Characterization of Ikperejere Iron Shale Deposits Using ICP-OES, XRD, SEM-EDS

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Abstract:- The study focused on the characterization of iron shale deposits from ikperejere area using **Plasma-Optical** Inductively Coupled Emission Spectroscopy (ICP-OES), Scanning Electron Microscope (SEM) with Energy Dispersive Spectrometer (EDS) and X-Ray Diffraction Studies (XRD). Studies using (ICP-OES), shows the mineralogical and micro-structural analysis which allows the mineral phase identification, nature of iron containing minerals, and gangue minerals. Studies using SEM-EDS shows the micro-morphological and mineralogical compositions of the individual elements present in the sample. Studies using (XRD) shows the individual patterns of mineral phases contained in the ore.

The characterization carried out on the shale using ICP-OES, SEM-EDS and XRD reveals a large content of Hematite, which when exploited can be very useful in the production of iron.

I. INTRODUCTION

In the planning and operation of mining and mineral processing activities, mineral identification and characterization are crucial [I]. There is a requirement for detailed information on a mineral deposit's mineralogical makeup which shows that mineral characterization forms an integral and often crucial part in the investigation of deposits [2]. The Mineralogical and chemical composition, morphology, and association with other minerals are thus intended to provide information and insights into the qualities, nature and amounts of minerals and elements present within the ore at various locations, which allows the optimal processing path for its constituent minerals to be assessed and determined.

Metallic iron may be economically produced from iron ore, which are rocks and minerals. The ores, which range in hue from dark grey to bright yellow to deep purple to rusty red, are typically prolific.[3]Iron bearing compounds that are commonly used are: Hematite Fe₂O₃(70% Fe); Magnetite Fe₃O₄(72.4% Fe) and of much less importance are Limonite (2FeO₃)3H₂O (60% Fe);Pyrite FeS₂(46.6%) [4]. These percentages of iron are in their purest form. More specifically, high grade ores have a content of more than 65 percent; medium or average grade ores have a content of 62-64 percent; and low grade ores have a content of less than 58 percent. [5]. These iron ores have been mined to make nearly every iron and steel object used today, from paper clips to the steel beams used in building construction. It is therefore necessary to identify new sources of iron ore deposits to meet the increasing demand for iron, thereby

leading to the uncovering and exploiting of high grade iron shale [6].

When the first photosynthesizing species began releasing oxygen into the oceans, iron ore deposits began to emerge. Hematite or magnetite was formed when oxygen reacted with the plentiful dissolved iron. Banded iron is formed when these minerals are deposited in large quantities on the seafloor. Because iron minerals accumulated in alternating bands with silica and in some cases shale, the rocks are called "banded." Seasonal fluctuations in organism activity could have caused the banding. [7]. These banded rocks gives rise to mud-rocks of shale and iron-sandstone containing Quartz (SiO₂), Hematite (Fe₂O₃) and Kaolinite (Al₂Si₂O₅(OH)₄).

Iron-shale is material made up of iron oxides and generated by the weathering of an iron meteorite, usually with a layered structure {8]. Iron-shale has drawn the attention of researchers from several disciplines throughout the world. These iron-shale deposits have been known to occur in the Ikperejere area of Ihitte Uboma. L.G.A of Imo State, South Eastern Nigeria. The iron shale is intercalations within the Benin formation. The mined rock is of high importance in the construction of roads and buildings.

II. STATEMENT OF PROBLEM

The characterization of ore and the quantitative analysis of the mineral content and chemical content of ore using advanced equipment such as the 1CP-OES, XRD, SEM-EDS has been often neglected as other means of mineral identification such as Leaching, Multi Gravity Separation and Ore Beneficiation has been employed: Identifying minerals using Leaching methods and Ore Beneficiation tends to be time consuming and does not give accurate results. The use of advanced equipment such as the ICP-OES, XRD, SEM-EDS in mineral characterization corrects the problem of result inaccuracy and it is also more efficient. These equipment reveal all minerals including the elements present in the iron shale.

III. AIM OF RESEARCH

The aim of the study is to find out the mineralogical composition and surface properties of iron-shale obtained from Ikperejere in Imo state Nigeria using the Inductively Coupled Plasma- Optical Emission Spectrophotometer (ICP-OES), X- Ray Diffractometer (XRD) and Scan Electron Microscope-Electron Diffraction Spectrophotometer (SEM-EDS).

This research work is being carried out to ascertain the chemical and mineralogical compositions of the iron shale found in Ikperejere Ihitte Uboma L.G.A of Imo State. This in turn reveals the quantity and quality of iron in the ironshale to know if the ore deposits will serve as raw materials for iron and steel industries.

Characterization of iron ores simply helps to show or bring to view the most noticeable qualities or features of an iron ore, using various steps and methods. Characterization has to be done holistically; that is with consideration of ore quality and product quality [9]. The steps and methods used in the characterization of iron ores includes: X-Ray Diffraction study (XRD), Scanning Electron Microscope with Energy Dispersive. Spectroscopy (SEM-EDS) and Inductively Coupled Plasma-Optical Emission Spectrophotometer (ICP-OES)

IV. MATERIALS AND METHODOLOGY

This section accurately accounts for the experimental techniques employed in the characterization of the iron shale deposit. Basic practical procedures and principals behind all the equipment's used are accounted for in this section.

- **Reagents:** All chemicals used for this research are analytical grade (Annular) provided by the chemical laboratory of the Chemistry Department of Federal University of Technology Owerri. The iron shale was gotten from Ikperejere inIhitte Uboma L.G.A. Imo State, Nigeria. At room temperature of 25°C, all practical experiments were conducted.
- Equipment: Scanning Electron Microscope (SEM) Joel JSM-6400 model, X-Ray Power Diffractometer (XRD) Scintag XDS 2000 model, Leman laboratory Inductively Coupled Plasma-Optical Emission Spectrometer (ICP-OES).
- Sample preparation: Representative samples were collected from the Ikperejere Iron shale deposit. The samples were collected randomly from different spots at a radius of 5 meters. 5Kg of the representative iron shale sample was grinded to particles and dried at 100°c overnight. The iron shale sample was further pulverized with an electrical ball mill and sieve using sieve sizes of ASTM -1 50mesh (<100utn)
- Chemical analysis: Mineralogical analysis on the iron shale sample carried out by the ICP-OES, SEM-EDS, and XRD are given as;
- Inductively Coupled Plasma- Optical Emission Spectrometer
 - > Procedure

0.2g of iron shale was put in a crucible containing I.20g of Lithium Metaborate (LiBO₂).

A fused digestion of the mixture of iron shale samples and Lithium Metaborate (LiBO₂) was carried out at 1000°cin a furnace for 20minutes. The red-hot fused mixture was placed into 100mL of 10% hydrochloric acid right away. A magnetic stirrer is used to agitate the solution until all of the solid particles have been removed. The solution is made up to 250m1 with deionized water; the solution analyzed with Leman Inductively Coupled Plasma-Optical Emission Spectrometer.

ICP-OES (Inductively Coupled Plasma Optical Emission Spectrometer) is a quick multi-element approach with a dynamic linear range and reasonable detection limits (-0.2-100 ppb). The equipment uses an 1CP source, created by ionizing argon gas to breakdown the sample into its constituent atoms or ions and excite them to the point where they emit light with a specific wavelength A single sample run of less than 60 elements can screen up to 60 elements in60 seconds and the samples can be analyzed in a range of organic or aqueous matrices There is a lower chemical interference than with FAAS (Flame Atomic Absorption Spectroscopy), but some spectral interference are feasible, however some elements have constraints. Sample quantities range from 2-4ml depending on the number of wavelengths required, and total dissolved solids must be <5 %.

Basic Principle

It depicts the quantitative elemental composition of the iron shale sample. It gives the quantity of elemental oxides expressed in percentages. Its precision is in parts per billion.

• X-Ray Power Diffraction (XRD)

Procedure;

Analysis of the iron shale sample using the X-ray Diffraction was performed as follows; The sample is held in a Lucite holder on the scintag XDS powder diffractometer's goniometer.

It was also equipped with a graphite monochromator and an IBM compatible workstation running Scintag DMSNT software under Windows NT. The monochromator for the diffraction beam worked at 20KVA with a step size of 0.02° c for 20 minutes to create X-ray patterns with sufficient intensities to make lines that can be used to detect minerals from two angles (5°-90°). Scanning rate was 0.75 degrees per minutes.

X-ray crystallography is a method for determining the arrangement of atoms in a crystal in which an X-ray beam strikes the crystal and causes the beam of light to scatter in many different directions. A crystallographer may use the angles and intensities of these diffracted beams to construct a three-dimensional image of the electrons within the crystal. The atoms' typical positions in the crystal, as well as their chemical bonds, disorder and other information, can be deduced using this electron density.

X-ray crystals structures can also explain a material's peculiar electrical or elastic properties, throw insight on chemical processes, and serve as the framework for developing disease-fighting medications.

In X-ray diffraction measurement, a crystal is mounted to a goniometer and spun slowly while being exposed to X-rays, resulting in a diffraction pattern of regular spaced spots termed reflections. The twodimensional images acquired at different rotations are transformed into a three-dimensional model of the density of electrons within the crystal using the mathematical method of Fourier transforms, together with chemical data for the sample. If the crystals are too tiny or have an uneven internal makeup, poor resolution or mistakes may occur.

Scattering electrons or neutrons can generate similar diffraction patterns, which are perceived similarly as a Fourier transform. Various additional Xray technologies can be used to provide less detailed information if single crystals of suitable size cannot be produced; such methods include fiber diffraction, powder diffraction and small-angle X-ray scattering (SAXSF). The scattering is elastic in all of the X-ray diffraction methods listed above; the scattered X-rays have the same wavelength as the incoming X-ray. Inelastic X-ray scattering methods, on the other hand, are beneficial for investigating the sample's excitations rather than the distribution of its atoms.

➢ Basic principle

The X-ray diffractometer depicts mineralogical composition of the iron shale sample that is the quantity of minerals present in the iron shale sample. The X-ray diffractometer displays the result in spectral peaks and intensities. It is a qualitative analysis.

• Scanning Electron Microscope (SEM) Procedure:

5kg of the representative sample was prepared using epoxy resins, polished and made conductive coding in carbon in a Dentom vaccum, DV-502A. A morphological analysis on the iron shale was analyzed using a JOEL JSM-6400 scanning electron microscope at accelerating voltage of 2KVA, real time of 21 -36 seconds and live time of 60 seconds. It had an ultra-thin window energy dispersive Xray spectrometry system, three WDS spectrometers, and a motorized stage. The back scattering electron detectors were used to create the images. Point analysis was used to illustrate the images of the ore particles. A scanning electron microscope (SEM) is a type of electron microscope that uses electrons to examine objects. It uses a raster scan pattern to image a material with a beam of electrons. Electrons interact with the atoms in the sample to produce signals that contain information about the sample's surface topography, composition, and other characteristics such as electrical conductivity.

Secondary electrons, hack-scattered electrons (HSE), distinctive X-rays, light (cathodoluminescence), specimen current, and transmitted electrons are some of the signals produced by a SEM. All SEMs feature secondary electron detectors, although it's uncommon for a single machine to have detectors for all potential signals. The signals are caused by electron beam interactions with atoms at or near the sample's surface. Secondary electron imaging, or SEI, is the most frequent or standard detection mode, and it allows the SEM to obtain very high resolution images of a basic surface, revealing details as small as I nm. With very narrow electron beam, SEM micrographs have a broad depth of focus, giving them a distinctive three-dimensional look that helps to grasp the sample's surface structure. Back-scattered electrons (BSE) are electrons reflected from a substance through elastic scattering. In analytical SEM, BSE are frequently employed in conjunction with spectra generated by the characteristic X-rays. The BSE signal is strongly related to the atomic number (Z) of the specimen, The distribution of various components in the sample can be determined using BSE images.BSE imaging can also view colloidal gold, which is difficult to detect in secondary electron pictures in biological specimens.

Basic Principle

The SEM is qualitative analytical equipment. This technique determines the minerals present in the iron shale sample and depicts them in phases and maps, spotting out these minerals and differentiating those, using colours or shades.

• X-Ray Mapping: The equipment indicates elements that were not detected by ICP-OES and XRD. In the iron shale sample, Tungsten containing particles and Fe were displayed in phases.

V. RESULTS

A. Elemental Analysis by inductively coupled Plasma-Optical Electrona) Microscope

The result of the elemental analysis of the iron shale by ICP-OES technique is summarized below.

b) Chemical Composition by ICP-OES

Elemental Oxide	SiO ₂	MnO	Fe ₂ O ₃	MgO	A1 ₂ O ₃	TiO ₂	CaO	Na ₂ O	K ₂ O	P_2O_5	LOI
Concentration	27.68	0.01	54.53	0.05	9.04	0.72	0.05	0.19	0.36	0.02	6.60
Table 1											

From table above, it is evident that the major elements detected by the ICP \neg OES are Fe₂O₃, SiO₂, Al₂O₃, while TiO₂. K₂O, Na₂O are the minor elements in the iron shale. Other metal oxides detected in the iron shale at trace levels were MnO, MgO, CaO, P₂O₅. This iron shale sourced from the south eastern part of Nigeria contains 55% Fe content.

c) X-Ray Diffractometer

Plate 1 shows the identified phases and their respective lattice plane with scintag file number.

d) XRD- Mineral Compositions





The X-ray Diffractometer data in Plate 1 shows that the major mineral composition is hematite Fe_2O_3 and the minor mineral compositions are quartz and kaolinite. It could be extrapolated from the spectra that peak intensities of H (hematite) are broad and pointed, compared to Q (Quartz) and K (kaolinite).Content is in the range of 55%.

The X-ray spectrum data in plate 1 apparently complements the results obtained of the elements analyzed

f) SEM-Kaolinite in iron Shale

by ICP-OES. It shows that iron Fe is present mainly as Fe_2O_3 with the result of the ICP-OES; Fe_2O_3 .

e) Scanning Electron Microscope

The SEM images the iron shale sample and spotting the specific minerals in Phase and maps. Images were made with the use of the back scattering detectors.



Plate 2: SEM point analysis of lkperejere iron shale showing the presence of Kaolinite

g) SEM-Iron Shale-Hematite



Plate 3: SEM point analysis of lkperejere Iron Shale showing the presence or Hematite

h) X-Ray Mapping

In the ICP-OES and XRD, tungsten-containing particles were not detected by these techniques. The tungsten-containing particles wrapped by hematite were detected by the x-ray mapping under SEM.

i) X-Ray Mapping on Iron Shale



Plate 4: X-Ray Mapping of Ikperejere Shale Deposit showing different phases of Iron Fe and Tungsten

j) Electron Dispersive Spectrometer

This technique shows the chemical element in the shale sample by the EDS. The spectral peak indicates the concentration or the elements contained in the iron shale sample. Fe consists of a broad and sharp pointed peak, with an intensity of 22052 .5 and a weight of 61.83%



Plate 5: EDS analysis of iron shale showing the elements of Fe, W, Zr in the SEM Images

VI. DISCUSSION

A. X-ray diffraction study

This method is widely used for purposes of identification, but Because various parameters like as texture, stress, and crystalline size can impact the intensities of the peaks, they are rarely employed for quantification. [10]The Rietveld method can also be used to measure crystalline phases, but only for samples with texture effects The technique is not easy to follow [11]. X-Ray diffraction analysis is an important tool in mineral exploitation, after its discovery, Mineralogists were among the first to create and promote X-Ray crystallography as a new field. Thus, the introduction of XRD has literally transformed the geological level to the point that they are now unthinkable without it, any geological group actively involved in mineralogical studies would be lost without XRD.

X-Ray diffraction studies carried out on Indian iron ore slime by Nayak Nirlipta p, Das Abliimanyu and Pal B.K was taken up with a view to identify mineral phases in the slime sample. In the diffractogram, it is evident that Hematite is the major iron bearing phase. And Goethite is the other iron bearing mineral phase. Kaolinite and Quartz is also supported by X-Ray diffraction data. XRD analysis of the clay material shows that it is mainly composed of kaolinite. Some of the ore fragments have undergone weathering, producing ochreous goethite and kaolinite. This can be called Laterite Ore, it is generally soft and friable and leads to slime generation during handling [12].

X-Ray diffractometer is used unambiguously to characterize the individual crystal structures. Each Mineral type is defined by a specific crystal structure, which will give a unique X-Ray diffraction pattern, allowing rapid identification of minerals present within a rock sample. The XRD data can be analyzed to determine the proportion of the different minerals present.

B. Scanning electron microscopy analysis (SEM)

This method of analysis helps to know the morphological and quantitative analysis of an ore. Scanning electron microscopy is mostly accompanied with energy dispersive X-Ray spectroscopy (EDS). The scanning electron microscope provides information on the physical properties of the minerals present in the ore while Energy dispersive X-Ray spectroscopy (EDS) provides information on the chemistry of the ore, it also helps to outline the elemental composition present in the minerals. EDS is based on the study of an X-Ray source's interaction with a sample. Its characterization skills are essentially based on the idea that each element has a specific atomic structure that allows for a distinct collection of peaks on its X-Ray spectrum. [13].

Scanning electron microscopy analysis carried out on Ishiagu complex sulphide ore by PA Olubambi et al, [14] provided details on their chemistry. Mineral analysis of typical samples was done in two stages using scanning electron microscopy, using SEM model JEOL 840. All the samples were carbon coated in order to make the minerals surface conductively. Samples for analysis were cut, polished, mounts in embedded epoxy resins and finally polished to obtain a mirror like surface. The polished surfaces were finally carbon coated before analysis. Qualitative chemical analysis of minerals was carried out on some mounts of the ore using SEM-EDS to produce back scattered images (BSI).

The scanning electron microscope (SEM) generates a variety of signals at the surface of solid specimens using a focused beam of high-energy electrons. The signals are derived from electron sample interaction; this gives information about the sample, such as its exterior appearance, crystalline structure, and material orientation. The SEM is also capable of performing analysis of selected point location on the sample; this method is particularly useful for determining chemical compositions qualitatively or semi-quantitatively utilizing EDS.

C. Inductively coupled plasma-Optical emissionSpectrometry (ICP-OES)

Inductively coupled plasma-optical emission spectrometry (ICP-OES) also referred to as inductively coupled plasma atomic emission spectroscopy (ICP-AES), is a method for detecting trace metals that uses analytical techniques. It's a sort of emission spectroscopy in which an inductively coupled plasma produces excited atoms and ions that emit electromagnetic radiation at wavelengths specific to a certain element. The intensity of this emission indicates the element concentration in the sample. [15].

The ICP-OES is used in mineral processing, to offer information on the grades of various streams, and to determine metals in win; Food containing arsenic [16]. The ICP-OES is composed of two parts: The ICP and the OES. The ICP has a torch consisting of three concentric Quartz glass tubes. The output coil of the radio frequently (RF) generator surrounds a portion of this quartz torch. The plasma is usually created with argon gas. In the powerful electromagnetic field, the Argon gas is ionized and flows in a specific rotating symmetrical way towards the R.Fcoil's magnetic field.As a result of the inelastic collision between the neutral Argon atoms and the charged particles, a stable, high-temperature plasma of around 7000k is created.During analysis with ICP-OES light is separated into its different wavelengths(colours) within the optical chamber(s), the light intensity is measured with a photo multiplier tube or, to view the specific wavelength for each element line involved, or the separated colours fall upon an array of semiconductor, photo detectors such as charged couple devices. The intensities of all wavelengths can be measured simultaneously in units using those detector arrays, allowing the instruments to analyze for every element to which the unit is sensitive all at once. As a result, the samples can be evaluated fast.

D. Characterization of some Nigerian iron ores

Mineralogical study, aids in identifying mineral phases and associated gauge materials since these materials greatly influence the bulk chemistry of the process [17]. A clear mineralogical analysis of iron ore samples of any particular deposits helps in selecting suitable blast furnace grade ore through the usual practice of blending, beneficiation.

Chemical characterization involves the analysis of iron ore and agglomerates. The major constituents of iron burden are Fe,₂O₃, FeO, SiO₂, Al₂O₃ and trace elements like Cu, Ni, Co, Pb, Zn and Mn.

The determination of trace elements in mineral solid samples were first used with ICP-OES[18] and with ICP-MS Inductively Coupled Plasma-Microscopic Spectrophotometer [19], it has the capability of determining the concentrates of trace elements in ppb levels in geologic and biologic solid micro samples.

Annelore ct al, [20] in their characterization of sedimentary quartzite artifacts from Mesolithic sites using Scanning Electron Microscope and ICP-OES gave the concentrates of the chemical elements; Zirconium, titanium and iron which showed a difference between the three quartzite varieties.

According to keeling, et at [21] in their research work, conducted and mineralogical analysis of Proterozoicmagnesite using 1CP-OES, quantitative XRD and SEM showed that both Fe and Ca are present and substitute for mg in magnesite with an average of Fe_2O_3 content 1.5% and a CaO content of 1.7%. During the SEM investigations, Quartz was not observed but was reported in the XRD.

The geochemical data of a given set of samples are obtained by 1CP-OFF analysis, a research work carried out by Suhr, et al [22]. The lithium Metaborate Procedure is exerted on these pulverized homogenous samples to determine these major elemental oxides Al₂O, CaO,Fe₂O₃, K₂O, P₂O₅ and TiO₂ and trace elements Ba, Cr, Cu, Sc, Sr and Zr.

Only elements as Al₂O₃, Ba, Fe₂O₃, Sr, TiO₂ and Zr, are used for the characterization of the quartizites because of its high precision. The best results of the sample were obtained from the optical microscope by determining the grain size distribution of the samples and from 1CP-OES in particular; the elements titanium [from the mineral rutile], Zirconium [from the mineral zircon], iron and aluminium with the aid of discriminant function analysis in a research work carried out by Ahmed, et al [23], on the effective processing of lowgrade ore through gravity and magnetic separation techniques characterized the ore using X-ray differential Analysis found that the iron ore sample consisted of goethite, hematite, quartz, calcite, kaolin and feldspar. The major constituents were goethite as an iron mineral and quartz as a gangue mineral. Under optical microscope, it was observed that the iron minerals and quartz are finely disseminated. The iron content in their research work is relatively low based on the chemical analysis conducted on the low-grade ore, in the range of 36% and the major gangue mineral is quartz in the range of 48% SiO₂.

Olubambi et al, [2008] conducted a research work on the combination of SEM combine with Energy Dispersive

X-ray analysis EDX,X-ray diffractometry XRD, X-ray Fluorescence, and Inductively Coupled Plasma-Optical Emission Spectrophotometer ICP-OES, to characterize the bulk complex sulphide ore deposit. Mineralogical analysis on this study revealed the presence of siderite, sphalerite, quartz, galena and the traces of pyrite and Cu bearing minerals. The SEM revealed that the ore is made up of fine to coarse grains with a simplerinter growth of the constituent crystalline phases both at the interstitial and boundaries with low inter-penetrations. The ore sample here, showed a characteristic hydro thermal complex sulphide deposit, nature of fine to coarse grains inter growths of the constituents crystalline phases [24].

A research work on the characterization of Indian iron ore slime by Nayak, et al, [25] revealed that these slimes contain substantial iron values in the range of 54-58% Fe. The mineralogy of the slime sample though indicated that hematite is the major iron bearing phase, goethite also occurs in substantial quantity. The XRD study depicted Kaolinite and Quartz occurring as the major gangue phases.

According to Pinaki, et al [26] researched on the characterization of iron-rich kaolinite clay using (SEM-EDX) scanning electron microscope and Energy Dispersive X-ray Spectrometer. Their study revealed that this kaolin clay deposit contains high amount of iron.

VII. CONCLUSION

The chemical composition and micro structure of raw iron shale deposits from the lkperejere area were investigated. Based on the results obtained during characterization of the shale using X-Ray diffraction analysis, it shows conclusively that the shale sample deposits from lkperejere were recorded to be more of a hematic nature. It was also discovered that, while the chemical content of the samples is nearly same, the microstructures differ significantly. They are more explicitly dependent on the diverse spontaneous conditions that existed during the sample's formation.

The iron content from this shale sample amounts to 6 I.85%Fe and an intensity of 22052.5 as shown by the EDS spectra. On the basis of the findings, it may be inferred that the shale sample contains high-grade hematite ore that can be profitably mined for iron production.

VIII. RECOMMENDTION

It is worth clarifying that the aim of this work is not to suggest that the ICP-OES, SEM-EDS and XRD technologies. Should replace or supersede conventional technologies such a Multi Gravity Separation, Leaching and Beneficiation processes but to highlight that the ICP-OES, SEM-EDS and XRD gives accurate and on point analysis, shows the microstructure of the ore to be analyzed and it is also a step in the appreciation of advanced technology.

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