled plasma optical emission spectrometry (ICP-OES) using standard procedure as described in Asuke, 2014

2.8   pH Measurement of the acids before and after leaching

The pH of the leach solution before and after leaching was measured. This was carried out using the hand-held pH/ORP meter (Model No. 8651). The pH meter was calibrated before use and the probe was always rinsed in fresh de-ionized water before use in order to remove any impurity adhering to the probe.

2.9 Determination of the Amounts of Iron dissolved in acid during Leaching.

The amount of iron, aluminium and silicon lost to the leaching process was calculated using the results obtained from the ICP-OES analysis and equation (2.2);

$$R_{Fe} = \frac{\%Fe_B - \%Fe_A}{\%Fe_B} \qquad (2.2)$$

R_{Fe} = dissolved iron content (%), Fe_B, Fe_A = iron content before and after leaching (%) respectively,

The principle applied in equation (2.2) was also used to determine the amount of aluminium, silicon, manganese, cobalt and nickel dissolved in the acid after leaching.

3.0 RESULTS AND DISCUSSIONS
3.1 XRF Analysis of the selected particle sizes before leaching

XRF analysis of the selected particle sizes-355 +250µm and -180 +125µm before leaching are presented in Table 3.1. Based on the distribution of the iron and phosphorus within the particle sizes. Particle size of -355 +250µm containing the highest amount of P_2O_5 (1.98%) is selected to allow maximum exposure of phosphorus to the leaching condition, while -180 +125µm was selected to investigate the effect of particle size on the leaching with respect to the highest distribution of iron

(69.81%) which is also the liberation size as earlier reported elsewhere (Thomas 2008).

Table 3.1: XRF of selected particle sizes before leaching.

	Percentage Composition	
Constituent oxides	**-355 +250 µm**	**-180 +125 µm**
SiO_2	15.81	14.06
TiO_2	0.24	0.22
Al_2O_3	12.20	12.25
Fe_2O_3	67.73	69.81
MnO	0.45	0.43
MgO	0.47	0.39
CaO	0.35	0.22
Na_2O	*<0.01*	*<0.01*
K_2O	0.08	0.08
P_2O_5	1.98	1.85
Cr_2O_3	0.02	0.02
NiO	0.02	0.02
V_2O_5	0.07	0.07
ZrO_2	0.01	0.01
SO_3	0.15	0.17
WO_3	0.07	0.05
BaO	0.07	0.07
Cl	0.06	0.05
CuO	*<0.01*	0.01
ZnO	0.05	0.05
Co_3O_4	0.07	0.07
SrO	0.05	0.05
MoO3	0.02	0.02
RuO4	*<0.01*	*<0.01*
Rh2O3	0.01	0.01

Note: All particle sizes are in µm.

3.2   Leaching of -355 +250µM Particle Size.
3.2.1 Variation of Agitation on leaching of -355 +250µm particle size.

The first stage was to investigate the effect of variation of agitation on the leaching of -355 +250µm particle size fraction.

The agitation was varied from 100 – 500rpmat constant time of one (1) hour, temperature of 30ºC and 0.3M acid concentration for sulphuric acid. Generally after leaching and washing, XRF was carried out on the residue from where values are used to calculate the amount of phosphorus leached using equation (2.1). Table 3.2. Present the effect of the variation of agitation on the amount of phosphorus leached.

Table 3.2: The effect of agitation on the leaching of -355 +250µm particle size.

ACID	AGITATION (rpm)/ PHOSPHORUS LEACHED(%)					
	100	200	300	400	500	
H_2SO_4	10.10	21.71	11.11	22.73	23.74	

Table 3.3: Variation of temperature with the amount of phosphorus leached

ACID	TEMPT (^{0}C)/ PHOSPHORUS LEACHED (%)					
	30	40	50	60	70	80
H_2SO_4	23.74	17.68	12.12	11.11	5.55	8.08

Table 3.4: Variation of sulphuric acid concentration and time on the leaching of Phosphorus in the -355 +250µm particle size.

CONC.	TIME(Mins)/LEACHING RATE (%)					
(M)	20	40	60	80	100	120
0.15	7.58	20.71	9.6	7.58	19.7	18.69
0.30	6.57	7.07	23.74	22.73	8.08	20.71
0.60	20.71	7.58	6.06	7.07	21.21	7.58
0.90	9.6	25.76	33.84	41.41	32.83	19.7
1.20	10.61	10.1	11.11	25.76	10.1	21.21

Table 3.5: The developed optimum leaching conditions for the selective leaching of Phosphorus from -355 +250 particle size of the Koton-karfe iron ore using sulphuric acid.

ACIDS	CONDITIONS				Leaching rate (%)
	Temp. (^{o}C)	Conc. (M)	Time (mins)	Agitation (rpm)	
Sulphuric	30	0.9	80	500	41.41

From Table 3.2 the least percentage of phosphorus leached (10.10%) is obtained at 100rpm while the highest phosphorus leaching (23.75%) is obtained at 500rpm.

3.2.2 Variation of Temperature on leaching of -355 +250µm particle size.

The effect of the variation of temperature on the leaching of -355 +250µm particle size was the second stage of the leaching. The temperature was varied from 30 – 80ºC at an interval of 10ºC and a constant time of one (1) hour and concentration of 0.3M with agitation of 500rpm.Table 3.3present the result for the variation of temperature on the phosphorus leaching.
From Table 3.3 the acid performs better at 30ºC with phosphorus leaching of 23.74% for sulphuric acid, the phosphorus leaching drops as the temperature increases.

This is in line with initial reports by Xia *et. al* 2011.

3.2.3 Variation of Concentration and time on leaching of -355 +250µm particle size.

This stage involves the variation of time from 20 – 120minutes on an interval of 20minutes and concentrations of 0.15, 0.3, 0.6, 0.9 and 1.2M. The leaching was carried out at constant temperature of 30ºC, and agitation of 500rpm. Table 3.4 presents the effect of variation of time and concentration on the selective leaching of phosphorus with sulphuric The results from Table 3.4 show that the best leaching rate was obtained at 0.9M concentration at a time of 80minutes. The results obtained were recorded as the optimum leaching conditions and summarized in Table 3.5.

Appendix Table A1 show XRF result for leaching at optimum developed conditions.

3.2.4 Leaching of -180 +125 particle size
The developed leaching conditions were used to leach -180 +125µm particle size. Table 3.6 presents the XRF of the ore after leaching

Table 3.6: XRF Results for the leaching of - 180 +125µm particle size under optimum leaching conditions

Constituent	Before (%)	After (%)
SiO_2	14.06	14.63
TiO_2	0.22	0.22
Al_2O_3	12.25	12.05
Fe_2O_3	69.81	70.03
MnO	0.43	0.39
MgO	0.39	0.33
CaO	0.22	0.13
Na_2O	*<0.01*	*<0.01*
K_2O	0.08	0.02
P_2O_5	1.85	1.01
Cr_2O_3	0.02	0.02
NiO	0.02	0.02
V_2O_5	0.07	0.07
ZrO_2	0.01	0.01
SO_3	0.17	0.75
WO_3	0.05	0.03
BaO	0.07	0.06
Cl	0.05	0.01
CuO	0.01	0.01
ZnO	0.05	0.05
Co_3O_4	0.07	0.05
SrO	0.05	0.04

Table 3.6 reveal a drop in phosphorus content from 1.85 to 1.01%P_2O_5which represents 45.40% leaching of phosphorus. This shows an improvement from what was recorded from the leaching of -355 +250µm particle size (41.41%). The improvement in selective leaching of phosphorus with the -180 +125µm particle size is attributed to the increase in surface area which aids the exposure of more mineral to the leach liquor and therefore increases the leaching of phosphorus.

3.5 Phosphorus Leached for the Various Particle Sizes.
Asuke (2014) reported that phosphorus in-355 +250µm and-180 +125µm size fractions exist as $AlPO_4$and in solid solution with goethite, and that -355 +250µm and -180 +125µm size fractions contain 36.74 and 36.65% phosphorus respectively in the $AlPO_4$phase with the percentage balance in solid solution with goethite. Based on this and other reports by Matsuo *et. al*., (1980), Peixoto (1991) and Belikov *et. al*., (2003) it can be concluded that most of the phosphorus leached by sulphuric acid in the koton karfe iron ore is mostly that associated with the $AlPO_4$ phase. Sulphuric acid was only able to leach a little out of the P associated with the Goethite phase, with 41.41% P leached with -355 +25µm particle size (i.e 4.6% above 36.74%P in$AlPO_4$) and 45.40% P leached with -180 +125µm particle size (i.e 8.75% above 36.65% P in $AlPO_4$).

3.6 Dissolution of Iron During Leaching.
Tables 3.7 presents the ICP-OES analysis for the sulphuric acid used before and after leaching the -180 +125µm particle size, it shows the dissolution of some elements in the acid during leaching. Using equation (2.2) the percentage dissolution is calculated.

Table 3.7 reveals dissolution of 0.22%Si, 0.047%Al, and 0.011%Fe. The high dissolution of Silicon associated with sulphuric acid leaching is confirmed by the observation of the formation of a light grey jelly like substance which is suspected to be amorphous colloidal silicon in the course of leaching at higher concentration. Hence the dissolution of iron associated with sulphuric acid at optimum leaching condition is said to be minimal

3.7 pH Values of Acid after Leaching.
The pH values of acid before and after initial leaching at optimum conditions for -355 +250µm particle size (19.3^{o}C) are 0.7 and 0.76 respectively while after leaching the -180 +125µm particle size the pH is 0.78.

Table 3.7: ICPOES analysis of Sulphuric acid before and after leaching at optimum leaching concentration.

	Concentration (mg/l)		Percentage (%)
	Before	After	After
Al	0.7962	467.61	0.0467
Fe	3.1892	110.189	0.0107
Mn	1.2052	16.0269	0.0015
Si	216.21	2442.49	0.2226
Pb	0.0268	0.0738	4.7×10^{-6}
Co	0.4299	0.8006	3.7×10^{-5}

This reveal that the amount of acid used up for the leaching of the -180 +125µm is more than that used for the -355 +250µm. This is in line with the initial results obtained. From the pH values it can therefore be concluded that minimum amount of sulphuric acid is used up for leaching. Therefore, the used-up acid can be treated to reclaim the sulphuric acid and can be recycled.

4.0 CONCLUSIONS
From the analysis and discussions of the results the following conclusions are drawn:
The optimum leaching condition for the selective leaching of phosphorus from the Koton-karfe iron ore with sulphuric acid were attained at a time of 80minutes, concentration of 0.9M, agitation of 500rpm and temperature of 30°C. Leaching of -355 +250µm and -180 +125µm particle sizes at optimum condition gave 41.41 and 45.40% dephosphorization respectively. This indicates that mostly phosphorus association with AlPO$_4$is leached. The iron that dissolved in the leach solution is minimal, and minimal amount of acid is used, therefore acid can be reclaimed and recycled.

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APPENDIX

Table A1: XRF results for the leaching of **-355 +250** µm size fraction at optimum leaching condition.

Constituent	Percentage Composition of **-355 +250** µm	
	Control	After
SiO_2	15.81	16.55
TiO_2	0.24	0.22
Al_2O_3	12.20	11.24
Fe_2O_3	67.73	68.54
MnO	0.45	0.39
MgO	0.47	0.42
CaO	0.35	0.13
Na_2O	*<0.01*	*<0.01*
K_2O	0.08	0.03
P_2O_5	1.98	1.16
Cr_2O_3	0.02	0.02
NiO	0.02	0.02
V_2O_5	0.07	0.07
ZrO_2	0.01	0.01
SO_3	0.15	0.78
WO_3	0.07	0.04
BaO	0.07	0.06
Cl	0.06	0.01
CuO	*<0.01*	0.01
ZnO	0.05	0.05
Co_3O_4	0.07	0.06
SrO	0.05	0.04
MoO_3	0.02	0.01