EFFECTS OF PROCESS VARIABLES ON THE LEACHING OF ITAKPE IRON ORE USING TETRAOXOSULPHATE VI ACID

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ABSTRACT

Effects of process variables (acid concentration, solid/liquid ratio, temperature and time) on the leaching of Itakpe iron ore were examined. The iron ore sample was obtained from Itakpe, Kogi State, Nigeria. It was ground into a fine powder and then sieved to create fractions of 0.1 mm in size. To determine the type of iron ore, XRD (x-ray deffractometer) was employed. The functional groups of the sample were ascertained using FTIR (Fourier transform infrared) spectrophotometer. The total iron content of the ore was measured using an atomic absorption spectrophotometer and the redox titrimetric method. The iron ore sample was beneficiated by treating it with H_2SO_4. For subsequent measurements of iron in aqueous solutions, the redox titrimetric method was employed since the two methods did not differ by more than 5%. The experimental screening found that Itakpe iron ore is largely composed of quartz and hematite. The compositions of the ore changed due to acid treatment, with hematite and goethite as dominant iron minerals. Barite, quartz and halite were present as minor minerals. The FTIR analyses revealed the presence of heteroatoms with free electron pairs and substituent groups in the ore. Leaching efficiency of H_2SO_4 was found to be time, acid concentration, mass/volume ratio and temperature dependent. Maximum value of Fe leached was recorded as 92.05% in the beneficiation process. It showed that H_2SO_4 is suitable for the leaching of Itakpe iron ore.

Keywords: Leaching, Quartz, Hematite, Iron ore, H_2SO_4

1.0 Introduction

Rocks and minerals that contain metallic iron that may be profitably removed are referred to as iron ore. The ores might be dark grey, bright yellow, deep purple, or rusty red, and they are abundant in iron oxides. The iron can be found in various forms such as magnetite (72.4% Fe), goethite (FeO(OH); 62.9% Fe), hematite (Fe_20_3, 69.9% Fe), limonite (FeO(OH), n(H_2O) 55%Fe, or siderite ($FeCO_3$, 48.2% Fe). Ores classified as natural or direct shipping ore have relatively high concentrations of hematite or magnetite (more than around 60% iron). To get to the iron pieces, you have to dig and process the ore. The process of mining involves taking precious minerals out of the earth's crust and organizing related activities including blasting, haulage, drilling, and beneficiation, which eliminates related gangues to improve the ore's grade. In contrast, smelting involves heating the ore until the metal softens and the chemical constituents begin to decompose. The main advantage is that oxygen, a key component of the majority of typical iron ores, is released from the ore. A bloomery is the most basic kind of facility used to smelt iron.

Rocks and minerals that can be economically used to extract metallic iron are known as iron ores. The ores range in colour from dark grey, bright yellow, deep purple, to rusty red and are typically rich in iron oxides. Magnetite (Fe_3O_4), hematite (Fe_2O_3), goethite (FeO(OH) containing 62.5% Fe), limonite ($Fe_2O_3.3H_2O$) containing varying amounts of Fe, siderite ($FeCO_3$) containing 48.3% Fe, pyrrhotite (FeS containing 61.5% Fe), and pyrite (FeS_2 containing 46.7% Fe) are the most common forms of iron itself. Natural ore is defined as an ore containing very high concentrations of hematite or magnetite (greater than 60% iron). (Adeleke, et al., 2014). A mineral body that is large enough, contains enough iron, and has the physical and chemical characteristics to be exploited as an iron source right away or perhaps through leaching is called an iron ore deposit. However, the amount of leachates released into water bodies will depend on the topography of the area and the chemical composition of the mine's wastes. Igneous activity, direct sedimentation, bedded sedimentary deposit creation, segregation or replacement deposits, and enrichment from surface and near-surface weathering are the three geological processes that lead to the production of iron ore deposits.

Iron-rich deposits are created when heavier minerals with iron separate by gravity during the crystallisation process from the solution (Thomas et al, 2019; Itodo et al, 2019; Ani et al, 2020; Orugba, 2014). When surface water falls or subsurface water rises through rock layers, iron-bearing minerals, notably gangue materials like silica, can be deposited. Investors around the world are burdened by this water's continuous flow because it carries pollution with it. In aquatic animals, these contaminants result in longer mortality, health or cognitive problems, and a decline in species variety. Because of iron ore mining, surface water may contain both non-toxic and harmful substances. Some potential sources of harmful pollutants from iron ore mining include tailings ponds, open pits, tailings, ore and sub-ore stockpiles, waste rock dumps, mill tailings from mining, concentration of metal ores, and cleaning. Mechanical operations carry, erode, and deposit rich iron-weathered, insoluble rock minerals. Due to their higher specific gravity, the iron-bearing minerals are deposited before the higher materials. This formation process produces some sedimentary hematite deposits and magnetite beach sands.

Although siderite carbonate may be the only iron mineral found there, magnetite or hematite oxides are found in iron ores. Sequences containing maffic volcanic rocks, shales, and dolomites are frequently linked to iron formation; these sequences are consistent with the depositional environment of the shallow marine shelf (Mitra, 2013). Steel is subsequently made primarily from the iron that is extracted from iron ore. Steel is utilised in the construction of thousands of different products, including bicycles, paper clips, paper clips, locomotives, ships, cars, and beams. In terms of tonnage and use, it is the most popular metal.

Leaching is a macroscopic process where a substance's mass flows through the permeable solid's boundaries (Crundwell 2013; Ekmekyapar et al, 2015; Adeleke et al, 2011; Van et al, 2009). When a solid substance dissolves in an aqueous solution, this process occurs. In other words, metals that are immobilised within minerals are converted into metal ions and released into the aqueous solution, where they become mobilised (Mandal et al, 2001; Alafara et al, 2017; Alafara et al, 2018; Almeida and Schneider, 2020). Because of the continuously rising concentration of iron ore weight-input-fixed hydrogen ions, it was observed that the concentration of dissolved Fe increased gradually with an increase in the weight-input of iron oxide ore.

Experiment design is the process of planning experiments such that pertinent data may be analyzed using statistical methods to provide trustworthy and unbiased results. However, estimating the impact of changing input elements on the experiment's outcomes or solutions is one of the primary objectives of experimental design. When planning an experiment, deliberate modifications are made to the input variables of a system or process, and the impact of those modifications on the response variables is tracked (Kalil et al, 2000). The design of experiments can be beneficial for both physical processes and computer simulation models (Xian et al, 2012; Qui et al, 2014; Onyedika et al, 2013). Factorial, Response surface, combined mixture, and other techniques are used in experiment design.

Response surface methodology (RSM) is a set of statistical and mathematical methods that are useful for developing, improving, and streamlining processes. According to specific factorial-based experimental designs, it can be used to describe the relationship between the response and the independent variables with a small number of trials. Shorter turnaround times and reduced costs are RSM the main advantages of RSM over traditional techniques (Silver et al, 2004). It can be used to optimise any process where multiple variables affect the desired response (Saliuet et al, 2009). The purpose of this study is to examine the optimisation of iron ore leaching using H_2SO_4 in various Nigerian sediments.

2.0 Materials and Methods

The following tools were used in this study: x-ray diffractometer (XRD); atomic absorption spectrophotometer; Fourier transform infrared spectrophotometer (Cary 630, Agilent Technologies USA); volumetric flasks; beakers; conical flasks; measuring cylinder; funnel; electronic weighing balance; water bath; stop watch; thermometer; retort stand; petri dish; H_2SO_4; distilled water; and scanning electron microscopy (SEM – model: Rhenom Prox, Phenom World Eindhoven, Netherlands). The iron ore sample utilised in the experimental investigation came from Nigeria's Kogi State (Itakpe). The material was ground into a fine powder and then sieved to create fractions of 0.1 mm in size.

2.1 Iron Ore Sample Characterization

The sample composition was determined using standard procedures. To determine the type of iron ore, XRD was used. The functional groups and surface morphologies of the samples were ascertained using FTIR and SEM. The total iron content of the ore was measured using an atomic absorption spectrophotometer and the redox titrimetric method. For subsequent measurements of iron in aqueous solutions, the redox titrimetric method was employed since the two methods did not differ by more than 5%.

2.2 Determining the Interactive Impact of Process Variables on the Leaching Process

Response surface methodology was used to optimize the leaching process. It was facilitated by design expert software as used by Omotioma and Onukwuli (2017).

3 Results and Discussion

3.1 XRD result of the iron ore sample

Figures 1 and 2 show the qualitative analyses of the untreated and treated Itakpe iron ores (as determined by XRD). The predominant components of Itakpe iron ore are quartz and hematite. Treatment with acid altered the ores' compositions. The peaks were seen at lower 2θ values, which suggest that the sample's interplanar distance was higher (Kazim et al. 2011). The diffraction patterns also exhibit small peaks while no evidence for the exfoliation was found. The observed characteristics showed the ore is rich in Fe with traces of impurities.

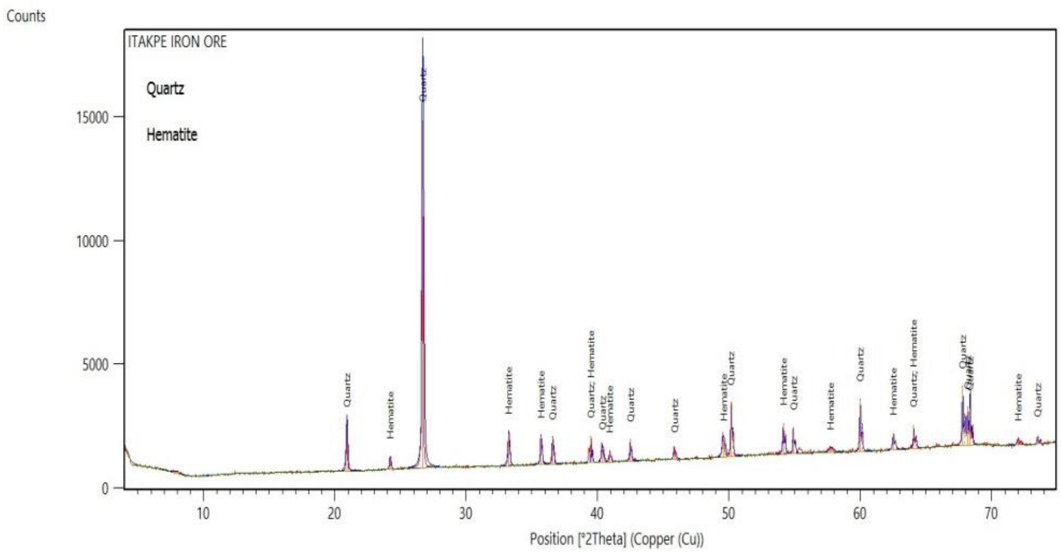

Figure 1: Mineralogical Compulsion of Itakpe Iron Ore

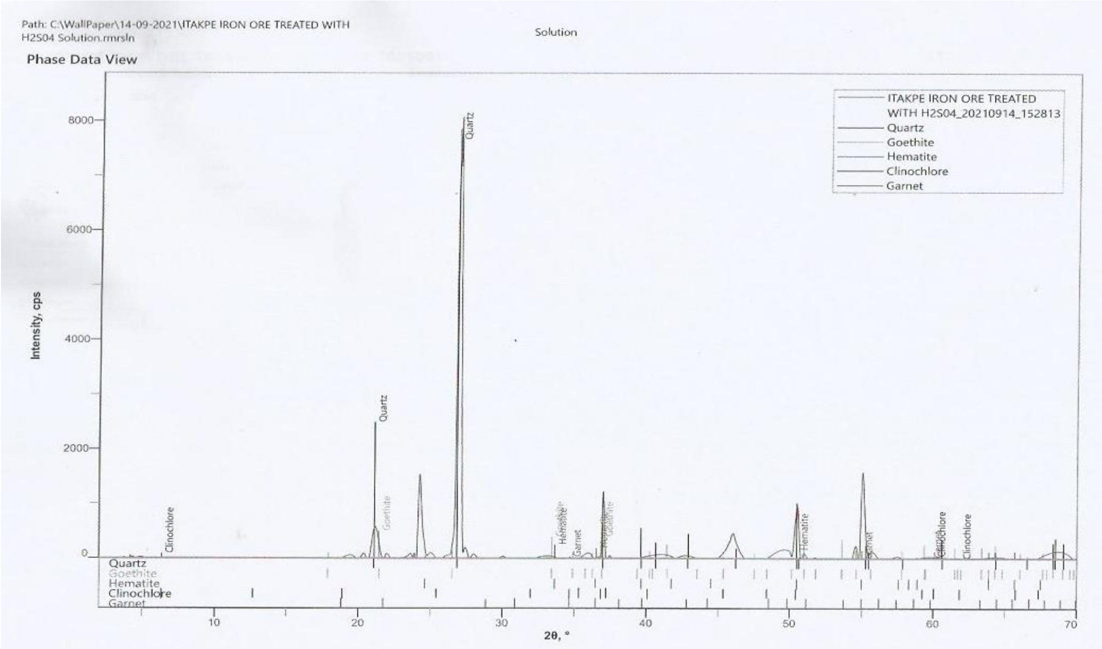

Figure 2: Mineralogical Compulsion of Itakpe Iron Ore treated with H_2SO_4

3.2 Functional groups of the samples.

The FTIR absorption peaks of Itakpe iron ore and the Itakpe iron ores treated with H_2SO_4 are shown in Tables 1 and 2 respectively. From the Table I, it is shown that the absorption bands for Itakpe iron ore occurred at 3697.5, 3574.5, 3656.5, 3362.1, 3399.3, 2557.0, 2117.1, 797.7, 1080.9 and 693.3cm^{-1} due to N-H stretch, O-H stretch, O-H stretch, C≡C stretch, C-H bend, C-O stretch and ≡C-H bend (for Itakpe iron ore); N-H stretch, O-H stretch, C=O symmetric and C=O stretch (Itakpe in H_2SO_4). The Itakpe ore and Itakpe iron ore treated with H_2SO_4 show the presence of heteroatoms with free electron pairs and substituent group (Omotioma and Onukwuli, 2015).

Table 1: Functional groups of Itakpe Iron Ore

Peak	Functional Group
3697.5,3574.5, 3656.5,	N-H stretch
3362.1, 3399.3	O-H stretch
2557.0	O-H stretch
2117.1	C≡C stretch
797.7	C-H bend
1080.9	C-O stretch
693.3	≡C-H bend

Table 2 Functional groups of the Itakpe Fe ore treated with H_2SO_4

Peak	Functional Group
3693.8, 3652.8, 3552.2	N-H stretch
3399.3	O-H stretch
1774.2	C=O symmetric
1654.9	C=O stretch
1524.5	N=H bend
752.9	C=H bend
1028.7	=C-O-C sym.

3.3 Surface morphologies of the Itakpe Iron Ore

Microstructures of the Itakpe iron ore and treated Itakpe ores with H_2SO_4 medium as observed by SEM are presented in Figure 3 and 4 respectively. The coarse and lumped structure of Itakpe iron ores are clearly observed in Figure 3. However, in the H_2SO_4 – treated Itakpe ore structural changes with globular structure. (Onyedika et al, 2013; Alafara et al, 2017; Alafara et al, 2018; Almeida and Schneider, 2020). It revealed the interaction between the positive and negative charged particles on the surface of the samples. Moreover, some part of it stays free in the reaction mixture.

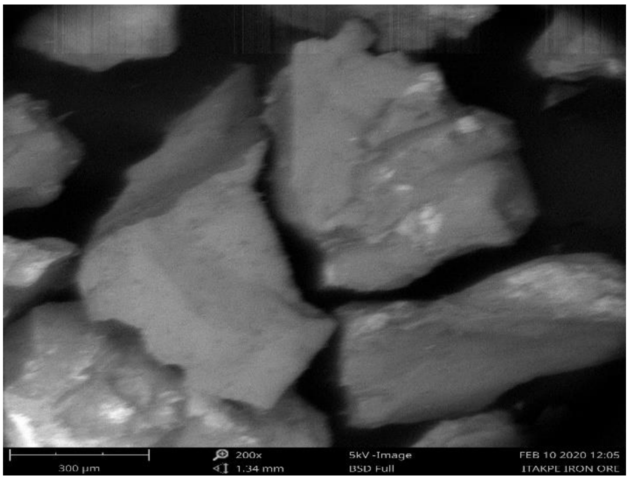

Figure 3: SEM analysis of Itakpe Iron Ore

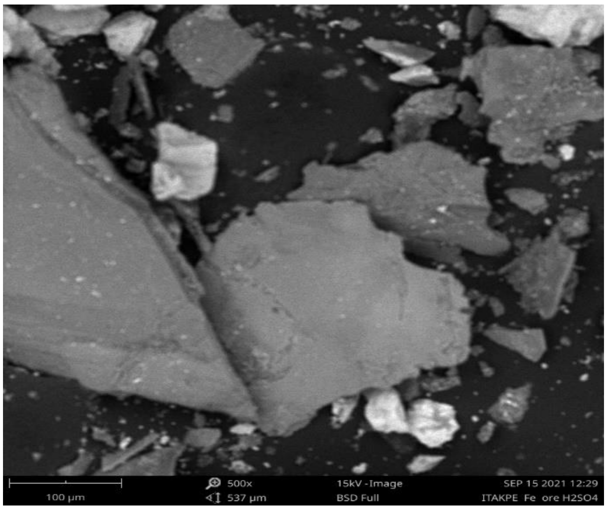

Figure 4: SEM analysis of Itakpe Fe Iron Ore H_2SO_4

3.4 Response surface methodology (RSM) results

In Table 3, maximum values of Fe leached was recorded as 92.05%, The high value of Fe leached showed that H_2SO_4 is suitable for leaching the ores. The variations of percentage of the Fe leached as a function of concentration, mass/volume ratio, temperature and time were further analyzed using graphs and mathematical model. It was determined by response surface methodology (RSM). The mathematical model generated showed leaching ability as a function of concentration, mass/volume ratio, temperature and time. The interactions among the factors were revealed in line the report of Onukwuli et al (2017).

Table 3 RSM results of Itakpe iron ore leached with H_2SO_4

Std	Run	F1 A: Acid conc. M	F2 B: Solid/liquid ratio g/ml	F3 C: Temp °C	F4 D: Time min.	R1 %tage of Fe leached %	R2 %tage of P leached %	R3 %tage of S leached %
4	1	9	0.25	30	30	82.78	21.06	15.80
21	2	5	0.15	30	50	88.44	30.12	21.45
28	3	5	0.15	50	50	92.05	34.04	25.00
10	4	9	0.05	30	70	80.51	17.40	13.51
23	5	5	0.15	50	30	85.65	25.66	18.66
13	6	1	0.05	70	70	76.10	10.25	9.11
17	7	1	0.15	50	50	81.17	18.40	14.16
25	8	5	0.15	50	50	92.05	34.04	25.00
9	9	1	0.05	30	70	76.73	11.29	9.75
2	10	9	0.05	30	30	78.16	13.58	11.17
1	11	1	0.05	30	30	75.28	8.93	8.28
16	12	9	0.25	70	70	92.05	34.04	25.00
27	13	5	0.15	50	50	90.81	33.90	23.81

14	14	9	0.05	70	70	82.55	20.65	15.56
26	15	5	0.15	50	50	92.05	34.04	25.00
19	16	5	0.05	50	50	82.61	20.72	15.61
20	17	5	0.25	50	50	89.72	32.17	22.72
6	18	9	0.05	70	30	79.81	16.21	12.80
11	19	1	0.25	30	70	77.75	13.00	10.75
22	20	5	0.15	70	50	90.18	32.88	23.16
5	21	1	0.05	70	30	75.35	9.07	8.34
12	22	9	0.25	30	70	88.47	30.18	21.49
3	23	1	0.25	30	30	76.38	10.68	9.37
29	24	5	0.15	50	50	92.05	34.04	25.00
15	25	1	0.25	70	70	79.87	16.31	12.86
30	26	5	0.15	50	50	92.05	34.02	25.00
8	27	9	0.25	70	30	84.23	23.35	17.24
7	28	1	0.25	70	30	76.9	11.56	9.91
18	29	9	0.15	50	50	89.54	31.90	22.55
24	30	5	0.15	50	70	90.24	32.67	23.26

3.5 Fit Summary

Table 4 displays the model fit summary of the Fe leaching %. Quadratic, cubic, two factor indicators (2FI) and linear models were tested. Sequential p-value (< 0.0001), adjusted, and anticipated R^2 values were used to select the quadratic model. The corrected R^2 and the expected R^2 accord rather well. The observation aligns with Omotioma and Onukwuli's (2015) report

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Table 4: Fit summary for %tage of Fe leached from H_2SO_4 treated Itakpe Iron Ore

Source	Sequential p-value	Lack of Fit p-value	Adjusted R^2	Predicted R^2	
Linear	0.0350	< 0.0001	0.2215	0.0550	
2FI	0.9745	< 0.0001	0.0349	-1.2878	
Quadratic	< 0.0001	0.0127	0.9578	0.9131	Suggested
Cubic	0.7761	0.0022	0.9453	-0.4370	Aliased

3.6 Mathematical models of the leaching efficiency

The leaching efficiency's mathematical model is shown in Equation 1. The model is quadratic since the greatest power of the variables is two. The equation defined in terms of coded factors can be used to predict the response for particular levels of each factor. A synergistic effect is indicated by a positive value, while an antagonistic effect is indicated by a negative value (Omotioma and Onukwuli, 2015; Ezeugo et al, 2018; Anadebe et al, 2018). The relative impact of the elements can be ascertained using the coded equation by comparing the factor coefficients.

$$\%\text{tage of iron leached} = +90.87 +3.48A +2.28B +0.6967C +1.65D +1.19AB +0.4150AC +0.7538AD +0.2838BC +0.6600BD +0.2138CD -4.54A^2 -3.73B^2 -0.5860C^2 -1.95D^2 \quad (1)$$

3.7 RSM results

Table 5 presents the RSM result. In addition to the corresponding projected (optimal) and experimental percentage of Fe leached, it includes the ideal circumstances for acid concentration, mass/volume ratio, temperature, and time. At an acid concentration of 5M, a mass/volume ratio of 0.15g/ml, a temperature of 50°C, and duration of 50 minutes, the ideal percentage of Fe leached from Itakpe ore treated with H_2SO_4 medium was 90.87% (Itakpe ore). The percentage deviation statistical technique was used to compare the experimental and projected results. The expected and experimental findings were nearly identical. The recorded percentage deviation is below the 5% threshold. This suggests that the link between the percentage of Fe leached and the variables of temperature, duration, mass/volume ratio, and acid concentration was sufficiently captured by the model that was generated. It demonstrates that RSM was suitable for leaching process optimization.

Table 5: Validation of Results

Process Parameter	Value
Acid Conc	5M
Mass/volume ratio	0.15 g/ml
Temperature	50 °C
Time	50 min
Experiment %tage Fe leach	92.05%
Optimum %tage Fe leach	90.87%
%tage Deviation	1.28%

4.0 Conclusion

It was found that quartz and hematite make up the majority of the Itakpe iron ore. The treatment of the ores with acid altered their compositions. Iron ore treated with H_2SO_4 revealed a slight increase in d-spacing. SEM studies revealed a faint globular structure in the iron ore. Surface morphology differences were noted between the treated and untreated iron ores.

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