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CHARACTERIZATION AND MINERALOGICAL STUDIES OF AKIRI COPPER ORE IN AWE LGA, NASARAWA STATE, NIGERIA USING ED-XRF, XRD AND SEM-EDS

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ABSTRACT
This study used ED-XRF, XRD, and SEM-EDS to evaluate the thorough characterization and mineralogical studies of Akiri copper ore in Awe LGA, Nasarawa State, Nigeria. The ore's exceptional grindability and flotation behavior are suggested by its physical properties, which include bulk density of 3.58 g/cm^3, compression test of 1.24 %, and hardness of 96.0N. For efficient copper recovery, the property might be investigated for leaching and solvent extraction. Over-size fractions above 250 µm were found in the +0-90, +125-90 µm, and +125-180 µm particle size analyses. According to the ED-XRF data, the raw copper ore's elemental composition was 2.711±0.003 %, with Loss on Ignition (LOI) at 3.11±0.750 %, moisture content (bMC) at 1.31±0.40 %, and a pH of 6.84±1.90, all of which were within the typical cutoff grades for copper mining. Pyrite (FeS_2), Quartz (SiO_2), and Chalcopyrite ($CuFeS_2$) were detected by XRD examination. The ore matrix contains 2.53 % copper as verified by SEM-EDS. These results imply that the ore may be used for mineral processing and extraction.

Keyword: ED-XRF, XRD and SEM-EDS of Akiri copper ore

INTRODUCTION
The unexpected need for renewable energy sources has led to an increase in demand for copper, a metal essential to modern technology and infrastructure (Minyo *et al*., 2024). Lower-grade ores must be mined because high-grade copper deposits are getting harder to come by. Because of their special characteristics and techniques of separation, these low-grade copper ores are challenging to treat. Studying the mineralogical properties of the ores is crucial to comprehending their physical and chemical characteristics (Wang *et al*., 2020).
In general, ore characterization studies offer comprehensive and useful details regarding the chemical and physical characteristics of low-grade copper ore. This covers the mineralogical makeup, minerals that include copper, and how they interact with gangue minerals, or undesirable minerals. Accurate study of the mineral phases, grain sizes, and elemental distribution is made possible by contemporary methods including energy dispersive spectroscopy (EDS) in conjunction with X-ray fluorescence (XRF), X-ray diffraction (XRD), and scanning electron microscopy (SEM). These tests assist quantify the overall copper content and identify any dangerous compounds that could impair processing and the quality of the final product. They also provide a precise understanding of the chemistry of the ore (Guo *et al*., 2021).
Akiri ores samples are classified based on their behavior, tissue qualities, particle sizes, structure, and the amount of metal or mineral they contain. These features are mostly used to determine the optimal beneficiation method and the states of mineral liberation. The mineral's values changed depending on the makeup of the ore; sulfur and copper ore are important components of the deposit. Therefore, the ore most likely contains a cuprite mineral. The ore may contain other minerals in addition to the higher amounts of Cu and Fe. The ore can be examined for Cu and other valuable minerals that include Zn and Mo. The development of copper ore deposits and other minerals resources will someday reposition the country's economy in the globe competitive market. Copper mining has the ability to promote foreign exchange, technological advancement and employment growth. Thus, the mineralogical characterization of Awe copper ore will support the government's continuous efforts to develop and benefit the ore industry (Ambo & Glass, 2020; Mohanraj *et al*., 2022).
In this work, comprehensive characterization was performed based on the chemical and mineralogical parameters of Akiri copper ore samples from Awe LGA, Nasarawa State. It is intended that the results will act as a database to provide greater insight into scientific investigations and the selection of beneficiation procedures for the ore in question.

MATERIALS AND METHODS
Study Area
The research region is the town of Akiri in Nasarawa State's Awe Local Government region (Fig. 1). The location is located in Nigeria's Middle-Belt trough, which is rich in minerals and is commonly referred to as a mineralized zone due to the presence of Pb, Zn, barite, coal, copper ore, and other valuable metals

(Ambo *et al*., 2023). The town is located in latitude N8.342825˚ and longitude E9.262246˚ on the global position system, with an elevation of 365 m above sea level. Awe Local Government Area is located approximately 99.3 kilometers from Lafia, the state capital, along Markudi Road in Benue state. The vein that contains the copper ore in Awe lies around 5 kilometers east of Azara hamlet (Oyebola & Wahab, 2015).

Figure 1: Map of Nigeria showing the location of Awe LGA (Hamza *et al*., 2020)

Sample Collection

The composite ores samples utilized in this investigation were gathered from active mining locations in Akiri, Nasarawa State, with the cooperation of miners. A total of 25 kg of the material was gathered into a plastic bag and delivered to the laboratory for chemical and mineral analysis.

Sample Preparation and Analysis

The entire sample was crushed, milled and ground to obtain 125/-0.90, 125/-180, and +0.90 μm particles sizes.Then subsequently sieved using standard sieves as described by the American Standard sieve series to obtain different size fractions of oversize (Above 250 μm) 125/-0.90 μm, 125/-180 μm, and +0.90 μm. Prior to analysis, the sample was homogenized (Amos *et al*., 2020). The physicochemical analysis (compression test, sieve analysis, bulk density and hardness were carried out according to the methods described by Ambo *et al*. (2023), while the elemental content was analyzed using Energy Dispersive X-ray fluorescence spectrometry (ED-XRFS) (mini PAL 4 model (c) 2005) at the National Metallurgical Development Centre (NMDC), Jos. The chemical and mineralogical characterizations were carried out using the procedures (Minyo *et al*., 2024).

Chemical Characterization using ED-XRF

Chemical characterization of the copper ore was carried out using Energy Dispersive X-ray Fluorescence Spectrometer (SKYRAY 3600B EDXRF analyzer). Twenty (20) grams of the sample was finely ground to pass through a 200-250 mesh sieve. Thereafter, the sample was intimately mixed with a binder in the ratio of 5.0 g sample(s) to 1.0 g cellulose flakes binder and pelletized at a pressure 5 of 10-15 tons/inch in a pelletizing machine. Then, the pelletized copper samples are stored in a desiccator before analysis. The ED-XRF machine was allowed to warm up for 2 h after switching it on before the samples were introduced to it. Finally, appropriate programs for the various elements of interest were employed to analyze the sample material(s) for their presence or absence. The result of the analysis was reported in percentage (%) composition for trace and major concentrations of elements.

Mineralogical Characterization using XRD

Qualitative and quantitative determination of the nature of the phases and the amount of the phases present in the sample were determined by a PAN analytical Empyrean diffract meter with Pixel-detector and fixed slits with Fe-filtered Co-Kα radiation. The material was prepared for XRD analysis using a back loading preparation method. The phases were identified using X' Per High score plus software. The relative phase amounts (% weight) were evaluated using the Rietveld method.

Mineralogical Characterization using SEM/EDS

Morphological and qualitative analyses of the bulk ore was performed using SEM-EDS. The SEM provides information on the spatial distribution of mineral phases present in the crude, while EDS provides information on their elemental composition. Mineralogical analysis via SEM-EDS was conducted on representative samples in two stages using SEM (Model: JEOL 840). All the samples were carbon-coated to make the mineral's surface conductive. Samples for analysis were cut, polished and mounted in embedded epoxy resin, then polished to obtain a mirror-like surface. The polished surfaces were finally carbon-coated before analysis. Qualitative chemical analysis of the samples was carried out using an EDS detector attached to the SEM.

RESULTS AND DISCUSSION

The result of the physical parameters of the copper ore revealed certain characteristics of copper ores (Table 1). From the analysis, the compression test clearly demonstrated that the relative deformation behavior of the copper ore was 1.24 % with a force yield of 8329N. The bulk density (g/cm^3) was determined to be 3.58, while the hardness (N) of the copper ore was 96.0.A compression test is important in analyzing the elastic and compressive fracture properties of a brittle material or low-density material (Mohanraj *et al*., 2022). It mostly describes the behavior or Responsiveness of the copper ore under crushing loads and measures the plastic flow behavior and ductile fracture limits of copper ore.

The Compression test result obtained was lower compared to the result of 66.6% reported for the influence of the degree of tension and compression of copper on the indentation size effect (Peter *et al*., 2024). The bulk density of copper ore is important in the area of impacting numerous operational factors, which include but are not limited to mine design, mine planning, equipment selection and operational performances (Makhuvha & Harney, 2014). The bulk density result of 3.58 g/cm^3 is higher when compared to the average bulk density range 2.3 – 2.3 Mg/m^3 reported for accuracy of the local assessment of the bulk density of copper-silver deposits in Legnica-Głogów copper district and its impact on the valuation of ore resource and mining production (Monika & Jacek, 2019). The hardness test describes the resistance to permanent distortion, penetration, indentation and scratching, which serves as an important parameter from an engineering point of view because of its resistance to wear by either friction or erosion from steam, and oil (U.S. Geological Survey, 2020). The hardness test result 96.0 (N) recorded for copper ore was higher than the result 3.5 using the Mohs hardness scale, recorded for the characterization of Azara copper ore deposit, Nasarawa state, Nigeria (Oyebola & Wahab, 2015).

Table 1: Result of physical parameter of Akiri copper ore sample

S/N	Parameter	Value
1.	Bulk density (g/cm^3)	3.58
2.	Compression Test (%)	1.24
3.	Hardness (N)	96.0

Table 2: Result of particle size analysis of the Akiri copper ore sample

S/N	Particle size (µm)	Weight (kg)	Normal aperture size (µm)
1	+0.90	7	–
2	+125-90	5	90
3	+125-180	6	180
4	Over size (above 250 µm)	4	250

The result of the particle size analysis (Table 2) indicated that the total weight of 4 kg was retained on the oversized fractions (Above 250 µm), 6 kg on+125-180 with normal aperture size (180 µm), 5 kg on +125-90 with normal aperture size (90 µm) and 7 kg on +0.90 µm, respectively. This suggests the excellent grind ability and flotation behavior of the ore. The property could be explored for leaching and solvent extraction for effective recovery of copper metal from the ore.

From the results of the Elemental composition of the raw copper ore by ED-XRF in Table 3, the results 2.71 % reported for elemental composition of Akiri copper ore was lower compared to the result 53.2 % reported for Anka-Brabra copper ore. The result (2.71 %) reported Akiri copper ore exceeds the 0.5 % cut-off grade specified as standard for mining of copper mineral (Barani *et al*., 2021; Ju *et al*., 2021).

Table 3: Elemental composition of the raw copper ore by ED-XRF

Component	Content (wt. %) ±SD
SiO_2	11.654 ±0.001
Al_2O_3	3.489±0.001
Fe_2O_3	46.621±0.000
CuO	2.711±0.003
SO_3	24.526±0.000
MgO	6.482±0.000
MnO	1.261±0.003
CaO	1.114±0.000
ZnO	0.523±0.003
CoO	0.210±0.001
[a]LOI	3.11±0.750
[b]MC	1.31±0.40
[c]pH	6.84±1.90

[a]Loss on ignition, [b]Moisture content, [c]Equilibrium pH

The ore characterized does not contain pure copper, but it does contain copper-sulphur-iron compound (Chalcopyrite) deposit, in most cases copper ores which contain 0.6-1 percent can generate a maximum of 6 to 10 kilograms of copper. This phenomenon is attributed to the geochemical formation of the deposit (Niu *et al*., 2024). The result is in accordance with the assertion of the nature of the ore. The loss of ignition ([a]*Loss on ignition* LOI) expressed as a percentage of the original weight of oven dry Akiri copper sample result 3.11 %, is lower as compared to the results (7.5 %) reported the optimization of copper recovery from oxide-sulfide ores through gravity separation and flotation technique (Arash & Bahram, 2026). The moisture content expressed as the ratio of the mass of water contained in the copper ore pore spaces to the solid mass of the copper ore, as expressed in percentage ([b]MC) result 1.31 % for Akiri copper ore (Table 3) was within the range of the tested granular material copper ore of particle size 0–2 mm and relative moisture content ranged 0.5–11 % results reported by Dariusz *et al*. (2023) for the moisture determination for fine-sized copper ore by computer vision and Thermovision method.

The equilibrium pH result 6.84 for Akiri copper ore was below than the range pH 8 to 10 reported for the Optimization of Copper-Ammonia-Sulfate Electrolyte for Maximizing Cu(I):Cu(II) Ratio, using pH and Copper Solubility (Zulgarnain & Joshua, 2024). The pH of copper ore varies depending on the chemical composition of the ore and the atmospheric conditions it has been exposed to.

XRD Result of Akiri Copper Ore

Figure 2 presents the XRD results of Akiri copper ore sample analyzed. The result revealed that the Akiri copper ore contains chalcopyrite ($CuFeS_2$: 01-011-0570). There are also traces of pyrite (FeS_2:01-071-4755) and quartz (SiO_2: 01-082-0511), as affirmed in Fig. 2. The XRD results above help to reveal the minerals or elements that occur along the mineral of interest (chalcopyrite), as expected, infinitesimal quantities of gangue material, such as pyrite and quartz was observed. Chalcopyrite was the most abundant; the presence of small quantities of quartz might be due to contamination during the collection of samples (Ambo & Glass, 2020). Thus, it can be inferred that the copper element in Akiri deposit occurs as chalcopyrite alongside associated minerals, hence there is need for the beneficiation of the ore to recover the copper mineral. The XRD results obtained also conform to that of the XRF analysis which affirmed the presence copper oxide in the ore matrix.

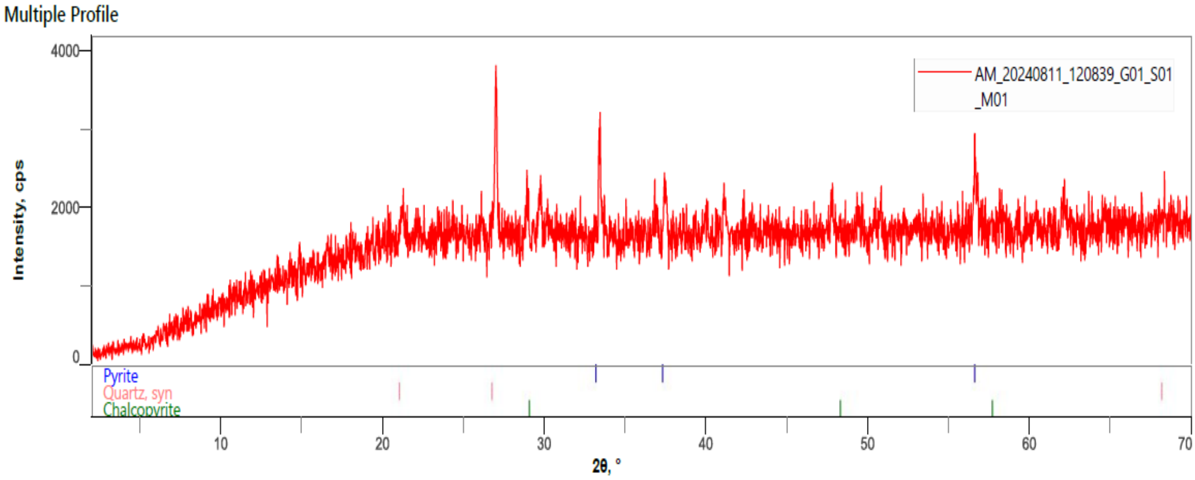

Figure 2: XRD pattern of the Akiri copper ore sample

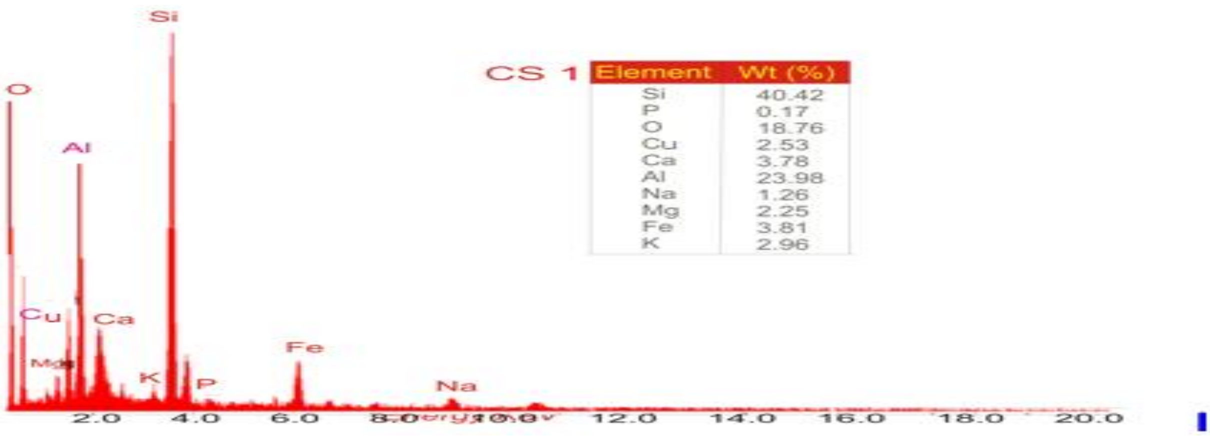

Figure 3: SEM-EDS micrograph of the Akiri copper ore

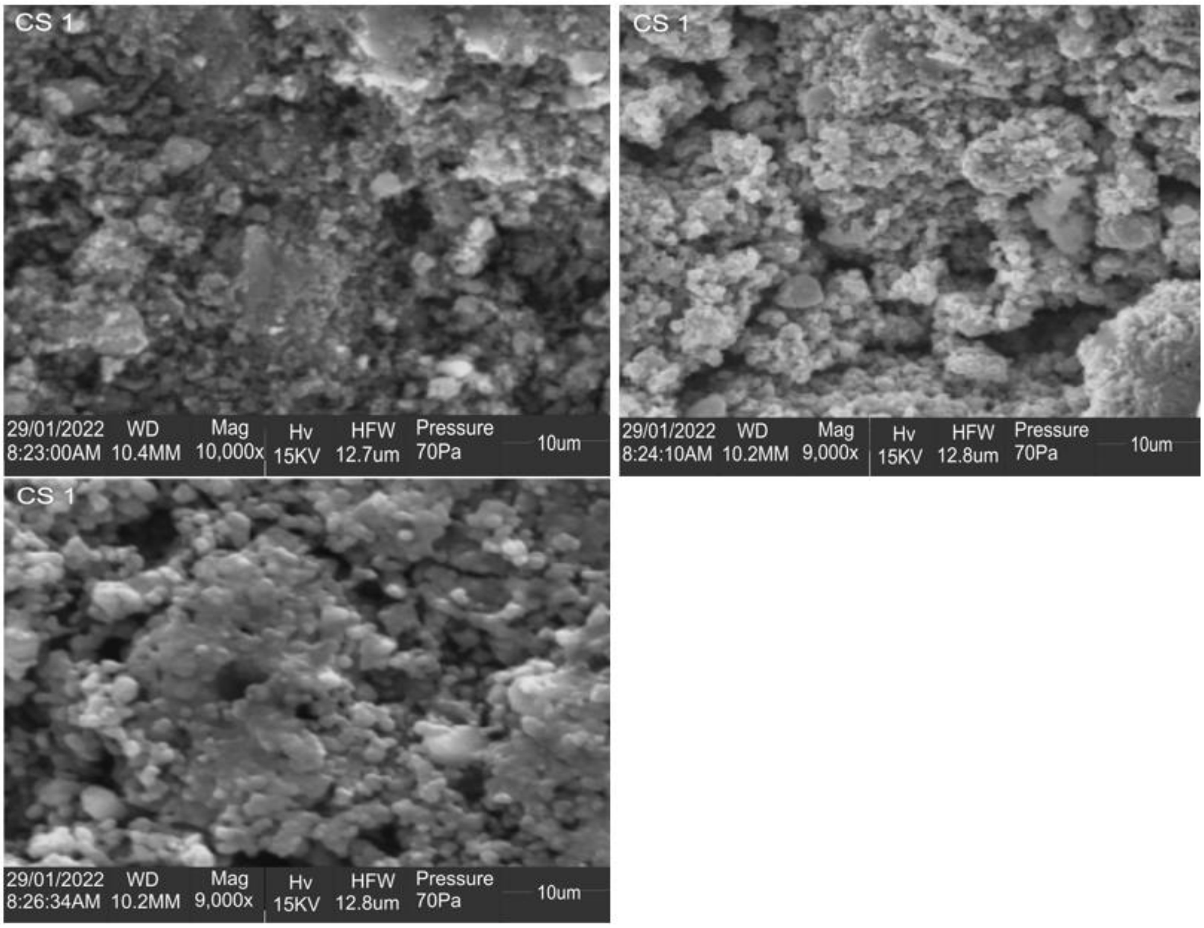

Figure 4: SEM-EDS images of the Akiri copper ore sample captured at varying magnifications

SEM/EDX Results of Akiri Copper Ore

Figure 4 displays pictures of the Akiri copper ore sample taken at different magnifications, while Fig. 3 (Tables CS1) displays the EDS analysis of the ore and identified mineral grains, respectively. It can be inferred from the micrographs that SEM peaks of the Akiri copper ore sample taken at different magnifications contain silicate, phosphorous, oxygen, copper, calcium, aluminum, sodium, magnesium, iron, and potassium grains scattered throughout the ore matrix. The ore's SEM-EDS examination results confirmed the presence of copper (2.53 %) within the ore matrix; however, the SEM-EDS result 2.53 % was lower as compared to the elemental composition result (2.71 %), as presented by the ED-XRF.

CONCLUSION

Using ED-XRF, XRD, and SEM-EDS, for the Characterization and Mineralogical Studies of Akiri Copper Ore in Awe LGA, Nasarawa State, Nigeria, it can be concluded that the ore contains copper (2.53 %) and chalcopyrite ($CuFeS_2$) in the ore matrix, which values fall within the s-off grade that is established as a standard for copper mineral mining. The outcome suggests that the ore may be investigated for the purpose of processing precious minerals.

Recommendations

From the findings of this research, we recommend the following:

1. Accurate assessment of the ore's chemical and mineralogical makeup, prior to moving the materials on to subsequent processing steps. For instance, before putting ores through processing, more focus should be placed on their analysis.
2. Low-grade ores or lean resources should be taken into account in order to lessen the over-reliance on oil as a source of income.
3. Mineral ore generally should be properly mined and processed, thereby adding value to them before exportation.

Conflict of interest: The authors declare no conflict of interest.

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