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CHARACTERIZATION AND MINERALOGICAL ANALYSIS OF EZEAGU IRON ORE, ENUGU STATE, NIGERIA

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Abstract

An investigation was conducted on the characteristics and mineralogical analysis of Ezeagu iron ore. Ezeagu is situated in the Ezeagu Local Government Area of Enugu State with its administrative headquarters located in the town of Aguobu-Owa. The characterization and mineralogical analysis were conducted by utilizing some basic techniques for analysis such Scanning Electron Microscopy (SEM attached with Energy Dispersive X-Ray Spectroscopy (EDX), X-ray fluorescence (XRF), and an X-ray diffractometer (XRD) and Fourier Transformed Infrared Spectroscopy (FTIR). These analytical techniques were used to determine all the necessary parameter for this research work. The XRF, XRD, EDX, and FTIR analyses performed on the iron ore revealed that it is a high-grade, high-silica, and low-alumina ore with a predominantly hematite mineralogy. The ore's average composition consists of 30.59% SiO_2, 2.11% Al_2O_3, 53.75% Fe_2O_3, and minor amounts of other oxides. The XRD analysis showed the presence of other oxides such as T_iO_2, MnO, MgO, and Na_2O in the ore sample. The EDX analysis indicated that the ore is predominantly composed of iron, with smaller amounts of titanium, manganese, and silicon. FTIR analysis identified functional groups such as Fe-O, Fe-OH, and Fe-H bonds, which are typical for iron oxides and hydroxides.

Key words: *Characterization, chemical, mineralogical, FTIR, SEM/ EDX, Ezeagu iron ore*

1.0 Introduction

Operationalization of an iron and steel sector are essential for the technological and infrastructure growth of every contemporary society. Nigeria possesses iron ore, coal, natural gas, and limestone, which are all essential raw materials for the production of steel [1] . Nigeria has the potential to become the center of the West African subregion's economy, but the nation's economy cannot be robust and dynamic without the expansion of its iron and steel industry and the utilization of these materials in a variety of other industries, including operation of its industrial base through the production of spare parts components and other metallurgical items for strong economic revolution [2]. One of the metals that is used the most in all of the many sectors of the global economy is iron, along with its generic goods and services that it renders for industrial transformation.

This article discusses the various factors influencing the production of iron, including the mechanical properties and cost considerations. It also highlights the blast furnace (BF) and direct reduction (DR) processes used to produce pig iron and sponge iron, respectively. Moreover, it explains the importance of beneficiation processes to improve the iron content of ores before feeding them into BF or DR furnaces. [3].

This article provides a comprehensive overview of the ironmaking industry, discussing the various factors and technologies involved in the production of iron. It is a valuable resource for anyone interested in understanding the iron and steel industry [3].

Iron-containing minerals that are most commonly found are oxides, of which hematite (Fe_2O_3) is the primary form this is also referred to Iron (iii) oxide it is the main component of iron ore. It is one of the most abundant iron ores on earth and it is often used as a source of iron in the steelmaking process. Iron ore exists in different forms, with the most common being hematite (60-70% iron content), magnetite (Fe_3O_4) and goethite (FeO (OH)). All three forms of iron ore can be found in sedimentary, metamorphic, and igneous rocks worldwide. Hematite is generally red and has the highest iron content, magnetite has a black-grey color, and goethite is typically yellow-brown.

The Okene magnetite complex is known to contain significant iron ore reserves, including the Itakpe Hill deposit, which is believed to be the largest hematite-magnetite-quartz body in the district. This suggests that Nigeria's iron ore resources are located primarily in North Central Nigeria [4].

However, it is a widely known fact that Nigeria's iron ore reserves at Itakpe and Agbaja have been identified for potential open pit mining to provide raw materials for the Ajaokuta and Delta Steel Companies. In this article, the authors discuss the potential for growth in Nigeria's steel industry, including the need for improved domestic production of iron ore and other raw materials to support local steel production. [5]. A few Nigerian iron ores are listed in Table 1 [6] together with their chemical compositions. The Agbado Okudu iron ore deposit was studied by Agava [7], who discovered that the iron ore's average composition was 38.82% Fe, 49.10% SiO_2, 0.05%% P_2O_5, and 0.03% S. Furthermore, magnetite and hematite are the main minerals that contain iron, according to a mineralogical analysis of the ore. It was found that the ore's work index was 4.32 kWh/t. [7]. Thomas and Yaro [8] examined the effects of calcination on the beneficiation of iron ore from the Koton Karfe deposit in Nigeria, providing valuable insights into the processing and upgrading of the country's iron ore resources. The study found that calcination effectively reduced the level of phosphorus in the iron ore, improving its quality and suitability for use in ironmaking. The researchers concluded that calcination is a promising method for beneficiating Nigerian iron ores, particularly those with high phosphorus content, and could contribute to the development of the country's iron and steel industry. They also recommend further research to optimize the calcination process and evaluate its economic viability on a larger scale.

Table 1: Some Nigerian iron ore deposits and their chemical compositions (wt.%)

Deposits	K_2O	CaO	TiO_2	MnO	Fe	MgO	Al_2O_3	SiO2	P_2O_5	S
Ajabanako	0.26	0.21	Trace	0.01	37.22	0.15	3.39	44.88	0.18	Trace
Agbado – Okudu	0.04	0.72	0.37	0.14	47.80	0.38	9.60	10.89	2.08	0.12
Bassa - Nge	0.02	0.17	0.17	0.13	46.90	0.40	8.28	8.28	1.45	0.03
Ochokochoko	0.53	0.15	0.16	0.08	34.45	0.18	9.67	51.07	0.02	Trace
KotonKarfe	0.02	0.45	0.25	0.56	48.18	0.07	-	5.13	2.14	0.04
Itakpe	0.42	0.30	0.10	0.05	36.88	0.20	-	44.88	0.18	Trace

Source: [4]

The researchers came to the conclusion that it contained 43.34% iron and only 10.14% silica. They concluded from their research that the ore's primary ingredients are magnetite, siderite, and goethite, with hematite serving as a minor mineral. The work index for the Koton karfe iron deposit was determined to be 11.33kWh/tonne for calcined samples and 17.00kWh/ton for uncalcined samples [9]. Asuke [9] describes the Koton Karfe iron ore has the ore that contain siderite, quartz, and hematite are merely traces of magnetite and goethite, as the ore's basic elements. It was also mentioned that the phosphorus content of the ore was uniformly distributed throughout the mineral matrix and can be lowered by employing an acidic leaching technique [9].

Working on the Gujeni iron ore deposit's characterization, Salawu [10] discovered that, on average, the ore contained 48.6% Fe, 0.2% Mn, 12.01% Ti, 2.06% P, 0.2% S, 6.0% Si, and 4.4% Al. The results of the mineralogical investigation indicated that the ore was mostly composed of rutile, goethite, and hematite, with smaller amounts of silicate, manganese oxide, zincite, and zirconium.

It was discovered that the ore had a work index of 13.96 kwh/t. Agava et al. [11] investigated the chemical, mineralogical, and liberation size of Ochokochoko iron ore. The ore was reported to contain 50.60% Fe, 17.70% SiO_2, 0.05% P_2O_5, 0.03% S, and to be largely constituted of magnetite, hematite, calcite, alumina, and silica. Furthermore, the study reported that the ore can be classified as medium grade and released at a sieve size of -180+125µm. Suleiman et al. [4], the XRF analysis of the Obajana iron ore in Kogi State, Nigeria. The result shows that the ore contains 47.82% Fe_2O_3 and 24.1% SiO2 as major constituents, with 11.43%K_2O, 0.24%MgO, 4.61TiO_2, 2.744P_2O_5, 0.23%PbO and 1.45%Al_2O_3 as minor constituents [4].

The X-ray diffraction (XRD) result showed that the main mineral phases found in the ore are biotite (K(Mg,Fe) 3AlSi_3O10$(OH)_2$), hematite (Fe_2O_3), and quartz (SiO_2).Agava et al. [11] determined the Ochokochoko iron ore's chemical, mineralogical, and liberation size. They said the ore was freed at a sieve size of -180+125 µm and might be classified as medium grade.

The Jaruwa Iron deposit was studied by Ahmed et al. [12] discovered that the ore is a rich hematite ore intercalated with kaolinitic clays coated in lateritic overburden and hosted mostly by amphibolites and meta-sediments. The principal objective of this study is to present detailed information about the characterization, chemical and mineralogical properties of the iron ore deposit at Ezeagu, with a view to determining the composition, the miller indices ,morphological structure, the functional groups The detailed chemical analysis of the high-grade iron ore provides valuable information for designing an effective mineralogical and beneficiation process that can efficiently extract the valuable iron content from the ore, enabling the development of a local iron and steel production industry in Nigeria.

1.1 Ezeagu iron ore Deposit

The Ezeagu iron ore deposit is located in Ezeagu which is a Local Government Area in Enugu State with the administrative Headquarters in the town of Aguobu- Owa.Its geographical coordinates are 6^o 23’ 0” North, 7^o 17’ 0“East and its original name (with diacritics). It has a total area of 633m^2(244sq ml) and the elevation of 350m (1,150ft) and a total population of 169,718 according to 2006 population census conducted. It shares borders with Udi, Uzouwani, Oji River, and Awka North LGAs. The LGA has a number of rivers flowing through its territory and these include the Duu, Kalawa and Nnam rivers. The average temperature of Ezeagu LGA is 28 degrees centigrade while the average humidity level of the area is 71%. The estimated reserve tonnage of mineral deposit as regards the availability of the ores found in this area has not been quantified. [13].

2.0 Materials and Methods

2.1 Materials and Equipment

Ezeagu iron ore samples, laboratory jaw crusher, sledge hammer, ball mill, XRD, XRF, SEM /EDX, FTIR analytical instruments and hardness testing machine model PRLH210 were among the materials and equipment used in the study.

2.2 Sample Collection

Iron ore samples were collected from six pits within the deposit. Grab sampling technique was utilized to collect the samples. 20 kg of sample material were extracted at a depth of 4m and 250m apart. This was carried out with a view to get samples that are the representative of the ore deposit.

2.3 Sample Preparation

The lump sizes of the ore samples were achieved by crushing and Ball Milling Machine. The four samples collected from different pits were crushed together and homogenized, this was performed to create a true representative sample for analysis, the coning and quartering method of sampling was employed.

2.4 Chemical Compositional Analysis

A representative sample was taken and analyzed using X-ray fluorescence (XRF) equipment fitted with a Model Phillips-JEE 4B. This analysis was carried out at the Soil Chemical Laboratory Sc 01, ROLAB Research and Diagnostic Laboratory, Challenge, Ibadan off the Lagos-Ibadan bye-pass, Oyo State.

2.5 Mineralogical Analysis of the Ore

The mineralogy of the ore was determined by using the Scanning Electron Microscopy (SEM) attached with Energy Dispersive X-ray (EDX), and X-ray diffraction (XRD). The Fourier Transformed Infrared Spectroscopy (FTIR).

2.6 Hardness Test

The sample was subjected to hardness test with the use of a Hardness Testing machine with model PRLH210.

3.0 Results and Discussion

3.1 Chemical Analysis of Ezeagu Iron Ore

The XRF chemical analysis of the iron ore from Ezeagu is presented in Table 2 with the oxide compositions and concentration weight percentage values. The constituents shown in the table were classified as major, minor and trace.

The result shows that the ore contains 59.79% Fe_2O_3 and 30.59 % SiO_2 as major constituents, with 0.04%K_2O, 0.70%MgO, 0.15TiO_2, 0.25 P_2O_5, 0.10CaO and 2.11%Al_2O_3 as minor constituents. These results obtained were in agreement with what was obtained be Suleiman et al., [4]

Table 2: Chemical composition of Ezeagu iron ore.

Oxides	%
SiO_2	30.59
Al_2O_3	2.11
Fe_2O_3	59.79
TiO_2	0.15
CaO	0.10
P_2O_5	0.25
K_2O	0.04
MnO	0.70
MgO	0.70
Na_2O	0.34
Cu	0.05
Zn	Nd
Rb	0.05
Zr	Nd
Br	0.09
Cr	0.10
LOI	5.44

The sulfur and phosphorus contents were below the detection limit. The grade of raw iron ore and its suitability for commercial use were primarily determined by its chemical composition [14]. For iron ores to be economically viable during exploitation, they should ideally include high levels of iron and low levels of impurity elements [15]. The XRF study results show that the iron ore deposits contain 30.59 % silica and 2.11 % alumina respectively. According to the findings as reported by Asuke et al. [9] the silica concentration was adequate, but the alumina content was significantly low and needs to be improved [16]. Additionally, a high Fe_2O_3 content of 59.59% was obtained from the XRF analysis, making it superior to the majority of iron ores from other regions of the nation. Asuke et al [17].

3.2 Mineralogical Analysis of Ezeagu Iron Ore

The X-ray diffraction (XRD) pattern of hematite (001) provides important information which determined the Miller indices of the planes with respect to the d-spacing and the peak width.[18].Figure 1 displays the typical findings of the iron ore deposit's where the XRD technique was used to examine the crystallography of the ore. Hematite (F_2O_3), magnetite (Fe_3O_4), and goethite (FeOOH) that occurred as the major oxides of the three main crystalline minerals in the ore sample. According to the XRF analysis there was an indication that the ore is a high-grade one. Asuke et al [9]

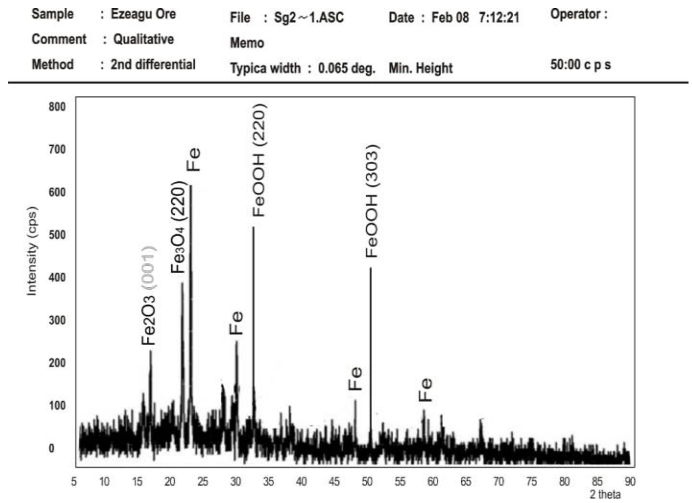

Figure 1: XRD pattern for Ezeagu iron ore

The XRD technique was further used to analyze the ore sample which determined other oxides, interplanar and the miller indices as indicated in Figure 2. The obtained results are as followed Fe_2O_3 (234), Al_2O_3 (112), CaC (111), Fe_2O_3 (112), CaC (101), $Ca_3Si_2O_7$ (221), MnO (101), CaO (220), Fe_2O_3(112), Al_2O_3(102), CaO (220) and Fe_2O_3(101).

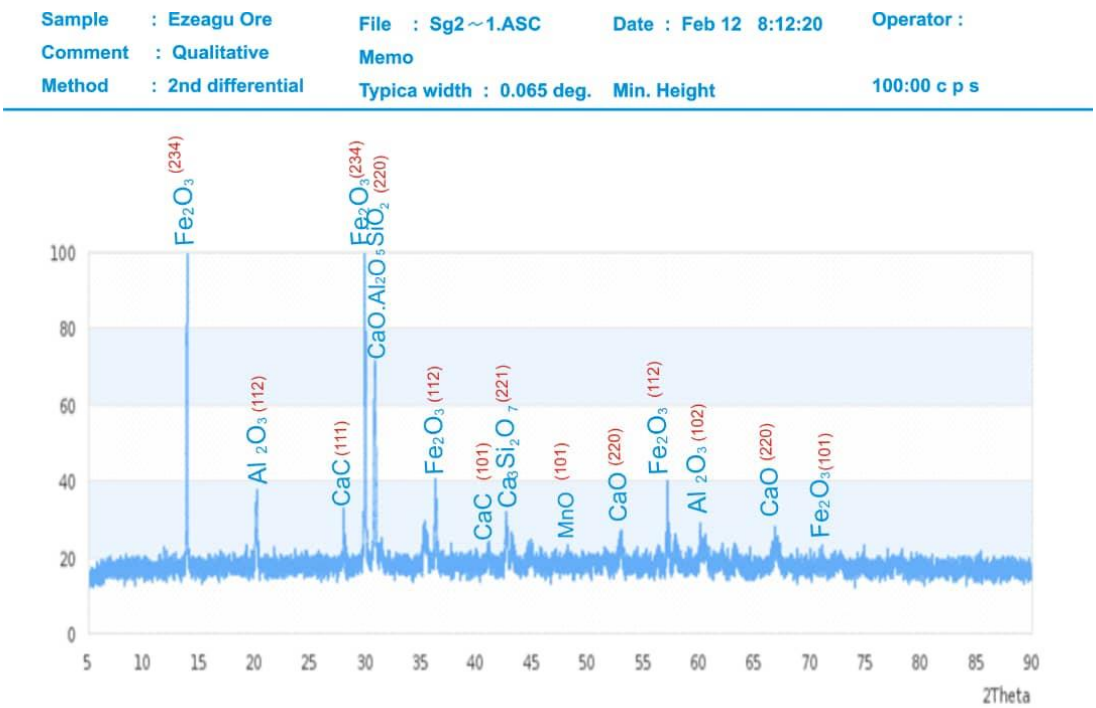

Figure 2: Elements and interplanar and Miller indices

The major diffraction peaks were identified at diffraction angles 2θ of 17.311, 23.387. 24.501 with corresponding interplannar distance of 4.313, 4.329 .4.330 A^o and relative diffraction intensity of 185, 220, 600 etc. The identified phases are displayed in table 3. The phases can be linked with the interaction between the Hematite (Fe_2O_3), Magnetite (Fe_3O_4), Goethite (FeOOH) and other elements.[16]. The findings validated the XRD study, which showed that hematite, magnetite, and goethite are the main minerals present along with minor oxides.

3.4 Scanning electron microscope

Figures 3a, 3b, and 3c show the SEM micrographs of the iron ore sample at three different magnifications of x7000, x 6000 and x5000. The findings showed that there is no mineral interlocking, that the sizes of the mineral particles vary, and that the minerals are separated by grain boundaries. These phenomena support previous findings by Oyeladun et al. [19] which indicated that the minerals can be separated from one another during comminution. The SEM results of ore sample shows the presence of hematite, magnetite, and goethite. Hematite has a crystal structure that consists of hexagonal layers of oxygen atoms, with iron atoms in octahedral coordination. The (001) plane of hematite was most prominent. Magnetite has a cubic crystal structure, and the (220) plane which was most prominent. Goethite has a crystal structure consists of layers of oxygen atoms, with iron atoms in octahedral and tetrahedral coordination. The goethite iron ore contained (220) and (303) planes these are prominent, the (220) plane a characteristic of “needle-like" morphology. Goethite is known to have a high degree of anisotropy, meaning that its properties vary depending on the orientation of the crystal. In terms of morphology, the (220) plane is often described as "porcupine-like", while the (303) plane is described as "acicular" or "needle-like" [20-21].

Figure 3a: SEM image at 50 microns and x 7000

Figure 3b: SEM image at 50 microns and x 6000

Figure 3c: SEM image at 50 microns and x 5000

3.5 The Energy Dispersive (X-Ray) Spectroscopy Ezeagu Iron ore

The result obtained revealed the compositions and phase distribution of the ore which include the formation of the different phases and their relationships to the ore' sample. The mineralogy and crystallography of the different phases and the mineral processing of the ore are affected by the properties of the ore. The EDX Spectrograph of Ezeagu Iron ore sample shown in figure 4 indicates the major concentration by weight %: Oxygen 25.2, Carbon 4.08, Fe 55.8, Ti 2.20 Mn 6.40. Si 4.20 and Al 2.12. The result obtained from the EDX agrees with the results obtained from the XRF which was 59.79 for Fe_2O_3.

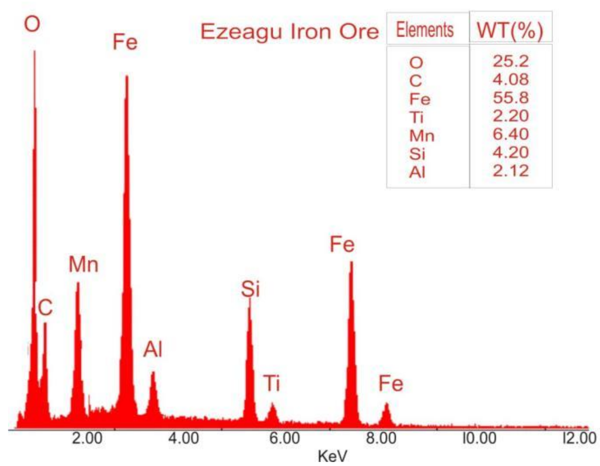

Figure 4: Major elements present in Ezeagu iron ore

3.6 Fourier Transform Infrared Spectroscopy (FTIR) of Ezeagu iron ore

The Fourier Transform Infrared Spectroscopy (FTIR) technique was used to determine the functional groups of the ore. The three phases detected by the technique was explained as they occurred. Figure 5: indicate the FTIR of the Ezeagu Iron ore indicating the Functional groups .

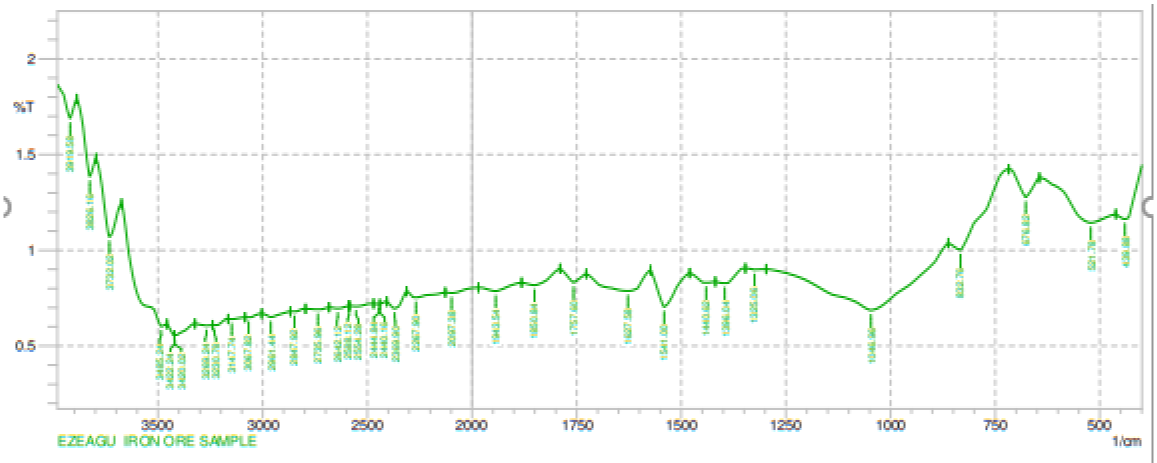

Figure 5: FTIR of the Ezeagu Iron ore indicating the functional groups

Hematite has several functional groups, including carboxylic acid groups, hydroxyl groups, and carbonyl groups. The carboxylic acid groups are present in the form of hydroxylated carboxylic acid, and they can be found in hematite nanoparticles. The hydroxyl groups can be found on the surface of the hematite nanoparticles, and they can be used to interact with other molecules. The carbonyl groups can be found in the form of carbonyl compounds. The main functional groups found in magnetite are hydroxyl, carboxyl, and carbonyl groups. The hydroxyl groups are present on the surface of the magnetite nanoparticles, and they can be used for adsorption and catalysis. The carboxyl groups are found in the form of carboxylic acids, and they can be used for metal binding and adsorption. The carbonyl groups are present as carbonyl compounds, and they can be used for sensing and biosensing. Goethite iron ore has several functional groups which include hydroxyl, carboxyl, carbonyl, and sulfate groups. The hydroxyl groups are found on the surface of the goethite nanoparticles, and they can be used for the adsorption of other molecules.

The FTIR technique was used to determine the functional group of the ore sample and the peaks number obtained was 34 spectra. The values of the three peak vales with the corresponding intensity are at 439.88cm^{-1}, 521.78cm^{-1} and 676.82 cm^{-1} with corresponding intensity values of 1.1634, 1.1439 and 1.2776 for the first three functional groups. These peaks correspond to the vibrations of the Fe-O bonds in the crystal structure of the ore. There are also some smaller peaks that was observed in the FTIR spectrum of the ore such as the peaks at3732.02cm^{-1}, 3826.16 $cm^{-1}$and 3919.58cm^{-1} with their corresponding intensity of 1.0706, 1.3852 and 1.6884 for the last three functional group. There was indication that the peak and the intensity values increase in respect of each other.

These peaks correspond to water and hydrox. The Fe-O, Fe-OH, and Fe-H bonds are the functional groups that was observed in the FTIR spectra of Ezeagu iron ore. The Fe-O bond has the strongest and most prominent peak, while the Fe-OH and Fe-H bonds are much weaker and more difficult to detect. In addition to these bonds, there were some minor peaks corresponding to other impurities or contaminants in the sample. These peaks were used to identify the presence of impurities which provide valuable information about the purity of the ore sample. The FTIR spectrum was used to identify the impurities by comparing the spectrum of the ore sample to the spectrum of a known impurity It is know that FTIR spectrum of the ore sample shows a peak at

1.046.96 cm^{-1}, this could be indicative of the presence of quartz impurities. This is because quartz had a characteristic peak at 1.46.96 cm^{-1}[22][23]. By comparing the FTIR spectrum of the ore sample to known impurities, it was possible to identify the presence of these impurities and which was subsequently used to determine concentrations in the sample.

3.7 **Hardness Value**

The sample was subjected to hardness test with the use of a Hardness Testing machine with model PRLH210.The hardness value was obtained at 6.0 Mohr's scale value. Through the hardness value of quartz (i.e7 on the Mohr's scale) is a bit higher than the iron ore (Hematite (5.5 -6.5). It is far more brittle than iron ore. The author provided a comprehensive overview of mineralogy, including the properties and characteristics of minerals such as iron oxides. The page numbers in the citation likely refer to sections in the book that discuss goethite and other minerals [24].

4. Conclusions

The following conclusions were drawn after the characterization, chemical analysis and mineralogical investigation of the Ezeagu iron ore.

i. The results obtained from the XRF analysis were classified as Major oxides: SiO_2 and Al_2O_3 are the major oxides in the iron ore, with 30.59% and 2.11% concentrations, respectively. These high values indicate that the sample is primarily composed of silicates and alumina, which are typical components of iron ore. Minor oxides: TiO_2, CaO, MnO, and MgO are present in minor amounts, with concentrations ranging from 0.10-0.70%. High-grade iron ore is typically defined as iron ore with an iron (Fe) content of 58% or higher. The sample in question contained 59.79% Fe, which meets this threshold and qualifies it as high-grade iron ore. The alumina content (2.11%) and silica content (30.59%) are both within acceptable limits for high-grade iron ore.

ii. The XRD analysis revealed that the ore contain oxide compositions of hematite, magnetite, and goethite. The characterization show that Hematite has a crystal structure that consists of hexagonal layers of oxygen atoms arranged in a hexagonal pattern. The results of the SEM analysis demonstrated that consistent grain boundaries separate the iron-bearing minerals in the ore from other minerals.

iii. The SEM/ EDX analysis revealed that the results obtained was in agreement with the result obtained from the XRF analysis. The result obtained from the EDX analysis revealed the compositions and phase distribution of the ore were formed at different phases in relations to the ore' sample. The mineralogy and crystallography of the different phases and the mineral processing of the ore are affected by the properties of the ore. The technique used further revealed the major concentration by weight % such as Oxygen 25.2, Carbon 4.08, Fe 55.8, Ti 2.20 Mn 6.40. Si 4.20 and Al 2.12. The result obtained from the XRF agrees with the results obtained from the XRF which was 59.79 for Fe_2O_3.

iv. Fourier transformed Infrared Spectroscopy revealed the positions that are related to the vibrations of the bonds within the molecules of the material being analyzed. The position of a peak in the spectrum were used to identify the functional groups present in the ore, and was able to determine the strength of the bonds. This technique m further revealed the stretching and vibrational positions of the peaks which provide detailed information on the oxidation of the ore.

v. The hardness testing instrument was used to test the hardness value of the ore m which gave a value of 6.0 on the Mohr's scale.
vi. According to the findings, Ezeagu iron ore is an additional prospective resource that should be investigated and used to produce iron and steel.

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