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Characterization and acid leaching beneficiation studies of copper ore deposit in Anka Zamfara State, Nigeria

Olatubosun A. B., *Alabi O. O., Gbadamosi Y. E. and Oladepo J. G

Department of Metallurgical and Materials Engineering, Federal University of Technology, Akure

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**Corresponding Author:*
aoalabi@futa.edu.ng

ABSTRACT

This study involved chemical and mineralogical characterization of the Anka copper based on its susceptibility to H_2SO_4 acid leaching. A crude sample of the ore, weighing 5 kg, was sourced from the mines, communiated, homogenized, and characterised. A 100 g fraction of the crushed sample was subjected to fractional sieve analysis and each sieve retained was chemically analysed. The leaching process was carried out on 100 g of -125+90 µm sieve size fraction using H_2SO_4 acid and the resulting product was chemically analyzed. The characterization result reveals 42.271% CuO using the energy dispersive x-ray fluorescence spectrometer and the x-ray diffractometer shows that the ore is mainly composed of Cu-carbonate hydroxide in the form of malachite mineral. The scanning electron microscope micrograph suggests that the ore contains dispersed grains of sodium aluminium silicate and copper carbonate hydroxide in a silica-dominated matrix. The ore's liberation size was determined to be at the sieve size of -125+90 µm. Finally, the ore was discovered to be responsive to the H_2SO_4 acid leaching operation, leading to an enrichment of the feed from 42.27% Cu to a pregnant solution of 52.51% Cu and the residue from the leaching was analyzed to be 4.69% Cu.

INTRODUCTION

Copper is a chemical element that can be found in a wide range of minerals. It is mostly found in the northern part of the country, in areas like Nasarawa, Plateau, Zamfara, Bauchi, Gombe State, and Kano State, as well as in the south, in locations like Abia. Copper mining is currently carried out on a small-scale basis throughout West Africa, particularly in Nigeria (Taofeek and Amos 2023). When investors execute maximal utilization or manufacturing, some of these can be exported to other countries in a minimal amount. It is estimated that the above places contain 10 million tons of copper ore resources (Apua and Madiba, 2021). Copper improves the processing, microstructure, characteristics, and performance of a wide range of metals. Furthermore, adding copper to other metals such as aluminium, zinc, tin, and others has improved their performance. This contributes to the need for the most efficient method of processing the copper mineral; however, early identification of copper minerals in an ore, as well as their behaviour, structural associations, and micro-chemical composition, plays a crucial role in determining the efficiency of the mineral processing and the minerals separation to use (Sekhar, 2023). The trust of this study is to boost the local supply due to its enormous applications because there is a steady and increasing demand for this mineral as it is of various industrial uses (Amos *et al*., 2020). This will facilitate better resource exploitation, which will, in the long run, sustain its enormous application toward the satisfaction of local content. This will go a long way toward promoting the development of the ore in terms of revenue generation, superstructure, and infrastructural development in the country (Apua and Madiba, 2021; Sekhar, 2023). The effectiveness of the acid-leaching process for copper depends on several factors such as the concentration and strength of the acid, the particle size of the ore, the temperature and pressure of the reaction, and the length of time the ore is in contact with the acid (Magwaneng *et al*., 2018). In addition to the environmental concerns mentioned earlier, the acid-leaching process can also be expensive due to the cost of the acid and the need for additional purification steps. However, it remains a popular and effective method for the beneficiation of copper ore (Chindo *et al*., 2022; Aigul *et al*., 2023).

Zamfara State is located in Northwestern Nigeria, at latitudes 10°5000 - 13°800N, longitudes 4°1600 - 7°1300E, and an elevation of 451.00 meters above sea level (Dalhatu *et al*., 2022). Zamfara State borders the Republic of Niger to the northeast, Kaduna State to the southeast, Katsina in the east, Sokoto and Kebbi in the north, and Niger in the southwest. The necessity for this research stems from the need to identify, process, and add value to Anka copper ore to the acceptable quality needed for industrial application.

MATERIALS AND METHODS

The materials used include a 5 kg crude sample of copper sourced from Anka (located in Zamfara State) having geological coordinates of latitude: 12°06.648'N and longitude: 5°56.443'E (Ogelekaa and Alaminiokumab 2020; Yahaya *et al*., 2022). The leachant used is Sulfuric acid (which is known in antiquity as oil of vitriol, which is a mineral acid composed of the elements sulfur, oxygen, and hydrogen, with the molecular formula H_2SO_4 and distilled water.

To prepare the sample for characterization, about 5 kg of the as-mined sample was first reduced to 50 mm size with a sledgehammer and charged into the Denver laboratory jaw crusher for further reduction to 5 mm. The sample was further reduced using the laboratory pulverizer machine after which thorough mixing was carried out to obtain a homogenized sample. 1kg of the homogenized sample was charged into the ball mill and was milled for 1 hour. The prepared sample was placed in the sample cup of the ED-XRFS instrument, ensuring that it was properly aligned and centred in the X-ray beam. The instrument was turned on, and the analysis was started following the instructions of the manufacturer. The analysis was allowed to run until it was completed. The software provided by the manufacturer was used to analyze the data obtained from the ED-XRF analysis. The software provided a readout of the chemical composition of the sample in terms of elemental concentration and relative abundance.

For the mineralogical characterization, the prepared powder was loaded onto a sample holder and the powder was evenly distributed across the surface and air gaps/uneven packing of the powder was avoided on the X-ray diffractometer. The X-ray diffractometer used in this study was capable of generating Cu-Kα radiation. The diffraction patterns were collected over a range of 5-70° 2θ with a step size of 0.02° 2θ and a counting time of 1 second per step. The X-ray beam was directed onto the sample, and the diffracted radiation was measured. The diffraction pattern was then recorded.

Further mineralogical characterization of the crude sample was performed using SEM/EDX. To do this, a small portion of the dried sample was placed on carbon tape and mounted on an SEM sample holder. The sample was then inserted into the SEM and imaging was performed at 500 magnifications to obtain high-resolution images of the mineral particles present in the sample. The SEM was operated at a low accelerating voltage to minimize damage to the sample and to obtain high-resolution images with high surface sensitivity. EDX analysis was performed on selected mineral particles to determine their elemental composition. The electron beam from the SEM interacts with the sample and generates X-rays, which are detected by the EDX detector. The fractional sieve analysis technique was adopted to ascertain the particle size distribution and the liberation size of the mineral. The set of sieves used for the analysis was properly cleaned to avoid contamination of the mineral sample. The sieves were arranged in conformity with the √2 series ranging from 1000-63 µm, as recommended (Wills and Napier-Mum 2006). A hundred (100) grams of the milled mineral sample was weighed and charged into the upper sieve (1000 µm). An automated sieve shaker was used to agitate the sample for 15 minutes. The process causes the undersized mineral particles to fall through successive sieves until they are retained on a sieve having an aperture lesser than the particle diameter. The particles retained on each sieve were weighed to determine the weight percentage of particles within each size range.

To conduct the leaching operation, 200 ml of 98.08% concentrated sulfuric (H_2SO_4) acid was charged into a beaker, followed by the addition of 100g of well-homogenized copper ore fines of (-125+90 µm) size. The mixture was then stirred vigorously using a magnetic stirrer for one hour and left for 24 hours to allow for complete leaching of the copper from the ore. After 24 hours, the solution was diluted with distilled water and filtered. The residue and the filtrate (pregnant solution) were then analyzed to determine the effectiveness of the leaching process (Aigul *et al*., 2023).

RESULTS

Chemical Composition of the Anka Copper Ore

Table 1 shows the results obtained from analyzing crude Anka copper ore using Energy Dispersive X-ray Fluorescence Spectrometer (ED-XRF). The analysis revealed that the ore contains various chemical elements in different percentages. These include SiO_2 at 11.48%, V_2O_5 at 0.054%, Cr_2O_3 at 0.009%, MnO at 0.092%, Fe_2O_3 at 30.042%, CO_3O_4 at 0.144%, NiO at 0.251%, CuO at 42.271%, Nb_2O_3 at 2.128%, WO_3 at 0.070%, P_2O_5 at 0.000%, SO_2 at 3.426%, CaO at 0.660%, MgO at 0.000%, K_2O at 0.375%, BaO at 0.137%, Al_2O_3 at 6.211%, Ta_2O_5 at 0.025%, TiO_2 at 0.067%, ZnO at 0.155%, Cl at 0.736%, ZrO2 at 0.706%, SnO_2 at 0.108%, PbO at 0.274%, HfO_2 at 0.468%, As_2O_5 at 0.000%, Bi_2O_5 at 0.072%, and Ce_2O_5 at 0.042%, as well as other trace compounds. It can be concluded from the result that the ore primarily contains copper, iron, sulfur, lead, and silicon in oxide forms and it exceeds the standard cut-off grade of 0.5% for mining copper minerals (Wills and Napier-Munn, 2006).

Table 1: Chemical Composition of Crude-Sample of Anka Copper Ore

Comp	SiO_2	Fe_2O_3	CuO	Nb_2O_3	SO_2	CaO	K_2O	BaO	Al_2O3	ZrO_2
%Conc	11.475	30.042	42.271	2.128	3.426	0.660	0.375	0.137	6.211	0.706

Mineralogical Characteristics of the Ore

The XRD pattern of the crude sample of Anka copper ore is depicted in Figure 1, it was observed that the ore is mainly composed of Cu-carbonate hydroxide in the form of malachite mineral (Cuz(CO,(OH)2)), which is almost pure. Additionally, the analysis identified traces of sodium aluminium silicate (NaZO.Al2O; SiO2) and two forms of silica (SiO), quartz, and cristobalite.

These suggest that copper in the Anka deposit occurs primarily as malachite and associated minerals, highlighting the need for ore beneficiation to recover the copper mineral. Plate 1 displays the SEM images of the crude sample of Anka copper ore taken at 500 magnification, while Table 2 and Figure 2 present the EDX analysis of the ore and the identified mineral grains, respectively. The micrograph shows that the ore contains dispersed grains of sodium aluminium silicate and copper carbonate hydroxide in a silica-dominated matrix. The EDX analysis confirmed the presence of copper (20.2%) within the ore matrix. The absence of Fe or Ca-bearing phases in the XRD result may be due to their concentration being below the detection limit of the XRD (2% by volume) or, as suggested by semi-quantitative EDX analyses, the Cu-carbonate contains traces of Fe. The Ca may exist at low concentrations in the NaAl-silicate, and the XRD result indicates that this phase corresponds to the mineral albite (NaAlSi3O8), which can contain traces of Ca.

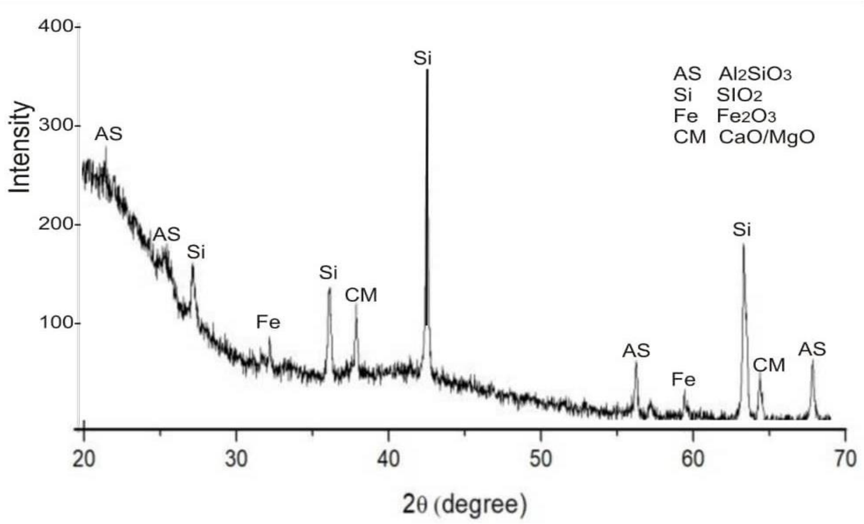

Figure 1: XRD Pattern of Crude Sample of Anka Copper Ore

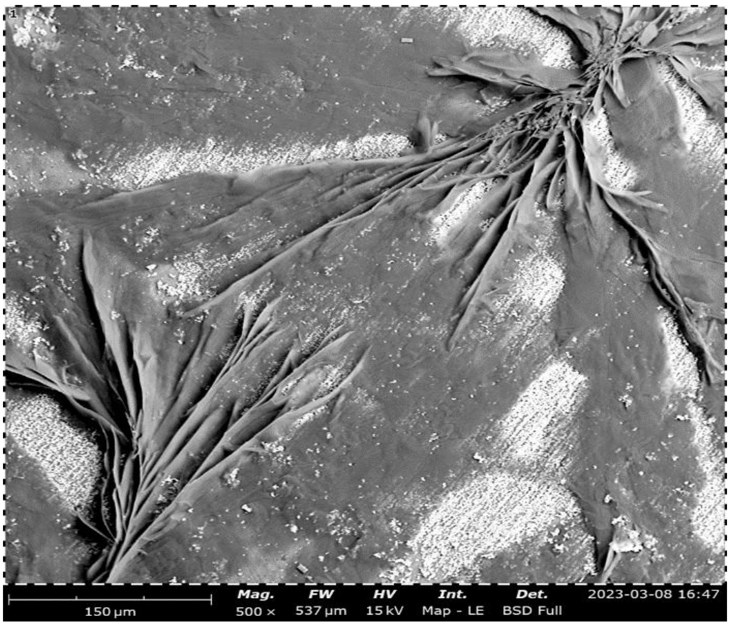

Plate 1: SEM Images of Crude Sample of Anka Copper Ore at 500 magnification using back-scattered electron

Table 2: EDX Analysis of the Crude Sample of Anka Copper Ore

Element Number	Element Symbol	Atomic Conc.	Weight Conc.
8	O	72.90	52.79
7	N	9.34	5.92
16	S	6.81	9.88
29	Cu	4.97	14.30
35	Br	4.51	16.32
6	C	1.46	0.80

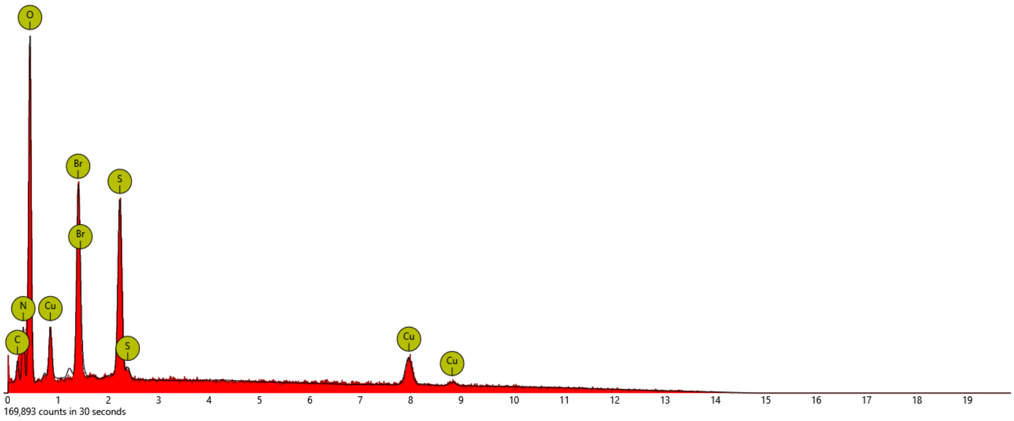

Figure 2: EDX Analysis of Identified Minerals in Anka Copper Ore Matrix

Results of Sieve Analysis

Table 3 presents the results of the fractional sieve size analysis of the crude sample of Anka copper ore, while Figure 3 shows a graph of the percentage cumulative weight retained and passing against sieve size. Based on the table, approximately 50% of the ore particles were retained at (-125+90 µm) sieve size, which is where the curves of % cumulative weight retained and passing intersect in Figure 3. The plot illustrates that the two curves are identical mirror images, indicating a direct correlation between % cumulative weight retained and passing. Wills and Napier-Mum (2006) proposed that the economic liberation size for a specific ore is the size at which 50% retention or passing is achieved. The weight of CuO (45.26%) from the compositional weight analysis also confirms -125+90 µm as the liberation size of Anka copper ore.

Output of Leaching Process

The process of acid leaching entails the utilization of sulfuric acid to dissolve the copper ions from the ore, resulting in a solution rich in copper that can be further treated to obtain pure copper. The efficiency of the acid-leaching procedure for copper is influenced by the concentration and potency of the acid, the size of the ore particles, the temperature and pressure during the reaction, and the duration of the ore's exposure to the acid. It continues to be a widely used and efficient technique for the processing of copper ore (Apua and Madiba, 2021; Aigul et al., 2023).

Table 3: Particle Size Analysis of Crude Sample of Anka Copper Ore

Sieve Range (µm)	Weight Retained (g)	% Weight Retained	% Cumulative Weight Retained	% Cumulative Weight Passing	% Weight of CuO
+1000	0.11	0.1132	0.113	99.889	15.52
-1000+710	0.06	0.0630	0.176	99.824	17.31
-710+500	0.88	0.8984	1.075	98.926	30.42
-500+355	1.70	1.7365	2.811	97.189	31.04
-355+250	7.10	7.2784	10.090	89.911	33.42
-250+180	5.82	5.9584	16.048	83.952	35.26
-180+125	15.07	15.4436	31.492	68.509	42.05
-125+90	14.56	14.9170	46.409	53.592	45.26
-90+63	17.36	17.7804	64.189	35.811	40.14
-63+Pan	34.96	35.957	100.00	0.00	33.46

Table 4 presents the residue and pregnant solution obtained after acid leaching of Anka copper ore. It was revealed that when the ore was subjected to acid leaching using 98.08% concentrated H2SO4, a pregnant solution with 52.51% CuO from the crude with 42.27% CuO and a residue with 4.69% CuO was obtained. This indicates that H2SO4 acid leaching can be an effective method for beneficiating copper from the ore (Koizhanova et al., 2023). Further chemical composition analysis of the pregnant solution using XRF revealed that it contains predominantly 52.51% CuO, 22.41% SO3, and 11.72% CaO. This composition indicates that acid leaching can also be used to separate the iron-bearing ore from the copper ore, thereby enhancing the copper recovery process. The results indicate that the Anka copper ore can be beneficiated via acid leaching using H2SO4. The technique was able to recover copper from the ore and also separate iron-bearing ore from the copper ore.

Table 4: Results on the Residue and Pregnant Solution Obtained After Acid Leaching of Anka Copper Ore

Comp	SiO_2	Fe_2O_3	NiO	CuO	Nb_2O_3	SO3	CaO	TiO2	Cl
Residue Conc. %	25.94	22.70	0.60	4.69	0.01	32.47	0.40	0.21	0.66
Pregnant Solution Conc. %	1.20	2.24	0.02	52.51	0.02	22.41	11.72	0.04	0.97

CONCLUSIONS

The characterization and acid-leaching beneficiation of Anka copper ore were studied. The following conclusions were deduced from the study. The energy dispersive x-ray fluorescence spectrometer yielded 42.271% CuO, and the x-ray diffractometer revealed that the ore is primarily constituted of Cu-carbonate hydroxide in the form of malachite minerals. The scanning electron microscope image indicates that the ore contains scattered grains of sodium aluminium silicate and copper carbonate hydroxide in a silica-rich matrix. The liberation size of the ore was -125+90 μm, as confirmed by sieve analysis, which means that particles larger than this size range are not economically viable to extract copper, while particles smaller than this size range can be extracted more efficiently. The ore was determined to be responsive to the H2SO4 acid leaching operation, which resulted in an enrichment of the feed from 42.27% Cu to a pregnant solution of 52.51% Cu, with a residue of 4.69% Cu.

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