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CHEMICAL, MINERALOGICAL AND PRE-CONCENTRATION ANALYSIS OF AZEKU IRON ORE, BAUCHI STATE, NIGERIA

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ABSTRACT

This research work was carried out to characterize chemical, mineralogical, and Pre-concentration of Azeku Iron Ore. The head sample was subjected to analysis using X-ray Fluorescence (XRF), X-ray Diffraction (XRD), Scanning Electron Microscopy (SEM) and Optical Microscopy (OPM). The Iron ore was then subjected to Scrubbing pre-concentration test for five minutes at a spindle speed of 750 rpm and was later analyzed to see the effect of the scrubbing. Chemical analysis of the representative sample reveals that the ore contains the following elemental compositions using XRF analysis: 55.44% Fe, 2.81% Al, 2.0% Si, 0.99% P, 0.024% S, 0.323% Ca, 0.199% Ti, 0.075% V, 0.058% Cr and 0.043% Mn. The mineralogical analysis results of XRD, SEM/EDS and OPM show the presence of goethite (80%) and cronstedtite (20%) that are separated by smooth grain boundaries and are not locked with other minerals hence can be liberated from other associated minerals. From the SEM image the average particle size and surface area of the mineral were found to be 27.44 µm and 44.63 µm^2 respectively using image J software. Pre-concentration test by scrubbing was carried out on the liberation size in order to enrich the ore. The XRF result of the scrubbed ore indicates 60.05% Fe (85.85% Fe_2O_3), 0.54% Al,1.12% Si,0.21% P,0.018% S from unscrubbed liberation size ore of 59.37% Fe,3.22% Al,2.56% Si,0.85% P and 0.026% S, composition. The XRD mineralogical analysis confirmed that the silica bearing mineral has been scrubbed, thereby leaving goethite mineral in the scrubbed concentrate. Based on the results obtained, it can be seen that Azeku Iron Ore could be another potential source of mineral for the Iron and Steel industry in Nigeria.

Key words: *Characterization, Scrubbing, Cronstedtite, Goethite, Pre-Concentration.*

1.0 INTRODUCTION

The economical and political consequences of perpetual dependence on imports of iron and steels raw materials and finished products by any nation are numerous and diverse. Economically, it leads to a drain of the nation's foreign reserves, to the advantage of the supplying nation (s). Politically, it affects the manufacturing, supply, construction and erection of portions or entire components of strategic military hardware, communication complexes. Oil rigs, pipelines, refineries, airports, seaports or harbours, and even offices and storage infrastructures leaving the host nation completely exposed to the terms of security sovereignty [2]. In the developed nations and even some developing ones, growth and development indices seem to be tied to the per capital consumption of iron and steel products, hence their continued dominance of the production of machinery and equipment to run the major industries of the word. Steel consumption increases when economic activities are growing, as government invest in infrastructure, transport, and build new factories and houses [3]. Table 1.1 presents the top ten steel producing countries.

In order to exploit any mineral deposit, it is necessary to provide comprehensive data on all mineral present and their respective proportion in the areas as well as waste and concentrate product, in addition to the spatial distributions of those minerals on the scale of the deposit [5]. Chemical composition of some Nigerian iron ores is presented in Table 1.2.

Table 1.1: Top Ten Steel Producing Countries

Rank	1	2	3	4	5	6	7	8	9	10
Country	China	India	Japan	USA	South Korea	Russia	Germany	Turkey	Brazil	

Iran

Source: [10]

Table 1.2: Chemical Compositions of Some Nigerian Iron Ore Deposits

Deposit	K_2O	CaO	TiO_2	MnO	Fe	MgO	Al_2O_3	SiO_2	P_2O_5	S
Itakpe	0.42	0.30	0.10	0.05	36.88	0.20	1.00	48.0	0.18	0.05
Ochokochoko	0.53	0.21	0.61	0.08	34.45	0.18	9.67	51.07	0.02	0.007
Ajanbanoko	0.26	0.15	Trace	0.01	37.22	0.15	3.39	46.50	0.10	0.003
Agbaja 0.04	0.72	0.37	0.14	47.80	0.38	9.60	10.89	2.08	0.12	
Koton–Karfe	0.02	0.45	0.24	0.56	48.13	0.07	6.70	5.13	2.14	0.04
Bassa-Nge	0.02	0.17	0.26	0.31	46.90	0.47	10.87	8.28	1.45	0.05

Source: [2,7,9].

1.1 Review of some literatures on characterization and pre concentration

Asuke, F. *et al*., [6] worked on chemical and mineralogical characteristic of Gidan Jaja Iron Ore in Zamfara state and they reported the chemical composition of 73.79% Fe_2O_3, 0.52% MnO, 17.50% TiO_2, 0.11% CaO, 0.50 % Cr_2O_3, 3.84% SiO_2, 0.43% Al_2O_3, 0.034% CuO, 0.02% NiO, 0.46% PbO and 2.76% LOI. Phosphorus and Sulphur were below limit of detection with mineralogical phases of magnetite, Ilmenite and spinel which are separated by smooth grain boundaries.

Thomas, D. *et al* [8] investigated the chemical, mineralogical and petrological analysis of Gyaza Iron Ore in Katsina State using XRF, XRD, SEM and OPM. The XRF result revealed that the ore contains 36.17%Fe, 0.17%P, 1.10%Al, and 17.21%Si. The XRD result revealed the phases of quartz, hematite, goethite and other minor complex mineral phases. The SEM and petrological analysis results revealed that the ore matrix is an assemblage of inter-layered different mineral crystals with different shapes, sizes and angles of orientations separated by grain boundaries.

Chabia, R .*et al* [4] worked on preliminary study on enrichment of Anini iron ore for use by Algerian metallurgical industries and reported that a chemical composition of 55% Fe_2O_3 , 22.6%SiO_2, 12% Al_2O_3. The Mineralogical analysis reveals the phases of hematite, quartz and clay. Thereafter the ore was enriched by pre-concentration using washing. The result of chemical composition of washed ore shows that the Fe_2O_3 was enriched to 62% where as the SiO_2 and Al_2O_3 decreases to 2.3% and 3% respectively. The XRD result of the washed ore also reveals the presence of hematite phase only, and they concluded that the washed-out ore conforms to standards used in metallurgy

Ali, M. *et al* [1] determined the mineralogical characterization of limonite iron ore from the Roina Mines in Algeria. This study involved the use of XRD and SEM to determine the mineralogical characteristics, on the other hand studying the effect of washing on its quality. The XRD result revealed the presence of quartz, goethite, hematite, Illite and calcite. The polished thin section result was also reported to contain quartz, goethite and hematite. The SEM result was also in line with that of XRD which shows the presence of quartz, goethite, and hematite with trace of calcite. Pre-concentration by washing was reported to be effective in reducing the clay content and other associate minerals (Calcite and silica) from the raw material as shown in the chemical analysis. The Fe content increased from 41.18%Fe to 51.03%Fe before and after washing respectively. Similarly, the alumina content decreased from 7.87% to 1.45% which confirmed the pre-concentration technique.

2.0 MATERIALS AND METHODS

2.1 Materials and Equipment

The materials used include; Azeku Iron Ore sample and distilled water. The equipment used include: X-ray fluorescence Spectrometer (XRF), X-ray Diffractometer (XRD), Scanning electron microscopy (SEM), Petrological microscope, Laboratory kiln, and pH metre.

2.2 Material collection and preparation

Representative samples of the ore were obtained from various points of the deposit at Azeku village of Dambam Local Government Area of Bauchi State. GPS (Global Positioning System) was used to measure the exact location at which samples were taken (N 11˚44ˈ28.64ˈ; E 10˚ 54ˈ55.4ˈˈ) . Grab method of sampling was adopted in collection of the samples.50kg of the samples were collected from (4) four different pits at an interval of 50 meters and 3 meters deep so as to ensure a wide coverage of sampling of the ore.

2.3 Laboratory Sample Preparation

Here, samples collected were comminuted, i.e. gradual reduction in size of an ore from crushing and grinding processes. The lump size ore sample was reduced to crusher acceptable size using sledge hammer. The sample was further crushed using jaw crusher and then pulverized using ball milling machine.

2.4 Chemical Analysis of the Ore Sample using XRF

Chemical analysis was carried out on the representative sample as to establish the chemical composition of the ore there by finding the percentage of each element present in the ore. This analysis was carried out on the ore head sample and on the scrubbed concentrate using XRF machine.

2.5 Mineralogical and Petrological Analyses

These tests were carried out using the X-ray diffraction (XRD), Scanning electron microscope (SEM) and Petrological microscope. The essence of these tests were to identify the different phases presence in the ore, establishing the major, minor and trace minerals, the degree of association of valuable and gangue minerals and to determine the grain size of the various minerals in the ore.

2.6 Pre-concentration of Azeku Iron ore (Scrubbing)

Fifty grams (50g) of the ore was weighed using digital weighing balance, it was then transferred into a beaker containing distilled water. The ore solution was subjected to soaking for 5 minutes. The pH was measured using a pH meter and then the solution was scrubbed at a spindle speed of 750rpm for 5 minutes, the pH was equally measured after the scrubbing, and finally the mixture was decanted to get the scrubbed solute, which is the product from the ore solution. The scrubbed product was dried in a laboratory kiln before assaying so as to check the effect of the scrubbing on the product.

3.0 RESULTS AND DISCUSSION

3.1 Chemical analysis result of the iron ore sample

Table 3.1 Presents the chemical composition of Azeku iron ore in oxide and elemental state using XRF.

Table 3.1 is the result of the chemical analysis of Azeku iron ore, it can be seen that the iron ore contains 55.44% Fe, 2.82% Al and 1.99% Si as the major constituent elements, 0.987% P and 0.043% Mn are the minor constituents and 0.024% S and other elements are present in trace amount. However, it could be observed that the percent contents of 55.44% Fe, 2.82% Al and 1.99% Si obtained for Azeku iron ore compare favourably with some major Nigerian iron ore deposits.

Figure 3.1 and Table 3.2 present the result of XRD analysis of the iron ore sample, it can be obviously seen that the major pattern lines at higher intensity are region where goethite exist , followed by the pattern lines where goethite are locked with cronstedtite at low intensities and only two pattern lines revelled cronstedtite at lower intensities. In general, the pie chart generally illustrates 80% goethite and 20% Cronstedtite. The XRD result confirmed the XRF result.

3.3 Scanning Electron Microscopy (SEM) of Azeku Iron Ore

Plate 3.1 And Figure 3.2 Shows the SEM micrograph of Azeku Iron Ore at X500 and the EDS pattern respectively.

Table 3.1: Chemical analysis result of Azeku ore from Cu-Zn method

Element	Al	Ca	Si	P	S	Fe	Ti	V	Cr	Mn
% Composition	2.82	0.33	1.99	0.987	0.024	55.44	0.2	0.075	0.058	0.043

3.2 XRD Analysis of Azeku Iron Ore Sample

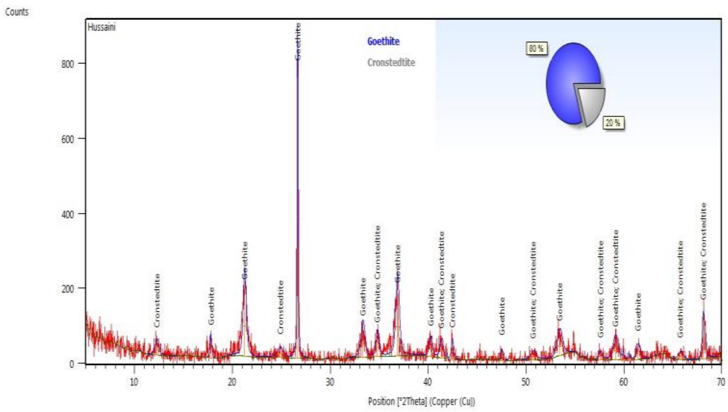

Figure 3.1: XRD pattern of Azeku Iron Ore

Table 3.2: XRD result of Azeku iron ore

Mineral name	Chemical formula	Crystal structure	% Composition
Goethite	$FeO(OH)$	Orthorhombic	80%
Cronstedtite	$FeSi(OH)$	Hexagonal	20%

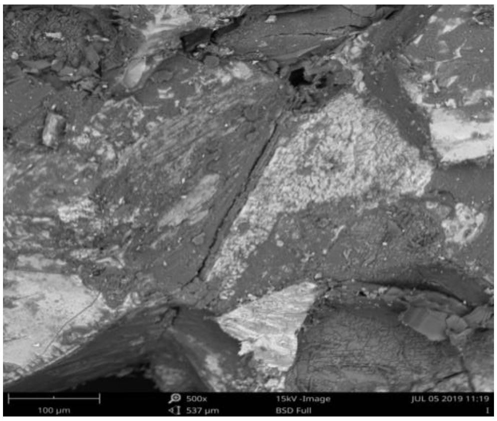

Plate 3.1: SEM Micrograph of ore sample (X500)

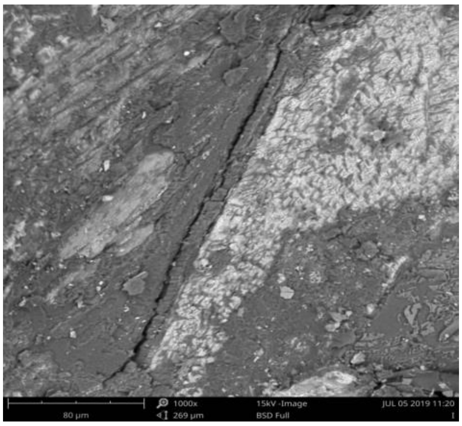

Plate 3.2: SEM Micrograph of ore sample (X1000) .

Plate 3.1 and plate 3.2 show the SEM results of Azeku iron ore at two different magnifications, it can be seen that the minerals present in the iron ore matrix are separated by grain boundaries, no interlocking of minerals and the mineral particles vary in sizes. Using Image J-software to analysis the iron ore micrograph it was found that iron ore minerals have an average particle size of 27.44 µm and surface area of 44.63 um^2. This indicates that the minerals can be freed from each other during comminution, possibly at medium coarse sieve size range with low requirement of energy for the liberation of the mineral of interest.

3.4 Petrological Analysis of Azeku Iron Ore

Plates 3.2 and 3.3 are the photomicrographs of Azeku iron ore observed under plane and cross polarized light respectively.

The Plates 3.2 and 3.3 revealed two minerals phases which are recognized both under plane and crossed polarized light examination. The phases are goethite (dark) and Cronstedtite (reddish brown) and void space that look whitish but not a mineral phase. The petrological result of the iron ore obtained correlate very well with that of its XRD result where similar phases were found to be contained in the iron ore matrix.

3.5 Pre-Concentration of Azeku Iron Ore using Scrubbing Method

The pre-concentration of the Azeku iron ore was carried out to ascertain the susceptibility

of the iron ore to concentration process. Table 3.3 presents the chemical analysis result of the pre-concentrated scrubbed iron ore sample.

From result in Table 3.3 it can be observed that the 59.39%Fe was upgraded to 60.05%Fe (1.1% increased) after scrubbing. While Aluminium content in Alumina was decreased from 2.81 to 0.539% and the silicon content from silica of 2.56% Si was reduced to 1.12% Si. The percent contents of the deleterious elements were also found to reduce drastically after scrubbing with a corresponding decreased in the pH value of the water used from 8.3 to 7.6 after scrubbing. The decreased in the pH value of the water used could be attributed to partial solubility of the deleterious elements contained in the ore matrix.

XRD Result of the Scrubbed Iron Ore Sample

The scrubbed pre-concentrated product of the beneficiated iron ore was analyzed using XRD. Figure 3.3 shows mineral phases of the scrubbed pre-concentrated iron ore sample product.

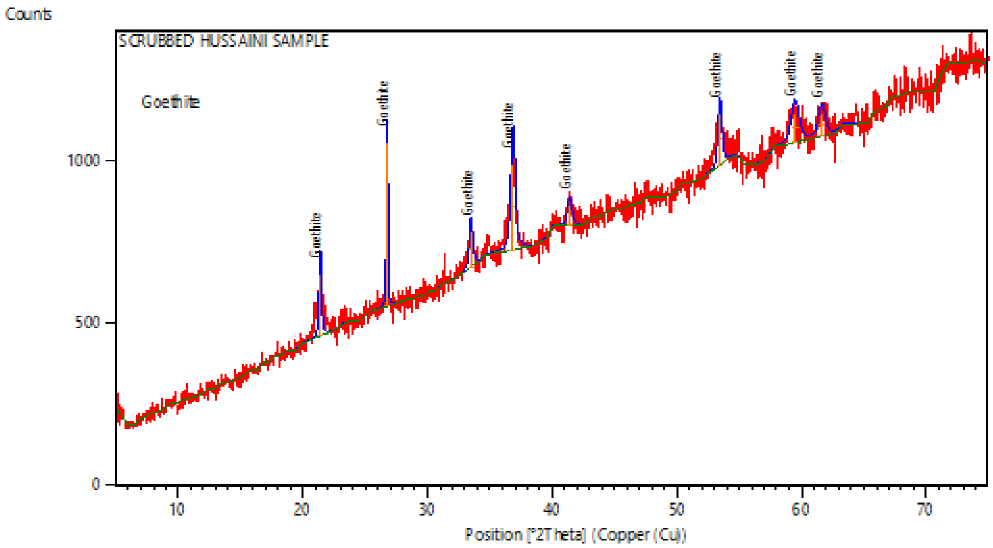

Figure 3.3: Spectrum of scrubbed Azeku iron ore sample by XRD

From the result in Figure 3.3 it can be observed that, only goethite phases are predominant in the matrix of the scrubbed product. Other mineral phases that were earlier contained in the iron ore matrix before scrubbing were partly scrubbed out.

Therefore, based on the results obtained the Azeku iron ore can be said to be susceptible to beneficiation technique.

Table 3.3: XRF result of the Pre-concentrated Iron Ore sample

Element	Al	Ca	Si	Fe	P	S	Ti	V	Cr	Mn
Concentrate %	0.539	0.03	1.124	60.05	0.206	0.018	0.081	0.039	0.034	0.282

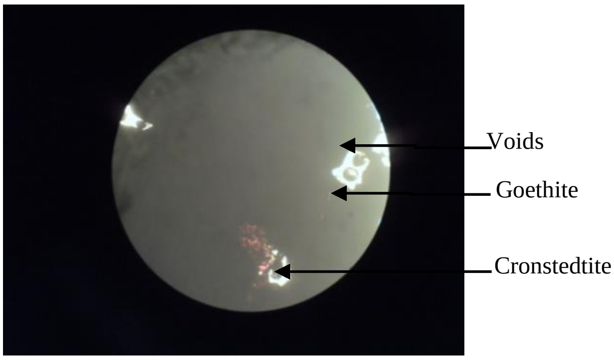

Plate 3.2: Photomicrographs from Petrological Analysis under Plane Polarized

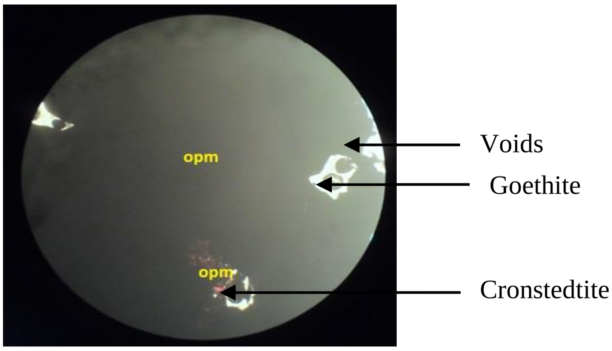

Plate 3.3: Photomicrographs from Petrological Analysis under crossed polarized

4.0 CONCLUSION

The characterization and Scrubbing of Azeku Iron Ore were carried out and the following conclusions drawn:

(i) Azeku Iron Ore contains 55.44% Fe, 2.81% Al and 1.99% Si, 0.98% P, 0.12% K, 0.11% Ti, 0.23% Ca and 0.42% Mn, 0.024% S, 0.078% Cl, 0.075% V, 0.058% Cr the trace elements.

(ii) The mineralogical analysis of the ore confirmed the prevailing mineral phase of goethite and cronstedtite.

(iii) The SEM analysis result indicates that the iron bearing minerals are separated from other associate minerals in the ore by smooth grain boundaries and non-interlocking by another mineral, which shows that the mineral can be easily separated from other associate minerals by comminution.

(iv) The deleterious elements were partly and effectively stripped from the surface of the minerals through scrubbing pre-concentration method. The iron ore was enriched by yielding 60.05% Fe, 1.12% Si, 0.018% S, 0.21% P, 0.54% Al from 59.34% Fe, 2.56% Si, 0.026% S, 0.85% P and 3.22% Al. Likewise, the XRD result shows only goethite, the mineral of interest as when compared to the XRD result of the iron ore head sample

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