

# CHARACTERIZATION OF AZARA COPPER ORE DEPOSIT, NASARAWA STATE, NIGERIA

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# CHARACTERIZATION OF AZARA COPPER ORE DEPOSIT, NASARAWA STATE, NIGERIA

**By**

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## Declaration page

I declare that the work in the Dissertation entitled: Characterization of Azara Copper Ore Deposit, Nasarawa State, Nigeria has been carried out by me in the Department of Metallurgical and Materials Engineering. The information derived from the literature has been duly acknowledged in the text and a list of references provided. No part of this project thesis was previously presented for another degree or diploma at this or any other institution.

| _____ | _____ | _____ |
|---|---|---|
| Name of Student | Signature | Date |

## Certification Page

This dissertation entitled: CHARACTERIZATION OF AZARA COPPER ORE DEPOSIT, NASARAWA STATE, NIGERIA by Oyebola AkanniWahab OYELADUN meets the regulations governing the award of the degree of M Sc. Metallurgical and Materials Engineering of the Ahmadu Bello University, and is approved for its contribution to knowledge and literacy presentation.

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## Dedication

This work is dedicated to God Almighty and my beloved wife and lovely children: Praise, Miracle, Joseph and Phoebe – Bethel.

**Acknowledgements**

I give all glory, honour and adoration to God Almighty for making it possible for the completion of this course despite all the challenges.

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## Abstract

The characterization of Azara copper ore was carried out. The samples were collected from three different locations in Azara, Awe Local Government Area, Nasarawa State, Nigeria. The physical observation shows that the ore has metallic lustre, dark bronze with greenish stripe and irregular or uneven breakages. The results obtained from physical analysis of the ore reveals that it has hardness of 3.5 on Moh's hardness scale, the specific gravity of 4.40 and electrical conductivity of$8.096 \times 10^{-7}\Omega^{-1}m^{-1}$. The XRF analysis of the sample reveals that the ore contains18.07% Fe, 3.30% Cu, 0.78% Pb, 0.48% Mn, 1.43% Ca, 0.66%Sn, 0.03%Sr, 0.11%Zn, 0.03%Zr, 0.34%Ti, 0.02%Nb, 0.03%Sb, 0.03%Ag, 0.07%As, 19.62%Si and 1.66%S. The mineralogical analysis of copper ore using XRD reveals that the ore containsanatase, zincite, chalcopyrite, hopeite, hematite, goethite, calcite, tenrite and pyrite. The result of SEM/EDS analysis reveals that the copper bearing minerals are separated from other minerals in the ore by smooth grains boundaries and are not locked with other minerals, hence, making it easy to free the copper from other associated minerals by comminution. The particle size sieves analyses reveals that the assays of the copper mineral are as follows:1.85% Cu in $-355+250\mu m$, 0.43% Cu in $-250+180\mu m$, 2.35% Cu in $-180+125\mu m$, 1.9% Cu in $-125+90\mu m$, 6.53% Cuin $-90+50\mu m$ and 6.73% Cuin $-50\mu m$.The liberation size of the ore was found to be below$50\mu m$ and the work Index of the ore was determined using the modified Bond method (Barry and Bruce method) and found to be 12.989kWh/tonne and classified as type **B ore**with soft medium texture using the Mathurgrindability chart. Hence the Azara copper ore could be seen as another potential source of copper mineral that can be beneficiated and utilized for the production of copper metal and alloys for the nation metallurgical industry.

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# CHAPTER ONE

## 1.0. INTRODUCTION

### 1.1. Preamble

In view of the desire of Nigeria to become one of the twenty leading economies in the world, there is a need to diversify the economic base of the nation. The mineral sector has the capacity to increase the fortune and create more jobs, thereby, enhancing industrial growth. It has been established that Nigeria is endowed with mineral resources; both metallic and non-metallic. However, the nature of these mineral types in a deposit ought to be established in order to facilitate the downstream concentration processes and production of high grade concentrate for extraction and utilization in metallurgical industries. These processes are no doubt relative to the nature of the composition, for example copper's existence as chalcopyrite ($CuFeS_2$), chalcocite ($Cu_2S$), covellite (CuS), malachite ($CuCO_3.Cu(OH)_2$), cuprite ($Cu_2O$), etc. is a typical case (Encyclopedia Britannica, 2014).

The extraction of specific valuable minerals from their naturally occurring ores is variously termed as “ore dressing” “mineral dressing” and “minerals beneficiation”. For most metalliferrous ores produced by mining operations, beneficiation is an important intermediate step in the transformation of natural ore to pure metal.Therefore, it is imperative that the key to successful beneficiation of an ore is greatly attached to the amount of information available on the nature and properties of the various components making up the ore. In order to achieve this, adequate chemical and mineralogical studies of an ore must be carried out (Craig and Vaughan, 1981).

According to data compiled by the International Copper Study Group (ICSG) in 2012, global consumption of refined copper in 2011 rose by 2.7% (500,000 tonnes) to 19.9 million metric

tonnes (Mt) and was 9% more than the prerecession peak in 2008. While consumption in North America remained stagnant in 2011 and declined slightly in the European Union, consumption in Asia rose by 2.7%, principally owing to a 7% increase in China's apparent consumption, which accounted for about 40% of total global consumption (Daniel, 2013).

Furthermore, the justification by Yue (2009) indicated that, the sharp increase in copper consumption in the world due to the rapid development of Chinese economy makes it one of the most important materials that are widely needed for electronic and machine copper – alloyed component production and other industrial use. This has made the global demand for copper to growat an estimated rate of 3.3% per year (Yue, 2009)

### 1.2. Statement of the Problem

i. The price of copper cables, copper alloys based machine components and interior decors and copper air – conditions pipes are on the increase despite the nation copper ore deposits. This increase in the price of the copper metal based materials is squarely attributed to nation lack of established copper mining and processing plant that can serve as a feed to local metallurgical copper metal extraction industry.

**ii.** The recent pronouncement by the Government of the day that it will focus its attention on mining and agriculture has aggravated the search for ways to meet the Government proclamation, but this may be hindered by the lack of available data on the characterization of Azara copper ore which has also hampered the development of the process route of the ore and hence, without the characterization of the Azara copper ore deposit, the deposit cannot be explored, developed and exploited for economic purposes.

### 1.3. Justification of the Study

The demand for copper metal and its other associated forms by electrical manufacturing, metallurgical, pharmaceutical and agricultural industries cannot be over – emphasized. Hence, the need for developing countries like Nigeria to explore and exploit its vast copper ore deposits for technological advancement and earn more foreign exchange, create jobs for her teeming population. Therefore, characterization of Azara copper ore will provide the necessary data that will enhance the development of its process route, develop the appropriate technology for the beneficiation of the copper ore and through such activities the nation local engineers will acquire the necessary skills for the exploitation of the ore. Hence, the need for research of this nature – characterization of Azara copper ore – will also encourage the diversification of the nation's economy and create the enabling environment for private investors to invest and contribute to national development.

### 1.4. Aim and Objectives of the Study

The major aim of the research is to characterize Azara copper ore deposit in Nasarawa State, and the specific objectives are as follows:

i. to determine the physico-chemical properties (colour, hardness, relative density, electrical conductivity) and also the chemical and mineralogical compositions of the ore sample using XRF, XRD and SEM-EDS analytical equipment.
ii. to carry out the sieve size/assay analysis, liberation study and determine the work index ofthe ore sample.

### 1.5. Scope of the Study

This research involves sourcing the Azara copper ore sample, determination of chemical composition by XRF, determination of the physico - chemical properties, mineralogical characteristic using the XRD, SEM and petrological microscope, liberation size analysis, particle

size analysis and work index of the copper ore sample to determine the energy requirement for its comminution.

# CHAPTER TWO

## 2.0. LITERATURE REVIEW

### 2.1. Copper Ore Type and World Deposits

Copper occurs as native copper or in minerals such as the copper sulphides: chalcopyrite ($CuFeS_2$); and chalcocite (copper glance or copper sulphide) ($Cu_2S$);covellite (CuS); bornite ($Cu_5FeS_4$); enargite ($Cu_3AsS_4$); tetrahedrite ($(CuFe)_{12}Sb_4S_{13}$); copper carbonates: azurite or basic copper carbonate ($Cu_2(OH)_2CO_3$), and malachite ($Cu_2CO_3$); copper(I) oxide: mineral cuprite or copper oxide ($Cu_2O$); chrysocolla ($CuSiO_3$). Most copper is mined or extracted as copper sulphides from large open pit mines in porphyry copper deposits that contain 0.4 to 1.0% copper. Examples include Chuquicamata in Chile, Bingham Canyon Mine in Utah and El Chino Mine in New Mexico, United States. According to the British Geological Survey, in 2005, Chile was the top mine producer of copper with at least one-third world share followed by the United States, Indonesia and Peru (Hammond, 2004). Chalcopyrite is the most common mineral found in porphyry-type deposits, while chalcocite occurs predominantly in hydrothermal veins (Simons and Reston, 1973;Encyclopedia Britannica, 2014). Chalcopyrite is the copper ore deposit of Falum, Sweden of Namagaland in South Africa. However, vast crystals or deposits occur in Ishiagu, Ebonyi State; South Eastern region of Nigeria. High great chalcopyrite deposit can also be found in Baluba, East of Lusaka, Zambia(Alafara at el, 2012).

The minimum grade of ore that can be mined profitably from a deposit is termed the cut – off grade. The yield and tonnage of ore above the cut – off grade are critical both in estimating the ore reserve and determining mine profitability. Hence, cut – off grade is a function of the type of ore and mining operation. For the copper ore, the cut – off for open pit mine which has gold and silver associated mineral ranges from 0.4 to 1.0% copper; while underground deposit is considered to be 0.8% copper(Weiss, 1985; www.nevadacopper.com, 2015).

## 2.2. Some Nigerian Copper Ore Deposits

Nigerian copper ore is mostly found as complex ore alongside zinc and lead(Damisa, 2008). The Table below presents some of the Nigerian copper ore deposits and their locations.

**Table 2.1: Some Nigerian Copper Ore Deposits and their Locations**

| S/No | Deposit | Location | Estimated Reserve |
|---|---|---|---|
| 1 | Akiri | Nasarawa State | Inferred |
| 2 | Anka | Zamfara State | Inferred |
| 3 | Nahuta | Bauchi State | Inferred |
| 4 | Zarara Hill | Kano State | Inferred |
| 5 | Yelwa Rishi | Bauchi State | Inferred |
| 6 | Rafin Gabas | Nasarawa State | Inferred |
| 7 | Ishiagu | Ebonyi State | Inferred |
| 8 | Azara | Nasarawa State | Inferred |

Source: Obaje, 2009; Damisa, 2008; NGSA, 2013; Oyeladun et al, 2012; Alafaraet al, 2012.

## 2.3.Previous Work done on Some Nigerian Copper Ore Deposits

Damisa (2008),reported that Nahuta Lead – Zinc – Copper complex ore has 5.2% copper. Hence, there is need for the ore to be explored and beneficiated for its copper mineral.Oyeladun et al (2012), worked on the determination of chemical composition and work index of Rafin Gabas chalcopyrite ore. They reported that the ore contains 12.6%Cu and has a work index of 22.38 kWh/tonne.Abubakre et al (2014), worked on chemical and mineralogical analysis of Akiri copper ore and established that the ore contains2.5%Cu in form of malachite.Jatau et al (2014), worked on the physico – chemical properties of Agwada chalcopyrite ore and established that the ore contains 4.62%Cu while the physical analysis carried out revealed that the ore has a hardness of 3.5, specific gravity of 3.8 and colour of light brown, green with reddish brown

streaks. Ali et al (2014), worked on the determination of liberation size of Agwada chalcopyrite ore and established that the liberation size of the ore to be 125μm having the highest assay of 8.17% Cu.

However, from some of these works carried out on some Nigeria copper ore deposits, it is clear that some of the Nigeria copper ore deposits with geochemical history are mineable and profitable as their percent copper cut – off grades are slightly above the specific cut – off grade of 0.4 – 1.0%Cu (Weiss, 1985).

### 2.4. Mineral Processing Conceptual Parameters for the Development of a Process Route for a Newly Discovered Ore

The following parameters listed below are the standard procedures usually followed in the development of flow sheet for the beneficiation of a newly discovered ore deposit after all the mining and geological investigation have been carried out and the representative sample has been taken:

a) **Chemical Analysis of the Representative Sample of the Ore:** this involvesconducting full chemical analysis of the representative sample of the ore to establish the chemical composition, determine the grade of the ore, impurities associated with the ore and also confirm the result of geologists and mining engineers who had already or probably worked on the ore.

b) **Mineralogical Characteristics of the Ore:** this is carried out with the aim of establishing the major, minor and trace minerals in the ore, allocation of each element to each mineral, the grain size of each mineral in the ore, in particular, the mineral of economic importance and the degree of association of the valuable and gangue minerals.

This also serves as a good guide to the estimation of the beneficiation requirement (Weiss, 1985)

c) **Particle Size/Assay Analysis:** this is to establish the distribution of both the valuable and gangue minerals in the various size fractions.

d) **Liberation Studies:** this is to establish the degree of freedom of the valuable minerals; which helps in the economic mesh of grind to be established. The emphasis is placed on the grain size of valuable minerals and probably that of the gangue which may likely affect the separation process.

e) **Determination of the Work Index:** this is carried out in order to establish the energy requirement for comminution of the ore and selection of the appropriate comminution equipment.

f) **Concentration Test:** this is to establish the beneficiation route using any known mineral concentration techniques. The type of concentration technique to be used in the beneficiation of the ore will depend largely on the information obtained from a – e above.

From the information above, general ore testing will help in obtaining and maintaining maximum concentration grades and recoveries which are recognized throughout the mineral and metallurgical industries. (Yaro, 1997; Thomas and Yaro, 2007)

### 2.5. Mineral Processing Techniques

The two major mineral processing techniques after mining are as follows:

- Comminution Process
- Concentration Process

#### 2.5.1.Comminution Process

Comminution is a process whereby particulate materials are reduced by blasting, crushing and grinding to produce sizes required for downstream processing or end use. In mineral processing, comminution operations are used to ensure that valuable constituents are physically liberated from the waste constituents (gangue) before physical or chemical separations are attempted (Fuerstenau and Han, 2003).

The energy used in comminution operation is up to about 50% of a mineral processing plants operating cost, this carries a larger percentage of capital cost price for a plant. This is one of the greatest challenges faced by mineral engineer. In U.S.A, 15 billion kWh per year is used for blasting, crushing and grinding materials of all types. Copper ore and iron ore consume up to about 60% of the total energy (Fuerstenau and Han, 2003).

The comminution process in mineral processing is the crushing and grinding of ores in order to liberate valuable minerals from the ore matrix, to increase surface area for high reactivity and to facilitate the operations. Size reduction begins at the mine/quarry after blasting (Fuerstenau and Han, 2003).

### A. Crushing

Crushing involves the size reduction of large particle, typically from the run – of – mine material down to about 6mm. This is first stage of size reduction in the plant. Crushing is a dry operation that can be performed either in two or three stage: primary, secondary, and tertiary crushing. This can be done either in open or closed circuit. Primary crushers are used to reduce run – of – mine ore down to a size suitable from transportation and it can become feed for crushers. Various equipment are used in size reduction i. e. comminution devices. Comminution devices include the following:

- Jaw Crusher

- Gyratory Crusher
- Cone Crusher
- Impact Roll
- Rotary Crusher

### B. Grinding

The next stage of size reduction for mineral processing operation is grinding. Grinding is a powdering or pulverizing process using the rock mechanical forces of impact, compression, shearing and attrition. Grinding is done primarily using rotating mill such as ball mills, rod mills, autogenous (AG) mill and semi – autogenous (SAG) mills etc. This can be done in either open or closed circuit, either wet or dry, depending on the device and application. Grinding methods can be by tumbling, stirring or vibration. The two main purposes for a grinding process are:

- to liberate individual mineral locked in ore for a subsequent separation
- to produce fine from mineral fraction by increasing the specific surface area

It is possible to calculate the specific energy required to grind from one size to another using one of the grinding relationships (eg) Bond's work index (Wills and Napier-Munn, 2006).

### C. Comminution Theory

Comminution technology can be described with respect to both theory and practice. Various theories have been propounded concerning the relationship between energy input and the produced particle size produced from a given feed size. The average energy of current comminution device is somehow less than 5%, because most of energy input to comminution is absorbed by machines and only small proportion of energy is really used in the breakage of ore particle.

The oldest theory is that of Von Rittinger (1867) in Wills and Napier-Munn (2006) which states that the energy consumed in the size reduction is proportional to the area of new surface

produced. The surface area of a known weight particle of uniform diameter is inversely proportional to diameter, hence Rittinger's law gives $E \alpha A; A \alpha \frac{1}{D}$

$$E = k\left(\frac{1}{D_2} - \frac{1}{D_1}\right) \quad \ldots (2.1)$$

*Where* $E =$ *the energy input*

$D_1 =$ *the initial particle size*

$D_2 =$ *the final particle size*

$k =$ *constant*

The second theory is that of Kick (1885) in Wills and Napier-Munn (2006) which stated that work required is proportional to the reduction in volume of the particle concerned. According to Kick's law, the energy required for comminution is proportional to $\log R/\log 2$

$$\text{(ie)}E = k(\log R/\log 2) \quad \ldots (2.2)$$

Where R = reduction ratio =f/p

f = diameter of the feed particles and

p = diameter of the product particles

Bond (1952) developed an equation which is based on the theory that work input is proportional to the new crack tip length produced in particle breakage and equals the work represented by the product minus that represented by feed. In particle of similar shape, the surface area of unit volume of material is inversely proportional to the diameter. The crack length in volume is considered to be proportional to one side of that area and therefore inversely proportional to the square root of the diameter.

The single – point energy – size relationships developed during the time is generated of the form:

$$\frac{dE}{dD} = -C(D)^{-\alpha} \quad \ldots (2.3)$$

Where for Rittinger ($\alpha = 2$), for Kick ($\alpha = 1$), for Bond ($\alpha = 1.5$), and Charles ($\alpha \geq 1$)

The Bond relationship has the integrated form given as follows:

$$E = \frac{k}{0.5}\left(\frac{1}{\sqrt{d_p}} - \frac{1}{\sqrt{d_f}}\right) \quad \ldots (2.4)$$

Where E = Energy required in size reduction

$k$ = constant and a function of ore characteristics

$d_p$ = the diameter in micron of the product (product particle size)

$d_f$ = the diameter in micron of the feed (feed particle size)

For practical calculations the size in microns which 80% passes is selected as the criterion of particle size. Hence, Bond's theory equation is expressed as followed:

$$W = 10W_i\left(\frac{1}{\sqrt{P_{80}}} - \frac{1}{\sqrt{F_{80}}}\right) \quad \ldots (2.5)$$

Where$F_{80}$ = the diameter in microns which 80% of the feed passes, $P_{80}$ =the diameter in microns which 80% of the product passes and $W_i$ = the work index (Wills and Napier-Munn, 2006).

### 2.5.2. Size Analysis

Size Analysis is to establish the distribution of both the valuable and gangue minerals in the various size fractions. This is very important in order to determine the quality of grinding and in establishing the degree of liberation of the values from the gangue at various particle sizes. In the separation stage, size analysis of the products is used to determine the optimum size of the feed to the process for maximum efficiency and to determine the size range at which any losses are occurring in the plant, so they may be reduced.

To achieve accurate and reliable result, sieve analysis can be employed for particle size analysis. Sieve analysis is one of the oldest methods of size analysis and is accomplished by passing a known weight of sample of ore through successive finer sieves and amount of sample collected on each is weighed. Each sieve weight is used to determine the percentage weight. Sieving is carried out with wet or dry materials and the sieves are usually agitated in order to expose the entire particle to the openings in the sieves at given time. Effective sieving depends on the amount of charge and agitation.

The process of sieving may be divided into two stages:

i. The elimination of particles considerably smaller than the screen apertures, which should occur fairly rapidly
ii. The separation of near size particles, which is a gradual process rarely, reaching final completion.

The choice of sieve sizes is essential since the apertures of consecutive sieve have a constant relationship to each other. According to Wills and Napier-Munn (2006), a useful scale is one in which the ratio of the aperture widths of adjacent sieves is the square root of 2( $\sqrt{2}$ = 1.414) (Wills and Napier-Munn, 2006). The advantage of this scale is the aperture areas double at each sieve, facilitating graphical representation of the result.

In the industry, classifier is installed to effect the separation of the various particle sizes. Classification refers to sizing operations that exploit the differences in settling velocities exhibited by particles size. Classification equipment may include ore sorters, gas cyclones, hydro - cyclones, rotating trammels, rake classifiers or fluidized classifiers (Gupta and Yan, 2006).

### 2.5.3. Liberation Size

The two fundamental operations in mineral processing are release or liberation or freeing of the valuable minerals from waste gangue minerals from each other and concentration, which involves separation of the liberated product. Comminution is the cause and liberation is its effect (Gupta and Yan, 2006). Liberation study is to establish liberation size of the valuable mineral from its gangues and mesh of grind. The physical liberation of the valuable mineral particles is achieved by the tendency of the ore to fracture along the interfaces (boundaries) thereby separating the mineral and the gangue (Mills, 2014).

It is important to bear in mind that the grain size distribution of a mineral determines, to a considerable extent, the ease with which that mineral can be liberated from its rocky matrix. Minerals occurring as large grains are liberated at comparatively large sizes; the liberated process involved will be comparatively cheap (Mills, 2014).

**2.5.4. Work Index**

Work index is the comminution parameter which expresses the resistance of the material to crushing and grinding; numerically it is the kilowatt hours per tonne required to reduce the material from theoretically infinite feed size to 80% passing 100μm. The determination of work index of an ore will help in calculating the energy requirement for comminution of the ore and selection of appropriate comminution equipment. The Bond work index of material is determined by ball mill grindability test (Bond, 1961) in Wills and Napier-Munn (2006).

Berry and Bruce (1966) developed a comparative method of determining the work index of an ore known as Modified Bond's Method. The method requires the use of a reference ore of known work index. Oyeladun et al (2012) adopted the comparative grindability test for the determination of the work index of Rafin Gabas chalcopyrite ore in Nigeria. The reference ore is ground for required time and the power consumption is recorded. An identical weight of test ore

is ground for the same period of time and the same power is consumed. Then the mathematically Bond's equation of the work index of an ore can be determined by comparing the work index and can be expressed as follows:

$$W_r = W_t = W_{ir}\left[\frac{10}{\sqrt{P_r}} - \frac{10}{\sqrt{F_r}}\right] = W_{it}\left[\frac{10}{\sqrt{P_t}} - \frac{10}{\sqrt{F_t}}\right] \ldots (2.6)$$

$$\text{hence}, W_{it} = W_{ir}\left[\frac{10}{\sqrt{P_r}} - \frac{10}{\sqrt{F_r}}\right] \Big/ \left[\frac{10}{\sqrt{P_t}} - \frac{10}{\sqrt{F_t}}\right] \ldots (2.7)$$

Where $W_r$ = Work input of reference ore; $W_t$ = Work input of test ore

$W_{ir}$ = Work index of reference; $W_{it}$ = Work index of test

$P_r$ = 80% of Product(reference ore)passes; $P_t$ = 80% of Product(test ore)passes;

$F_r$ = 80% of feed (reference ore)passes; $F_t$ = 80% of feed (test ore)passes

**Table 2.2: Work Indices of Some Mineral with their Physical Properties**

| S/ No | Minerals | Formula | Appearance | Hardness | Specific Gravity | Work Index (kWh/tonne) |
|---|---|---|---|---|---|---|
| 1. | Kaolinite | $Al_2(Si_2O_5)(OH)_4$ | Rarely seen fracture, dull, white | 2 – 2.5 | 2.6 | 7.10 |
| 2. | Chalcopyrite | $CuFeS_2$ | Uneven fracture, metallic, brass – yellow with greenish black | 3.5 – 4.0 | 4.1 – 4.3 | 13.3 and 23.38 |
| 3. | Pyrite | $FeS_2$ | Subconchoidal fracture, metallic, brass – yellow | 6.0 – 6.5 | 5.1 | 8.90 |
| 4. | Zincite | ZnO | Subconchoidal fracture, metallic, orange – yellow | 4.0 – 4.5 | 5.4 – 5.7 | 12.74 |
| 5. | Granite | – | | | 2.68 | 15.14 |

| | | | | | | |
|---|---|---|---|---|---|---|
| 6. | Hamentite | $Fe_2O_3$ | Subconchoidal fracture, submetallic, red – brown | 5.5 – 6.5 | 4.9 – 5.3 | 12.68 |
| 7. | Covellite | CuS | Subconchoidal fracture, submetallic, red – brown | 1.5 – 2.0 | 4.6 | 14.03 |
| 8. | Cassiterite | $SnO_2$ | Subconchoidal fracture, adamanite, red – brown | 6.0 – 7.0 | 7 | 11.20 |
| 9. | Rutile | $TiO_2$ | Subconchoidal fracture, submetallic, red – brown | 5.5 – 6.0 | 3.9 | 11.90 |
| 10. | Cuprite | $Cu_2O$ | Conchoidal fracture, submetallic, red – brown | 3.5 – 4.0 | 5.9 – 6.1 | 13.3 and 23.38 |
| 11. | Malachite | $Cu_2CO_3(OH)_2$ | Subconchoidal fracture, adamanite, bright – green | 3.5 – 4.0 | 3.7 – 4.0 | 13.3 and 23.38 |

Source: Fuerstenau and Han (2003), Oyeladun et al (2012) and Perkins (2007).

### 2.6 Concentration Processes

Physico – chemical properties of minerals and other solids are used in concentration operations. These properties include: density, magnetic susceptibility, electrical conductivity, surface properties and etc. Solid – solid separations are based on particle properties, this includes:

- Ore sorting
- Gravity concentration
- Froth flotation
- Magnetic and Electrostatic concentration

#### 2.6.1 Ore sorting

The beneficiation of an ore by sorting involves inspection and recognition of worth of each particle followed by separation into either a valuable or worthless fraction. Hand sorting is oldest

method of sorting. The success of ore sorting depends on valuable mineral essentially liberated from the gangue at coarse sizes and identifiable by a detector within the residence time. The ore sorters are classified on the basis of the type of detection system used, which include: conductivity, optical and photometric, radiometric and X – ray.

This method of solid – solid separation for minerals utilizes colour differences in some ores detected by naked eye. In some cases, electro – optic detectors collect data on the responses of minerals when exposed to infrared light, visible light, ultraviolet light, laser light and gamma radiation can be used in identifying and sorting out minerals. Equipment such as photometric and radiometric can be used in sorting out of minerals (Weiss, 1985).

**Table 2.3: Ore sorter sensor type**

| Sensor | Mineral Property | Application |
|---|---|---|
| Optical | Reflectance, fluorescence, transparency | Base metals, precious metals, industrial minerals, diamonds |
| Photometric | Monochromatic reflection, absorption | Industrial minerals, diamonds |
| Infra – red | Reflection, absorption, heat conductivity – dissipation. | Base metals, precious metals, industrial minerals |
| Radiometric | Natural or induced gamma radiation | Uranium, precious metals |
| X – ray | Atomic density – transparency, fluorescence | Base metals, precious metals, coal, diamonds |
| Electromagnetic | Conductivity | Base metals |

Source: The Australasian Institute of Mining and Metallurgy(2012)

### 2.6.2 Gravity concentration

This method uses differences in the density of the minerals to effect separation of one mineral from another. The separation by gravity can be categorized into two methods:

- Separation in water (gravity concentration)
- Separation in a heavy medium (Dense Media Separation)

Equipment used in separation in water are jig, shaking table and spirals.

In jigging, there are two actions; one is the effect of hindered settling, meaning that a heavier particle will settle faster than a light particle. The other one is the separation process in an upward flow of water which separates the particles by their density (Moore, 1990; Wills and Napier-Munn, 2006).

### 2.6.3 Froth Flotation

Froth Flotation is a process for making a solid – solid separation based on differences in the surface properties of minerals (composition and electrical charge). A froth is formed by introducing air into a fine particle slurry (pulp) containing a frothing (foaming) agent and other reagent inside the flotation cell. Particles with air affinity for air bubble (hydrophobic) are carried by the bubbles to the surface where they are removed by scrapping. The reagents available include:

- Collectors: Reagent used to coat the desired mineral with a hydrophobic coating and allows bubble to attach to the surface of the mineral.
- Frothers: Reagent used to create a stable froth, stiff enough to facilitate mineral separation, but not break so as to facilitate subsequent handling.

- Modifiers: Reagent used to ensure that the desired mineral float and the undesired mineral do not float. These include activator, depressant, pH modifying agents, blocking agents, etc. (Moore, 1990; Wills and Napier-Munn, 2006).

### 2.6.4 Magnetic Separation and Electrostatic Separation

**A. Magnetic Separation**

Magnetic concentration depends on the differences in magnetic susceptibility of minerals utilized in operation. All minerals can be classified into three groups based on their magnetic properties, namely; ferromagnetic, paramagnetic and diamagnetic properties. Paramagnetic minerals exhibit positive magnetism, hereby having a greater magnetic field flux. Diamagnetic minerals exhibit negative magnetism, hereby having a lower magnetic field flux. Ferromagnetic minerals are very strong paramagnetic mineral such as magnetite. The higher the magnetic susceptibility, the higher is the field density in the particle and greater is the attraction up the field gradient towards increasing field strength. Magnetic separation depends on a particle magnetic susceptibility in a magnetic field. The force, F acting on a particle is proportional to the product of the field strength and the field gradient$\frac{dH}{dl}$

$$\text{ie} F \ \alpha \ H \frac{dH}{dl} \ldots (2.8)$$

Where F is the force on the particle, H is the field intensity, and dH/dl is the field gradient.

Magnetic separator can be broadly classified into categories, namely:

- Low intensity: this is the separator used for ferromagnetic minerals such as magnetite.
- High intensity: this is the separator usedfor paramagnetic minerals.
- High gradient separator
- Superconducting magnetic separator.

**B. Electrostatic Separation**

Every mineral conducts or takes an electrical charge on its surface if brought into contact with a source charged to a sufficiently high electrostatic potential. Electrostatic separation applies to only a small number of minerals.

Electrostatic separation depends on differences in the electrical conductivity of minerals and electric field strength required. In Tribostatic separator, the mineral particles are charged by exposing it to high electrostatic potential. Some will take on a positive charge and others a negative charge. These charged particles then pass between charged plates with the positively charged particles attracted toward a negatively charged plate and the negatively charged particles attracted toward the positively charged plate. Tribostatic separation is only effective when the particles are dry and at temperatures greater than $100 - 110^0$C (Fuerstenau and Han, 2003).

**Table 2.4: Some Minerals with their Magnetic Susceptibilities and Electrostatic Properties**

| Mineral | (Dimensionless SI unit x $10^{-6}$) | Magnetic Susceptibility | Field Strength (Gauss, G) | Electrostatic Response |
|---|---|---|---|---|
| Cassiterite | 1100 | Diamagnetic | | Conductor |
| Chalcopyrite | 23 – 400 | Paramagnetic | | Conductor |
| Chromite | 3000 – 120000 | Paramagnetic | 10000 – 16000 | Conductor |
| Galena | – 33 | Diamagnetic | | Conductor |
| Biotite | 1500 – 2900 | Paramagnetic | 10000 – 18000 | Non – conductor |
| Geoethite | 1100 – 12000 | Paramagnetic | | Non – conductor |
| Hematite | 500 – 40000 | Paramagnetic | 13000 – 18000 | Conductor |
| Limenite | 2200 – 3800000 | Paramagnetic | 8000 – 16000 | Conductor |
| Magnetite | 1000000 – 5700000 | Ferromagnetic | 1000 | Conductor |
| Pyrite | 35 – 5000 | Paramagnetic | - | Conductor |
| Pyrrhotite | 460 – 3200000 | Ferromagnetic | 1000 – 4000 | Conductor |
| Siderite | 1300 – 11000 | Paramagnetic | 10000 – 18000 | Non – conductor |
| Sphalerite | – 31 – 750 | Diamagnetic | - | Conductor |
| Monazite | | Paramagnetic | | Conductor |
| Malachite | | Paramagnetic | | Conductor |
| Bornite | | Paramagnetic | | Conductor |
| Gypsum | | Diamagnetic | | Conductor |
| Rutile | | Paramagnetic | | Conductor |

Source: Hunt, Maskowitz and Banerjee, (1995); Outotec, (2011); Metso Minerals, (2006).

## 2.7. Analytical Methods Used in Metallurgical Analysis and Characterization

The chemical composition of a mineral is of fundamental importance because its properties greatly depend on it. Such properties, however, are determined not only by the chemical composition but also by the geometry of the constituent atoms and ions and by the nature of the electrical forces that bind them. Thus, for a complete understanding of minerals, their internal structure, chemistry, and bond types must be considered.

Various analytical techniques may be employed to obtain the chemical composition of a mineral. Quantitative chemical analyses conducted prior to 1947 mainly utilized so-called wet analytical methods, in which the mineral sample is first dissolved. Various compounds are then precipitated from the solution, which are weighed to obtain a gravimetric analysis. Since 1947 a number of analytical procedures have been introduced that provide faster but somewhat less accurate results. Most analyses performed recently have made use of instrumental methods such as optical emission, X-ray fluorescence, X-ray diffraction, and scanning electron microscopy analysis. Relatively well-established error ranges have been documented for these methods, and samples must be prepared in a specific manner for each technique (www.facweb.bhc.edu, 2014).

Production of a high copper ore requires a thorough knowledge of mineralogical parameters that may impact beneficiation. This is particularly important in choosing the beneficiation process given that mineralogical parameters significantly influence the efficiency of concentration process. Microscopic analysis of polished section of a sample will reveal the petrography of the rock. This is basically a topographic technique and similar to metallographic analysis. This analysis gives useful information on the identification of the rock, mineral and ore and also helps in the characterization of the ore. The mineralogical analysis can be done by using microscope with in – built camera, scanning electron microscopy (SEM) and X – ray diffractometer (XRD).

### 2.7.1. Microscopic Properties of Mineral and the Petrographic Microscope

Minerals are categorized as opaque (that will not transmit light) and non – opaque (that will transmit light) and non – opaque can either be isotropic or anisotropic. Isotropic have the same optical properties in all directions, while anisotropic have different optical properties in different directions. The anisotropic minerals are divided into according to whether they are uniaxial or biaxial and according to whether they have a positive or negative optic sign (Perkins, 2007).

Light interacts differently with anisotropic minerals depending on the light vibration direction relative to planes of atoms in the mineral structure. Petrographic microscope have rotating stage, so that the sample relative to polarizer(Klein and Doutrow, 2008).

A polarizing microscope, also call petrographic microscope, is a special microscope that uses polarized light for investigating the optical properties of specimen. There are two types of polarizing microscope: transmitted light model and incident light model. Similar to typical microscope, a polarizing microscope have the following added features: a polarizing condenser that includes a polarizer, a rotating stage that allows the position of the specimen to be set a strain – free objective for polarized light, a center able revolving nosepiece that allows optical axis adjustment for the objective, analyzer, a Bertrand lens for observing the pupil of the objective, a test plate, a compensator, diaphragm and an eyepiece with crosshair. An incident light polarizing microscope is used for the observation of metallic and opaque crystals. Polarizing microscope can be used with or without upper polarizer inserted. Without the upper polarizer inserted, the sample is observed in the plane polarized light, while with the upper polarizer inserted, the sample is observed in the crossed polarized light. Grain size, shape, colour, cleavage and optical properties such as refractive index and pleochroism are revealed in plane polarizer light, while crossed polarized light is used to determine properties such as retardation, optical sign and 2V (Perkins, 2007).

Light waves are transverse: that is, the vibrating electric vector associated with each wave is perpendicular to the direction of propagation Transverse wave light whose vibrations possess direction is called polarized light. There are two major types of polarized light: plane polarized light and cross polarized light. The specimen observed using plane polarized light produces relief. Relief is determined by the difference between the refractive index of the mineral and the

refractive index of its surroundings. The refractive index (n) of the mineral can be determined as follows:

$$n = \frac{v_v}{v_m} \qquad \ldots (2.9)$$

Where $v_v$ = velocity in the vacuum and $v_m$ = velocity in the mineral

The refractive index of the mineral is mostly between 1.5 and 2.0.

However, under cross polarized light mineral observed viewed between two perpendicular polarized filters: the polarizer below the sample which the analyzer above the sample inert using rod. When an isotropic substance is viewed in cross – polarized light, it appears dark due to the double refraction that happens. When polarized light enters anisotropic mineral, it is split into two rays which vibrate perpendicular to each other. All anisotropic minerals go extinct (black) four times as the stage rotates $360^0$ due to two perpendicular allowed vibration directions are parallel to polarizer and analyzer.

Table 2.5: Optical Classification of Some Minerals

| Opacity | Isotropy | Optical Class | Optic Sign | Examples of Common Mineral |
|---|---|---|---|---|
| Opaque | | | | Gold, copper, pyrite, pyrrhotite, magnetite, ilmenite |
| Non - opaque | Isotropic | | | Garnet, diamond, halite, fluorite, periclase, spinel. |
| | Anisotropic | Uniaxial | + | Quartz, zircon, brucite, rutile, leucite |
| | | | – | Apatite, calcite, dolomite, beryl, tourmaline, corundum, nepheline |
| | | Biaxial | + | Enstatite, diopside, sillimanite, gysum, plagioclase, barite |

| | | | | |
|---|---|---|---|---|
| | | | – | K – feldspar, muscovite, hornblende, plagioclase, fayalite, epidote |

Source: Perkins, 2007.

### 2.7.2. X-Ray Fluorescence (XRF)

X – ray fluorescence (XRF) analysis is a powerful analytical tool for spectrochemical determination of almost all the elements present in a sample. The sample is irradiated with polychromatic X – ray generated in a high intensity X – ray tube (Beckhoff et al, 2006). These incidents X – ray from the X – ray tube are, to considerable extent, absorbed according to Beer's law:

$$\log \frac{I_0}{I} = K_d \Delta_d \qquad \ldots 2.10$$

Where $I_0$ is the incident X – ray intensity; I is the intensity of the X – ray beam that was not absorbed in the sample; $k_d$ is a proportionality constant and $\Delta_d$ is the thickness of the sample.

The X – ray energy that is absorbed in the sample results in the generation of an X – ray emission spectrum that is characteristic for each element in the sample. In the process of the absorption of X – ray energy in the sample, electrons are dislodged from the innermost shells (K, L, M …). The prompt transition of outer shell electron (ie next outer shell eg L – shell) to replace an expelled electron (eg K shell) creates a new vacancy, which is filled from the next and so on. Electrons that "fall into" into inner electron shells move from higher to lower energy levels and as a result emit energy in the form of characteristic X – radiation. The groupings of spectral lines are classified as K - , L – or M – spectra in terms of the patterns in which the outer electrons fall into the lower energy states. Each element has characteristic spectra lines with specific wavelengths superimposed on a low intensity continuous background spectrum.

Measurement of the spectrum of the emitted characteristic fluorescence radiation is performed using wavelength dispersive (WD) and energy dispersive (ED) spectrometers. In wavelength dispersive, X – ray fluorescence analysis (WDXRF), the result is an intensity spectrum of characteristic versus wavelength measured with a Bragg single crystal as dispersion medium. The crystal diffracts the various $\lambda$ values of the impinging of X – ray according to the Bragg equation:

$$n\lambda = 2dsin\theta. \qquad \ldots(2.11)$$

Where n = an integer, $\lambda$ = wavelength of the incident wave, d = the spacing between the planes in the atom lattices and $\theta$ = the angle between the incident ray and the scattering plane or the angle over the X – ray is reflected by crystal.

In energy dispersive X – ray fluorescence (EDXRF) analysis, a solid state detector is used to count the photons simultaneously sorting them according to energy and sorting the result in a multi - channel memory. The intensity and position of each spectral line diffracted by crystal in the spectrometer is recorded by an electronic X – ray counting device.

Qualitative X – ray fluorescence (XRF) analysis involves identification of the various spectral lines with the elements responsible for them. Quantitative analysis involves each X – ray intensity. Both peak and background intensity must be compared with that of standard (of known composition) of the same elemental makeup (Klein and Dutrow, 2008).

### 2.7.3. X-Ray Diffraction (XRD)

X-ray Diffraction (XRD) is a powerful technique used to uniquely identify the crystalline phases present in materials and to measure the structural properties.

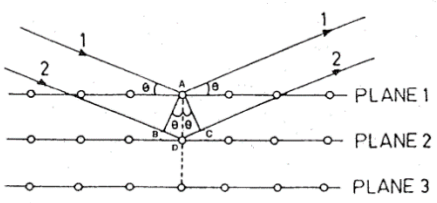



Figure2.1: X – rays incident on a plane

Figure 2.1 shows waves 1 and 2 in phase with each other, glancing off atoms A and B of a crystal that has a separation distance d between its atomic, or lattice, planes. The reflected (glancing) angle θ, as shown by experiment, is equal to the incident angle θ. The conditions for the two waves to stay in phase after both are reflected is that the path length CBD be a whole number (n) of wavelengths. But, from geometry, CB and BD are equal to each other and to the distance d times the sine of the reflected angle θ, or d sin θ. Thus, $n\lambda = 2dsin\theta$, which is the Bragg law. As may be seen from the diagram, when n = 2 there is only one wavelength along path CB; also, the reflected angle will be smaller than that for, say, n = 3. Waves reflected through an angle corresponding to n = 1 are said to be in the first order of reflection; the angle corresponding to n = 2 is the second order, and so on. For any other angle (corresponding to fractional n) the reflected waves will be out of phase and destructive interference will occur (Ron, 2000).

The X – ray diffraction is a phenomenon in which the atom of a crystal, by virtue of their uniform spacing causes an interferences pattern of the wave present in an incident beam of X – rays. X – ray diffraction procedure will reveal the detail about crystal size, crystal orientation and etc. The three methods of examining and measuring of the crystal structure are as follows:

- Laue Method
- Powder Method

- Rotating Crystal Method

**a) Laue Method:** This is application of single crystal or polycrystalline masses. In this method, continuous radiation is used and falls on a stationary crystal. The crystal diffracts the X – ray beam and produces a pattern of spots which conform exactly with the internal symmetry of the crystal. There are two ways of approaching this method, namely: transmission method and black reflection method. These approaches, depending on the relative position of the X – ray source, the crystal and the photographic film are placed in order to detect the diffracted X – ray. The Laue method is used to mainly for the determination of crystal orientation and assessment of crystal quality because the position of spot on the film depends on the orientation of the crystal with respect to the incident beam.

**b) Powder Method:** This method is applicable to finely divided crystalline powder. In this method, characteristic X – ray radiation of a fixed wavelength (monochromatic) is used. The mineral to be studied is in the form of a very fine powder, each particle of the powder is a very small crystal. A monochromatic X – ray radiation of one fixed wavelength is beamed on the crystal. If one plane is set at exactly the correct value $\theta$ for diffraction, then one and only one reflected (diffraction) beam from the crystal is observed. Hence, this satisfies the diffraction condition of Bragg's law: $n\lambda = 2dsin\theta$, where n = an integer, $\lambda$ = wavelength of the incident wave, d = the spacing between the planes in the atom lattices and $\theta$ = the angle between the incident ray and the scattering plane or the angle over the X – ray is reflected by crystal.

**c) Rotating Crystal Method:** This is also applicable to single crystal and is used for determining complex crystal structure from single crystal and thin film material. In the rotating crystal method, crystallographic planes are bought into reflecting position by

rotating a crystal about one of its axis with simultaneously irradiating it with a beam of monochromatic X – ray (Cullity, 1956; Ron, 2000).

### 2.7.4. Scanning Electron Microscopy (SEM)

Scanning Electron Microscopy (SEM) is used to obtain information on the morphology and surface features of a material in a micrometer scale including direct observation of crystal intergrowth, textures or reaction relationship. The quality and resolution of SEM images are function of three major parameters:

a) instrument performance,

b) selection of imaging parameters (e.g. operator control), and

*c)* nature of the specimen.

To acquire images, the SEM has an electron optical column in which a finely focused electron beam can be scanned on specified area of specimen. The resolution in SEM ranges from $50\ to\ 50\dot{A}$. The impact of an electron beam on the surface of solid sample cause various types of radiation signals that are recorded by detector above the specimen as shown in Fig 3. The intensity of signal detected in a backscatter electron detector is related to the average atomic number of the sample, its crystallographic orientation and surface topography. Hence, SEM has many applications in mineralogy. For good quality result, it is better to polish the surface of mineral.

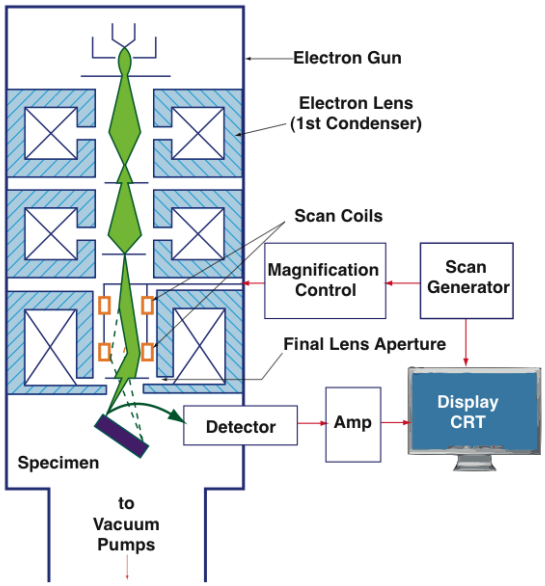



Figure 2.2: The main components of a typical SEM (Anon, 2014)

## 2.8. Azara Copper Ore Deposit

### 2.8.1. Location of Project Area

The study area is Azara Copper Ore Deposit in Awe Local Government Area of Nasarawa. Azara is on latitude N8.342825$^0$ and longitude E9.262246$^0$ on the global position system and on an elevation of 365m above sea level. The vein which hosts the copper ore in Azara is about 5km East of Azara village. Azara is located about 99.3km away from Lafia, the state capital, and off Markudi Road.

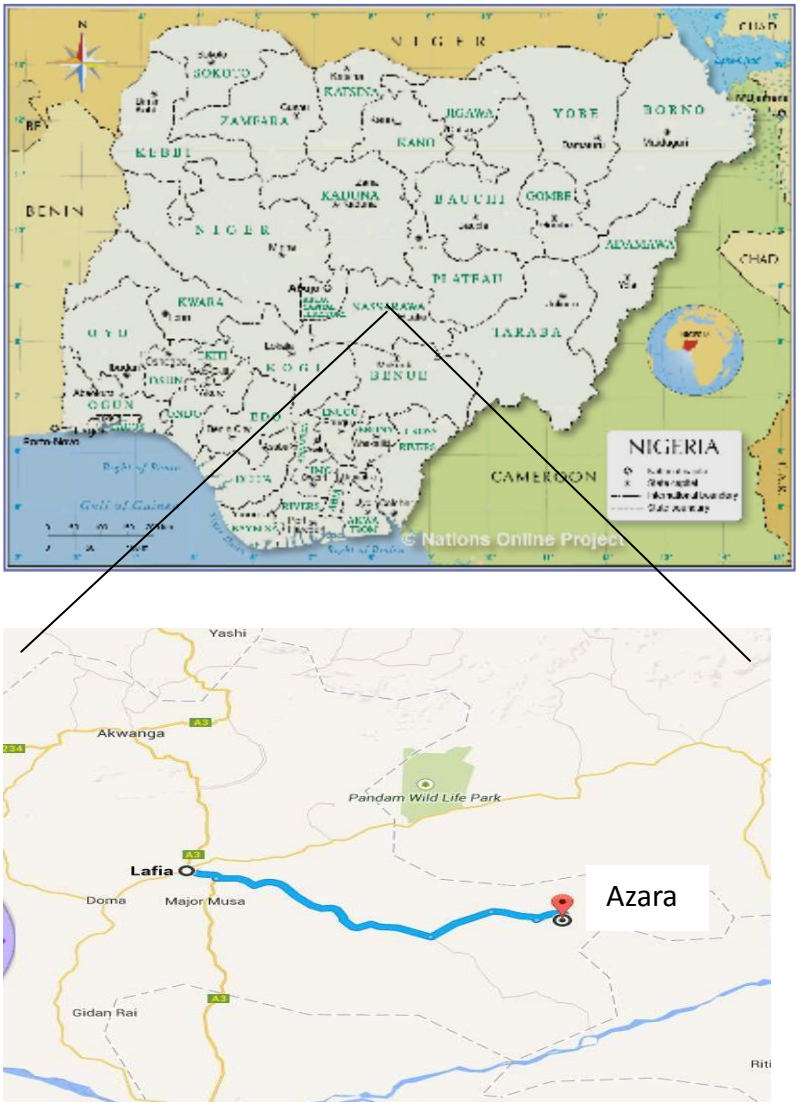



Figure 2.3: Map of Nigeria showing Azara in Nasarawa State (Source: Google map, 2014)

# CHAPTER THREE

## 3.0 METHODS AND MATERIALS

This chapter explains the various procedures used in carrying out the various characterization techniques that were used conducted in this research work.

### 3.1 Equipment and Materials Used

The following are the equipment used forthe research work:

- GPS unit
- Shovel
- Sample container
- Sledge hammer
- Laboratory jaw crusher
- Pulverizer
- Jones riffle
- Specific gravity bottle (Pycnometer)
- Weighing balance
- Beaker
- Retort stand
- X – ray fluorescence (XRF) Machine– Model: X – MET 5100 in Kaduna Polytechnic, Kaduna and Model: FXL – 83358 in Nigerian Institute of Mining and Geosciences, Jos.
- X – ray diffractometer (XRD) Machine – Shimadzu XRD Model: 6000 in National Steel Raw Materials Exploration Agency, Kaduna.
- Scanning Electron Microscope (SEM) – PHENOM WORLD MODELPROXin Katsina State University, Katsina.
- Denver pulverizing machine
- Denver sieve shaking machine
- Denver jaw crusher machine
- Denver ball milling machine
- Denver polishing machine

Materials

- The mineral sample from Azara in Awe local government area, Nasarawa
- Granite ore of known work index
- Water
- Canada balsam
- Aluminum oxide
- Carborundum (silicon carbide)

### 3.2 Methods

#### 3.2.1 Sample Collection

Samples of Azara copper ore which were representative of the ore wassourced from Azara in Awe Local Government Area in Nasarawa State. Grab method of collecting was employed and the location where each sample taken was recorded using Global Positioning System equipment (GPS). Twenty - four kilogram (24kg) sample of copper ore was obtained from three (3) pits 100metres (100m) away from each other and 10 meters (10m) deep.

#### 3.2.2. Laboratory Sampling and Preparation

In the laboratory, the re-sampling was done using Jones riffle. The sample was pulverized and blended thoroughly so as to achieve homogeneity. The homogenous sample was used for both chemical analysis and work index determination.

#### 3.2.3. Physical Observation

Method

i. The sample colour was determined using colour chart.

ii. The sample was examined by looking at the geometric shape of the mineral

iii. The sample was checked to determine wherever the breakage was as either irregular or conchoidal.

### 3.2.4 Determination of the Hardness of the Ore

To determine the relative hardness of any mineral based on the Mohs Hardness Scale, it was necessary to find which of these minerals the unknown mineral can and cannot scratch. Minerals of known hardness from the Mohs Hardness Kits were used.

Method

i. The sample was scratched using the mineral from the kit
ii. This was done continuously with harder or softer minerals from the kit until the hardness was determined.

### 3.2.5 Determination of Specific Gravity (Relative Density)

Procedure

i. The sample was washed thoroughly in order to remove dust and weighed
ii. Empty specific gravity bottle (pycnometer) was weighed and the weight was recorded
iii. The ground sample was filled into the specific gravity bottle to about 1/3 of the bottle and weighed the sample and bottle and the weight recorded
iv. The remaining space in the bottle was filled with water and weighed
v. The content of the bottle was discarded and filled afresh with water and the weight of bottle filled up with water was determined.

$$\text{Specific gravity (SG)} = \frac{\text{Weight of Mineral in Air}}{\text{Weight of Mineral in Air} - \text{Weight of Mineral in Water}} \quad \ldots (3.1)$$

Mass of empty bottle, $M_1$

Mass of empty bottle + ore, $M_2$

Mass of empty bottle + ore + water$M_3$

Mass of empty bottle + water,$M_4$

Mass of water filling the bottle ,$M_4 - M_1$

Mass of water having equal volume as ore $= (M_4 - M_1) - (M_3 - M_2)$

Relative Density of the Ore (S. G.) $= \frac{(M_2 - M_1)}{(M_4 - M_1) - (M_3 - M_2)}$

### 3.2.6 Determination of Electrical conductivity

The electrical conductivity of a material is a function its resistivity. Therefore the determination of the resistivity of the mineral is crucial in knowing the electrical conductivity of mineral.

Apparatus

1. Portable resistivity meter.
2. Small box with inside dimensions of 125 × 93 × 32 mm

Procedure

The ground sample loaded into the box as shown below. The portable resistivity meter was connected to box. The reading on the meter was taken and record. This was repeated used another portion of the sample.

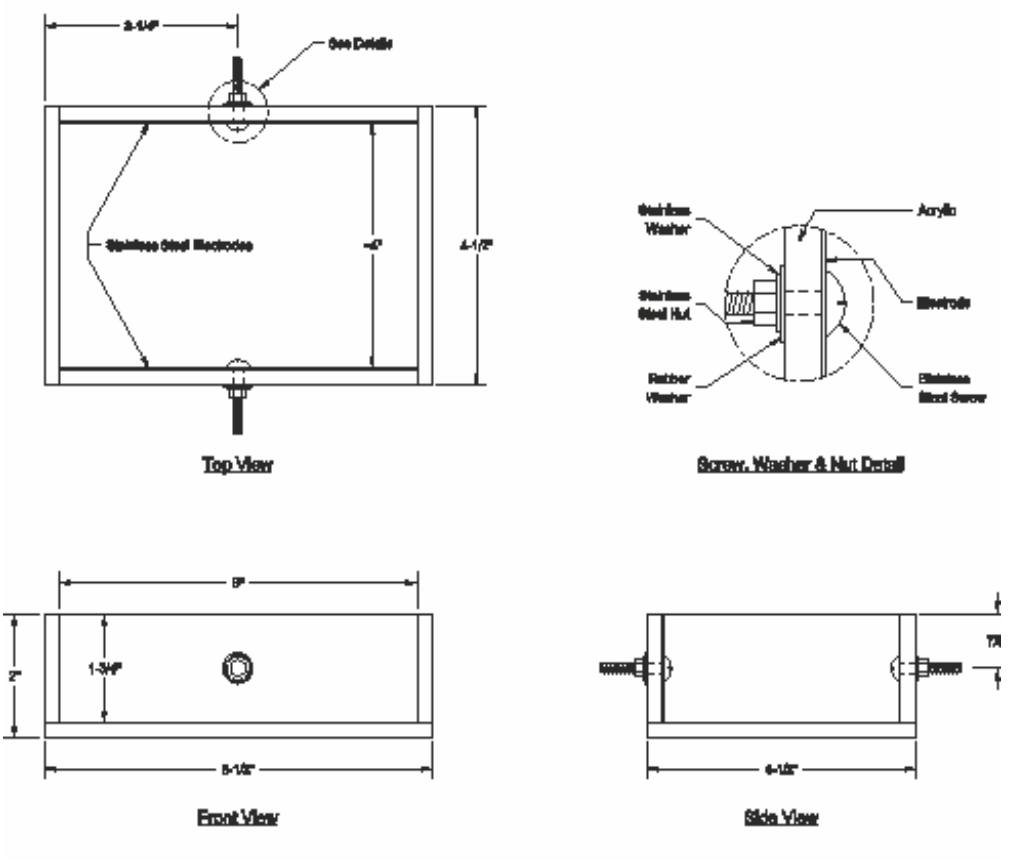



Figure 3.3: Box for Laboratory Resistivity Determination

The electrical conductivity is then calculated using the data generated and the results of these tests are presented in Table 4.2.

### 3.2.8.Determination of the Chemical Composition of the Ore

This analysiswas carried out in order to determine the chemical composition and the grade of the ore using X – ray fluorescence (XRF) for the elemental analysis. The head sample was analyzed by determining the percentage of composition of each element in the sample. The result of the analysis is presented in Table 4.3.

### 3.2.9. Mineralogical Analysis of the Ore

The mineralogy of the ore was determined using both X – ray diffraction (XRD), Scanning Electron Microscopy (SEM/EDS) and petrological analysis. This was carried out in order to identify the classification of individual minerals, mode of occurrence and the degree of association with each other.

#### 3.2.9.1. Preparation Procedures of Rock Section for Petrological Analysis

i. From the bulk sample, a section approximately 3mm thick was cut.

ii. The specimen was ground to produce flat and smooth surface which was free of gross deformation using carborundum (silicon carbide) of difference sizes ranging from 200 to 600 on the side to be examined.

iii. The specimen was washed, cleaned and dried. The specimen was mounted of the glass slide using Canada balsam as adhesive

iv. The specimen was further ground and polishedto mirror – like surface using aluminum oxide. The polished rock was mounted on the petrological microscope and examined with reflected rays of light.

### 3.2.10. Sieve Analysis and Liberation Studies

The size analysis was carried out in order to determine the distribution of various minerals especially copper in the various size fractions. This helped to determine the liberation size of the mineral in the ore.

The sample was ground and a weight of 200g was subjected to the size analysis using the laboratory sieve shaker. This was carried out using a nest of sieves based on $\sqrt{2}$scheme with the

coarsest on top and the finest sieve used at the bottom. The nominal apertures of the sieves used were 355μm, 250μm, 180μm, 125μm, 90μm and 50μm. 200g of the ground sample was placed on the coarsest sieve on the laboratory sieve shaker and operated for 10 minutes. The stack was unloaded after 10 minutes; the material retained on each sieve was weighed and recorded.

### 3.2.11. Determination of Work Index

The Modified Bond's method called Berry and Bruce method of determining the work index of an ore was used in this research work because of its simplicity, less time consuming and is called the comparative method of determining grindability parameter. This method required the use of a reference ore of known work index (Ore granite of known work index 15.14kWh/tonne). The procedure used in the determination of Azara copper ore included:

- The ore under test and the reference ore were crushed and pulverized.
- 200grams each of the test and reference ores were taken and sized by sieving into a number of size fractions using the automatic sieve shaker for 20minutes.
- Each sieved fraction of the test and reference ores was weighed and the value noted as the "Feed".
- The "Feed" test and reference ores were gathered together and introduced into the laboratory ball mill machine and ground for a specific time of one hour respectively.
- The test and reference ores from the laboratory ball mill machine were sieved and each sieved fraction was weighed and the value noted as the "product".

# CHAPTER FOUR

## 4.0. RESULTS AND DISCUSSIONS

### 4.1. Physico - Chemical Properties of Azara Copper Ore

#### 4.1.1. The Specific Gravity Test of the Ore

**Table 4.1: Result of Specific Gravity Test of the Ore**

| S/No | $M_1$ (g) | $M_2$ (g) | $M_3$ (g) | $M_4$ (g) | S. G. |
|---|---|---|---|---|---|
| 1 | 20.60 | 52.40 | 106.65 | 82.00 | 4.45 |
| 2 | 20.60 | 48.60 | 103.65 | 82.00 | 4.41 |
| 3 | 20.60 | 50.20 | 104.95 | 82.00 | 4.45 |
| 4 | 20.60 | 53.40 | 107.20 | 82.00 | 4.32 |
| 5 | 20.60 | 49.80 | 104.50 | 82.00 | 4.36 |

Average Specific Gravity = 4.40

Table 4.1 presents the result of the specific gravity test of the ore.The average specific gravity of the ore was found to be 4.40 and the value obtained compared favourably with the specific gravity value of chalcopyrite or colusite of 4.1 to 4.5 cited in the literature (Mursky and Thompson, 1958; MDP, 2001; 2005).This specific gravity value of 4.4 obtained for the ore sample indicates that the ore relatively dense and can be beneficiated using gravity method (Wills, 2006).

#### 4.1.2. Electrical Property of the Ore

The following results are obtained for the electrical property:

Length of the electrode, $l = 12.5cm$

Breadth of the electrode, $b = 9.3cm$

Resistance, $R = 3.4 \times 10^6 \Omega$

Ore Box Factor (OBF) $= \frac{\mathrm{A}}{\mathrm{D}}$,

Where A = Area of the electrode, $cm^2 = l \times b = 12.5 \times 9.3 = 116.25\ cm^2$;

D= Distance between electrodes, cm = 3.2 cm.

Ore Box Factor (OBF) $= \frac{116.25}{3.2} \approx 36.33cm$

The resistivity, in ohm – cm = OBF × Resistance = $36.33 \times 3.4 \times 10^6 \Omega$cm

$$= 1.235 \times 10^8 \Omega\text{cm} = 1.235 \times 10^6 \Omega\text{m}$$

The conductivity of the ore $= \frac{1}{\text{resistivity}} = \frac{1}{1.235 \times 10^6} = 8.096 \times 10^{-7} \Omega^{-1}\text{m}^{-1}$

**Table 4.2: Physico – Chemical Properties ofthe Ore**

| S/No | Property | Value |
|---|---|---|
| 1. | Physical observation | Metallic lustre, dark bronze with greenish strike and has irregular and uneven breakages. |
| 2. | Mohs Hardness | 3.50 |
| 3. | Specific Gravity | 4.40 |
| 4. | Electrical Conductivity | $8.096 \times 10^{-7} \Omega^{-1}\text{m}^{-1}$ |

Table 4.2 presents the physico – chemical properties of the ore;physical observation revealed that the ore possesses metallic lustre, is dark bronze with greenish stripe and irregular or uneven breakages. The Mohs hardness value was found to be 3.5, an indication that the ore is a soft texture type; electrical conductivity of$8.096 \times 10^{-7} \Omega^{-1}\text{m}^{-1}$ indicates that ore can allow

electricity to pass through it, hence the possibility of using electrostatic method among other methods to beneficiate the mineral ore. The colour and values obtained could also be attributed to the presence of the other associated elements like iron, zinc, tin and leadminerals in the ore that ought to be removed during mineral processing and extraction before the utilization of the metal copper. The values obtained also compared favourably with the results presented in Tables 2.1, 2.2 and 2.4 as reported by Anthony et al, (1995), Wills, (2006) and Weiss, (1985).

## 4.2 Chemical Characterization of the Ore Sample

### 4.2.1 Determination of the Chemical Composition of the Ore

**Table 4.3: Chemical Composition of the Head Sample (Azara Copper Ore)**

| **Element** | Fe | Cu | Pb | Mn | Ca | Sn | Sr | Zn |
|---|---|---|---|---|---|---|---|---|
| **% Composition** | 18.07 | 3.30 | 0.78 | 0.48 | 1.43 | 0.66 | 0.03 | 0.11 |
| **Element** | Zr | Ti | Nb | Sb | Ag | As | Si | S |
| **% Composition** | 0.03 | 0.34 | 0.02 | 0.03 | 0.30 | 0.07 | 19.62 | 1.66 |

Table 4.3(Appendix B: Figure B1) presents the result of chemical analysis of the head sample and reveals that the ore contains 18.07% Fe, 3.30%Cu, 0.78%Pb, 0.48%Mn, 1.43%Ca, 0.66%Sn, 0.03%Sr, 0.11%Zn, 0.03%Zr, 0.34%Ti, 0.02%Nb, 0.03%Sb, 0.03%Ag,0.07%As, 19.62%Si and 1.66%S.From the result in Table 4.3, it could be observed that silica, iron, copper, sulphurand calcium are predominant, while other associated elements are minor in the matrix of the ore. This phenomenon could be attributed to the mineralization of the deposit created by the geochemical formation of the ore deposit. Damisa(2008), reported that lead-zinc-copper complex ore situated within the Benue trough of which Nasarawa state is located; Jatau et al, (2014) also in their

findings, reported that the chemical analysis of the ore sample obtained from similar geological formation in Nasarawa state contains appreciable amount of copper. Hence, the similarity in the result obtained for the Azara copper ore deposit with those reported in the literatures. Therefore the Azara copper ore deposit could serve as another potential source for copper mineral since it contains3.30% copper and surpasses the 0.5% cut-off grade specified as standard for mining of copper mineral. In addition the ore also contains 0.66% Sn more than 0.45% cut – off grade specified for mining of tin mineral ores. The presence of silver as a precious metal in the ore could be seen as anotherbonus for the ore deposit (Weiss, 1985).

### 4.2.2 Mineralogical Characterization of the Azara Copper Ore Sample

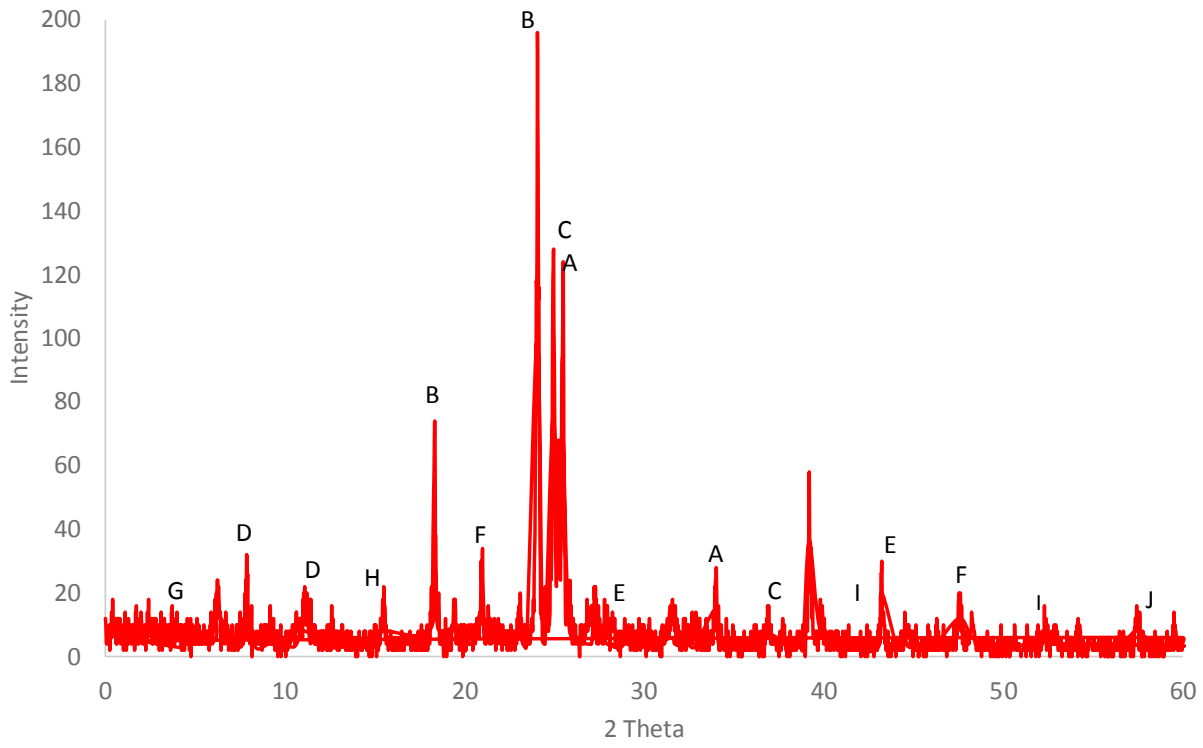



**Figure 4.1: XRD Pattern of the Head Sample**

**Table 4.4: XRD Analysis Result of the Head Sample**

| Mineral Name | Chemical Name | Chemical Formula | Estimated Frequency |
|---|---|---|---|
| Anatase, syn | Titanium Oxide | $TiO_2$ | 2.2 |
| Zincite, syn | Zinc Oxide | $ZnO_2$ | 2.5 |
| | Zirconium Oxide | ZrO | 2.2 |
| Chalcopyrite | Copper Iron Sulphide | $CuFeS_2$ | 3.8 |
| Hopeite, Syn | Zinc Phosphate Hydrate | $Zn_3(PO_4)_2.4H_2O$ | 5.4 |
| Hematite | Iron Oxide | $Fe_2O_3$ | 1.8 |
| Goethite | Iron Oxide Hydrate | $Fe_2O_3.H_2O$ | 5.4 |
| Calcite | Calcium Carbonate | $CaCO_3$ | 2.2 |
| Pyrite | Iron Sulphide | FeS | 3.2 |
| | Potassium Iron Magnesium Alumium Silicate | $K–Mg–Al–SiO_2.H_2O$ | 2.6 |
| Tenrite, syn | Copper Oxide | CuO | 2.6 |

| | | | |
|---|---|---|---|
| Dickite – 2/IT<br><br>(Kaolinite) | Aluminum Silicate<br><br>Hydroxide | $Al_2(Si_2O_5)(OH)_4$ | 7.2 |

Figure 4.1 and Table 4.4present the result of XRD analysis of the ore sample. The resultrevealed the presents of anatase, zincite, chalcopyrite, hopeite, hematite, goethite, calcite, tenrite, dickite and pyrite. From the result it could be observed that the minerals presents have the following: anatase(2.2), zincite(2.5), chalcopyrite (3.8), hopeite(5.4), hematite (1.8), goethite(5.4), calcite(2.2), tenrite (2.6) dickite (7.2) and pyrite(3.2) with chalcopyrite, hopeite, goethite and pyrite as the major minerals and anatase, zincite, and calcite as the minor minerals contained in the matrix of the ore. This findings compared favorably with similar works done by Jatauet al, (2014), Abubakre,et al (2014) and Damisa,(2008) where they stated that the copper ore minerals within the Nasarawa schist are predominantly chalcopyrite in association with iron, lead, zinc, titanium and bismuth as gangues. Hence, the need for the beneficiation of the ore in order to upgrade its copper content.

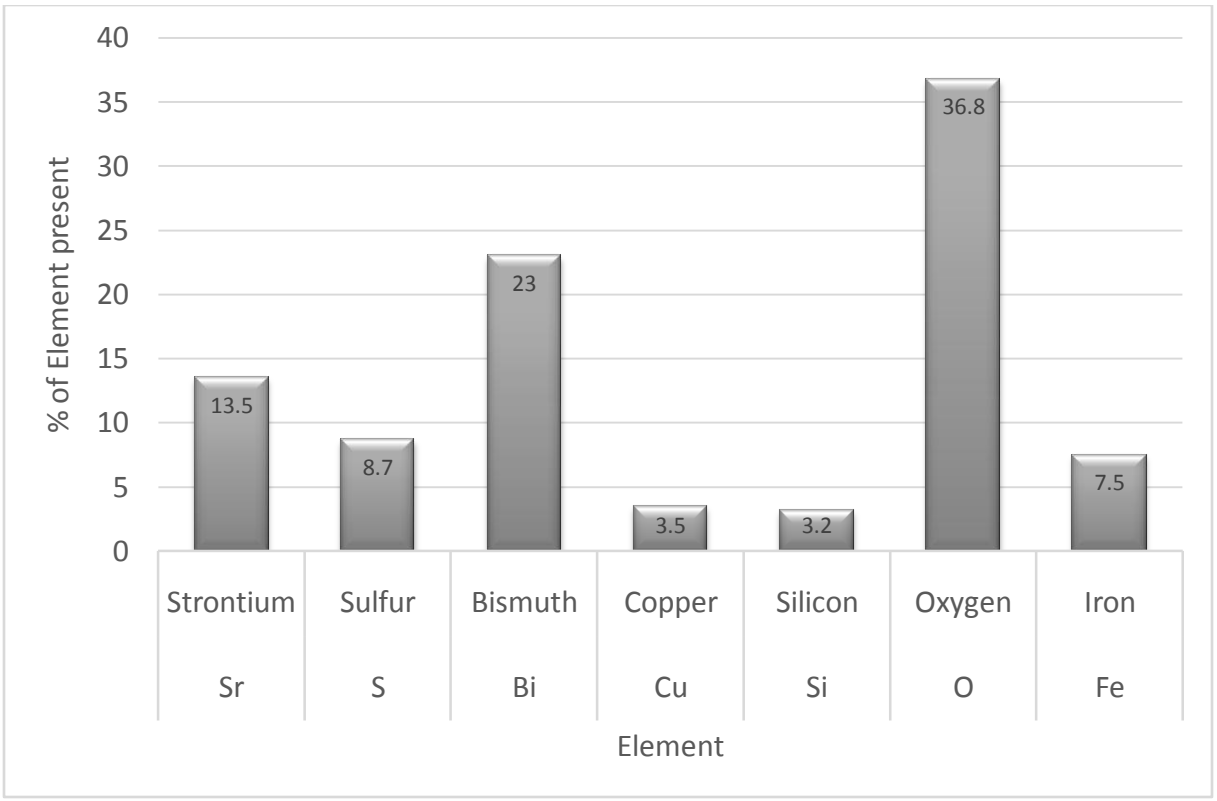



**Figure 4.2: ED X-ray Spectrum result of the copper ore sample**

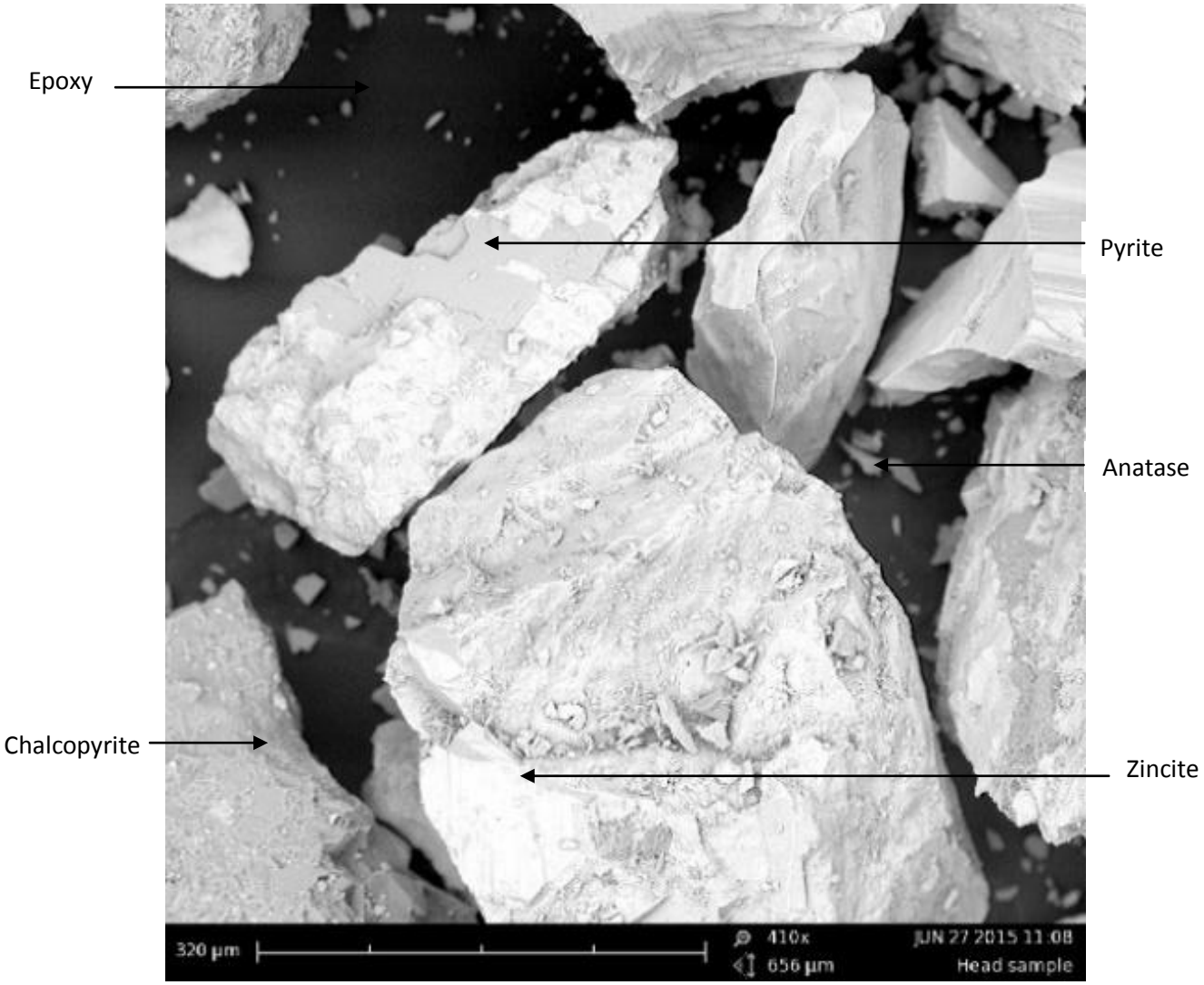



**Plate4.1: SEM image of the copper ore sample**

Figure 4.2 presents the EDS result of the SEM analysis of the head sample. From the result it could be seen that the ore contains 3.5%Cu, 23%Bi, 7.5%Fe, 13.5%Sr, 8.7%S, 3.2%Si and 36.8%O on the average. Furthermore, the result also confirmed the presence of copper in association with other elements as revealed by the XRF, but with the revelation of the present of bismuth in the ore matrix.The presence of these associated minerals are no threat to the utilization of the ore since the technology of beneficiating the associated minerals can be developed (Weiss, 1985).

Plates 4.1 to 4.5 shows the SEM micrographs of the copper ore sample. From the results can be observed that five phases can be recognized: the embedding resin (black background),chalcopyrite (dark grey), pyrite (light grey), zincite (white) and anatase (grey particle).Furthermore it could also be observed that the minerals are separated by grains boundaries, no interlocking of minerals and the mineral particles vary in sizes ranging from 80 to 320µm.This phenomenon indicates that the minerals can be easily freed from each other during comminution, hence the low requirement in the energy required for the liberation of the copper mineral (Oyeladun et al, 2012).

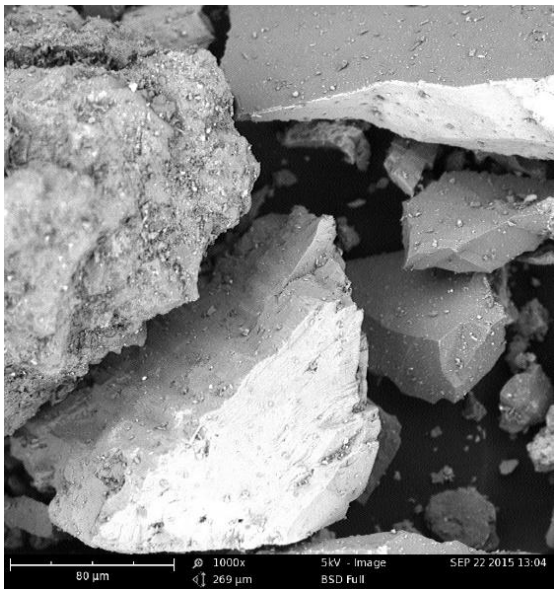



Plate 4.2: SEM Image of Head Sample (× 1,000)

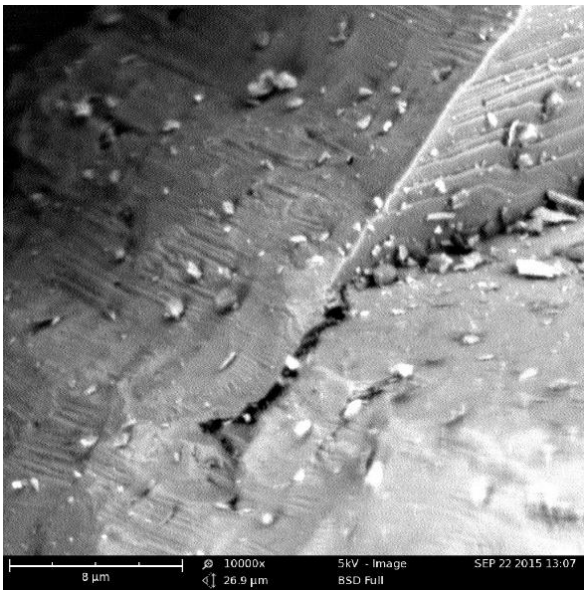



Plate 4.3: SEM Image of Head Sample (× 10,000)

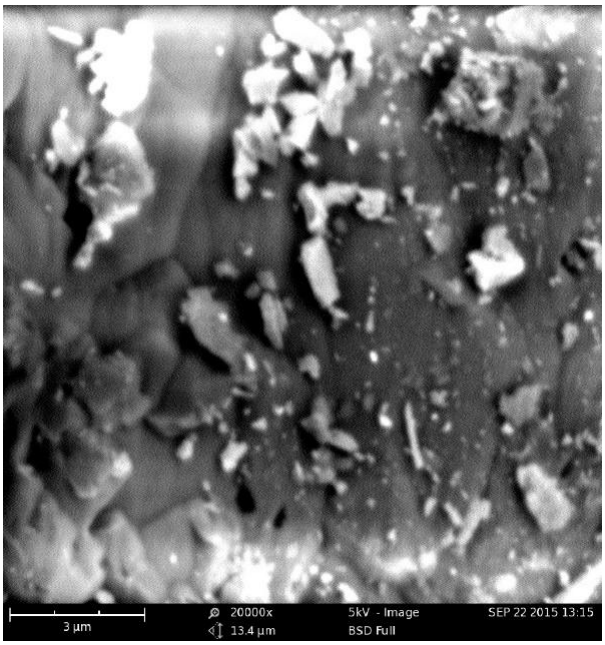



Plate 4.4: SEM Image of Head Sample (× 20,000)

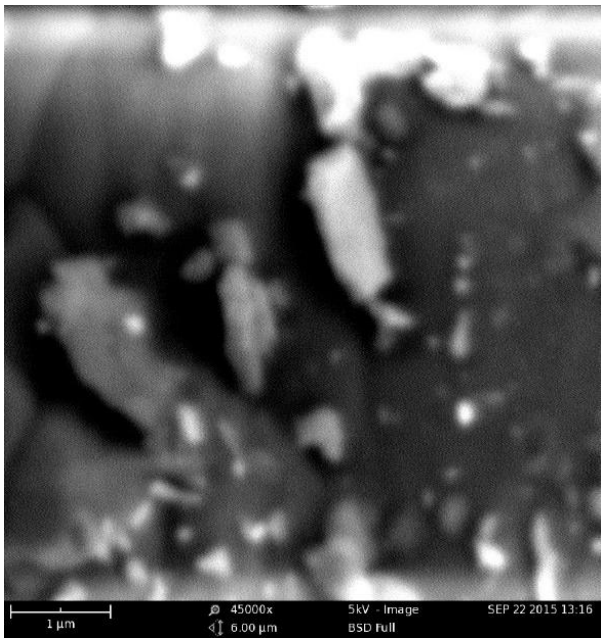



Plate 4.5: SEM Image of Head Sample (× 45,000)

### 4.2.3. Petrological Analysis of the Head Sample

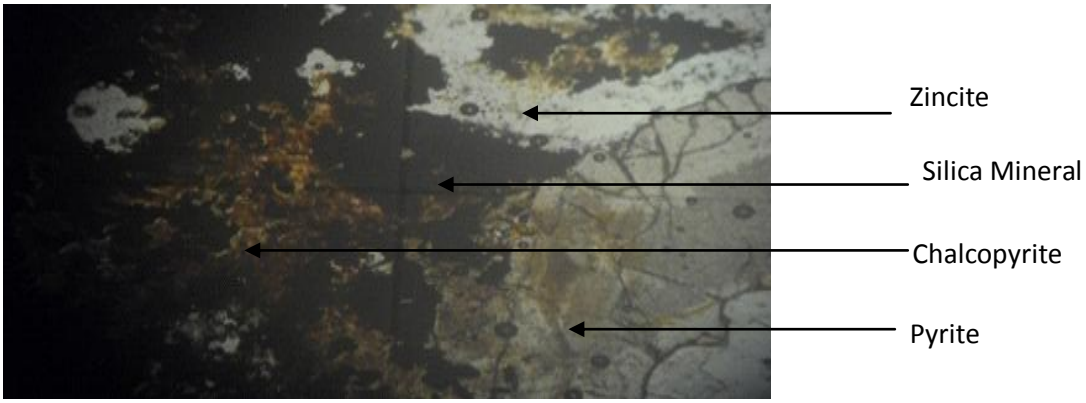


Plate 4.6: Micrograph of Copper Sample under plane polarized light (X 100)

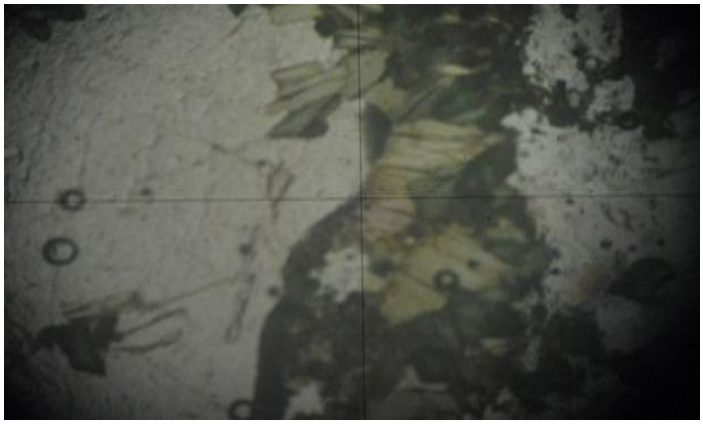


Plate 4.7: Micrograph of Copper Sample under crossed polarized light (X 100)

Plates 4.6 and 4.7 show the petrological micrographs of the copper ore sample. Fromthe result four mineral phasescan be recognized under plane polarized light and cross polarized light, the phases are chalcopyrite (brass yellow), pyrite (pale yellow), zincite (grey) and silica mineral (light dark). This result confirmed the result of XRD analysis, which indicated that the mineral present are chalcopyrite, pyrite and silica mineral and also confirmed that the mineral of interest copper is actually presence in the Azara copper ore sample.

## 4.3. Sieve Analysis and Liberation Studies

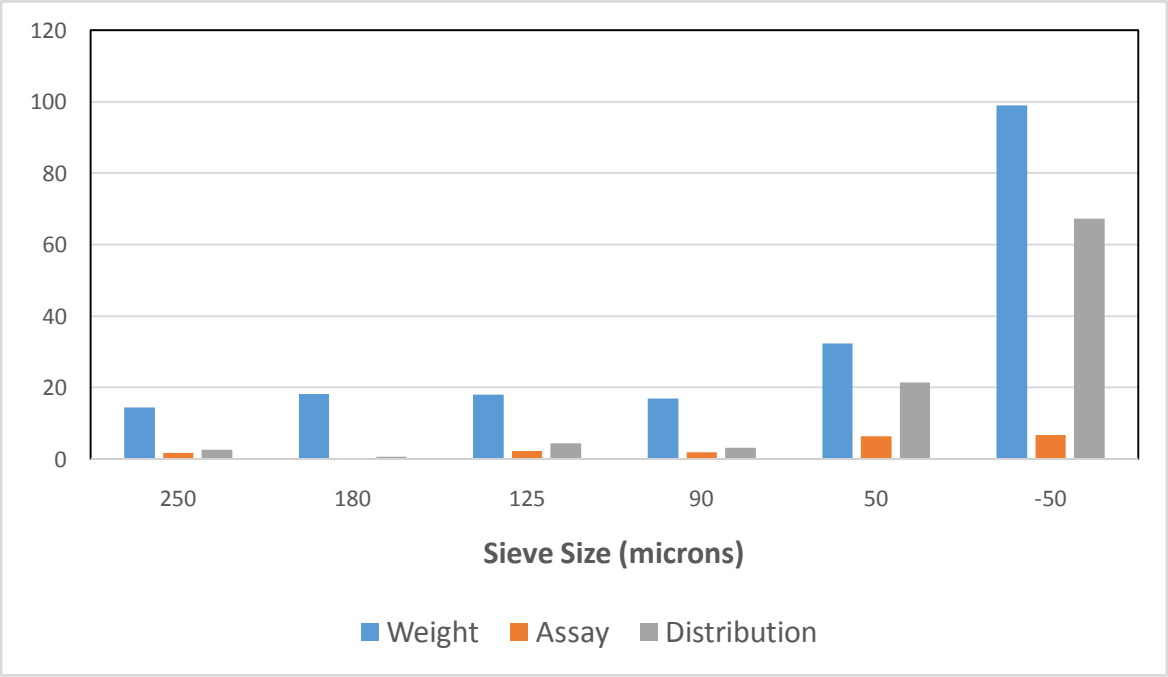



**Figure 4.3: Size Distribution of the Copper in the Ore Matrix**

Figure 4.3 (Appendix B: Tables B2 and B3) presents the result of the sieve size and assay analysis of the ore sample. From the result, the following assay values were obtained with 1.85%Cu in $-355+250\mu m$, 0.43%Cu in $-250+180\mu m$, 2.35%Cu in $-180+125\mu m$, 1.9%Cu in $-125+90\mu m$, 6.53%Cuin $-90+50\mu m$ and 6.73%in $-50\mu m$.This phenomenon indicates that the copper mineral distributed itself normally and the content increase as the particle seize fraction reduces. This trend compared favourably with works done by Damisa(200), Weiss, (1985), Wills, (2006) and Abubakre et al,(2014) where they also observed similar trends. Furthermore, the liberation size of the copper ore is found to be$-50\mu m$being the sieve size with the highest assay value of the total copper content of 6.73% and distribution of 67.35% asdefined(Wills, 2006; Mills, 2014).

## 4.4 Determination of Work index

Figures 4.3 to 4.10 (Appendix B: Tables B6 to B9) present the results of particle size analyses of both referenceore (granite) and the test sample (copper ore). The values of 80% passing for feeds and products for both reference and test samples were obtained usingthe semi log plots and calculated using Gaudin-Schuhman logarithm equation to verify if the values obtained from the plots are the same. The value of the work index of the reference ore(15.14kWh/tonne) and 80% passing of the reference ore were used to determine the work index of the copper ore.

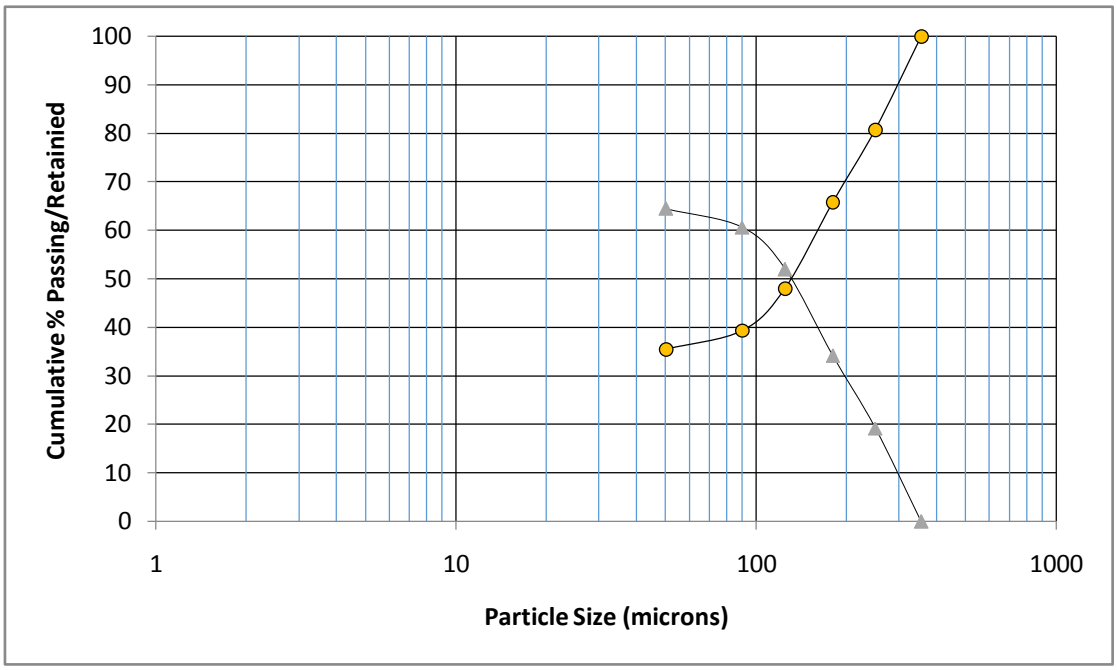



**Figure 4.4: Plot of cumulative % undersize (passing) and oversize (retained) for test ore feed.**

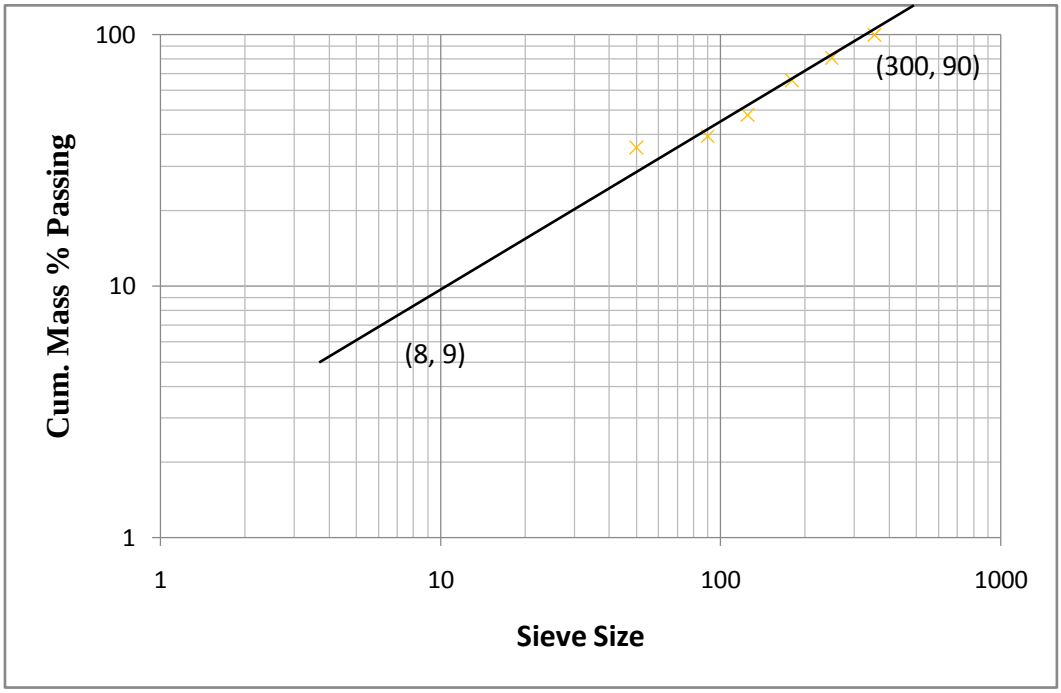



**Figure4.5: Log-log plot of cumulative % passing (undersize) vs size, (Gaudin-Schuhman plot for feed of the test ore)**

The Gaudin – Schuhman distribution given as:

$$y = 100\left(\frac{x}{k}\right)^{a}$$

Where y = cumulative mass % passing size

x = screen aperture size

k = size parameter

a = distribution parameter

From figure 4.5,

$$\text{Slope} = \frac{\log 90 - \log 9}{\log 300 - \log 8} = 0.6353$$

Using point (8, 9) in Gaudin-Schuhman logarithm equation, we have

$$Y = 0.6353X + 0.3805$$

$$Where\ Y = \log y\ and\ X = \log x$$

Calculating 80% passing, gives

$$\log 80 = 0.6353 \log x + 0.6353$$

$$x = 249.3\ \mu m$$

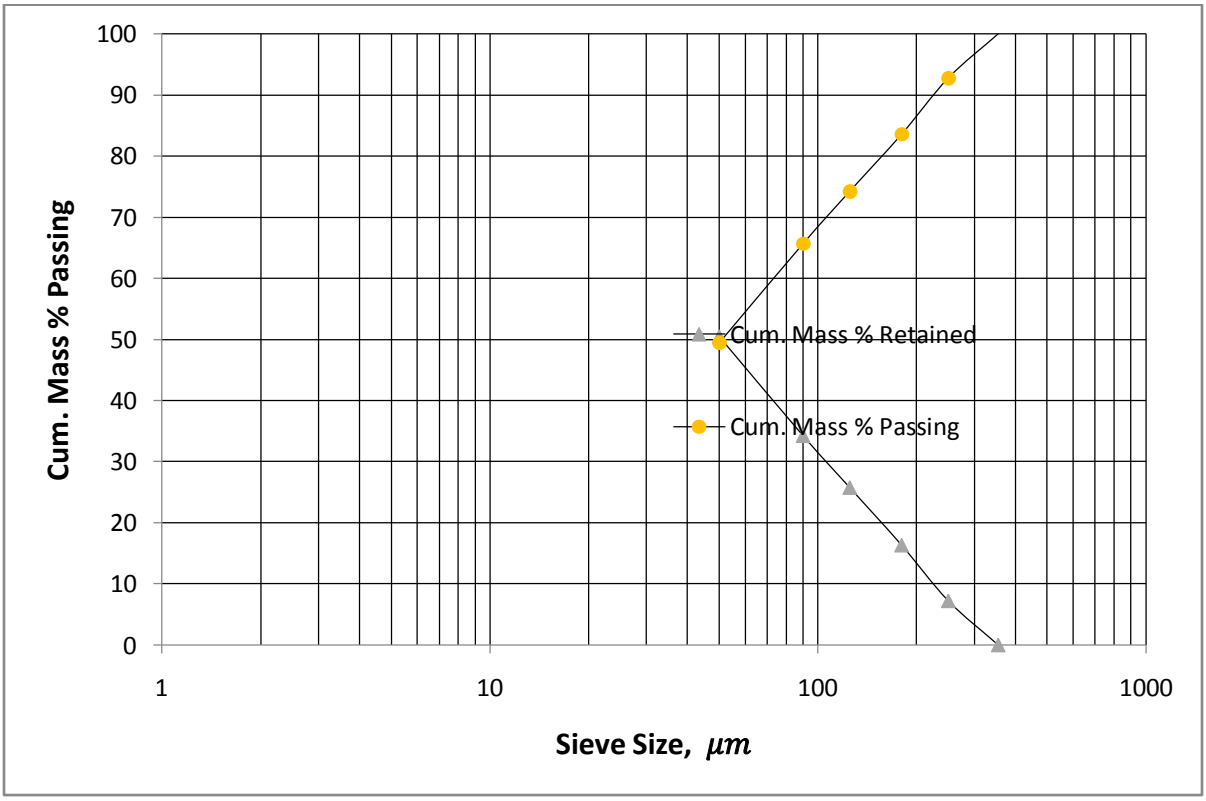



**Figure 4.6: Plot of cumulative % undersize (passing) and oversize (retained) for test ore (product).**

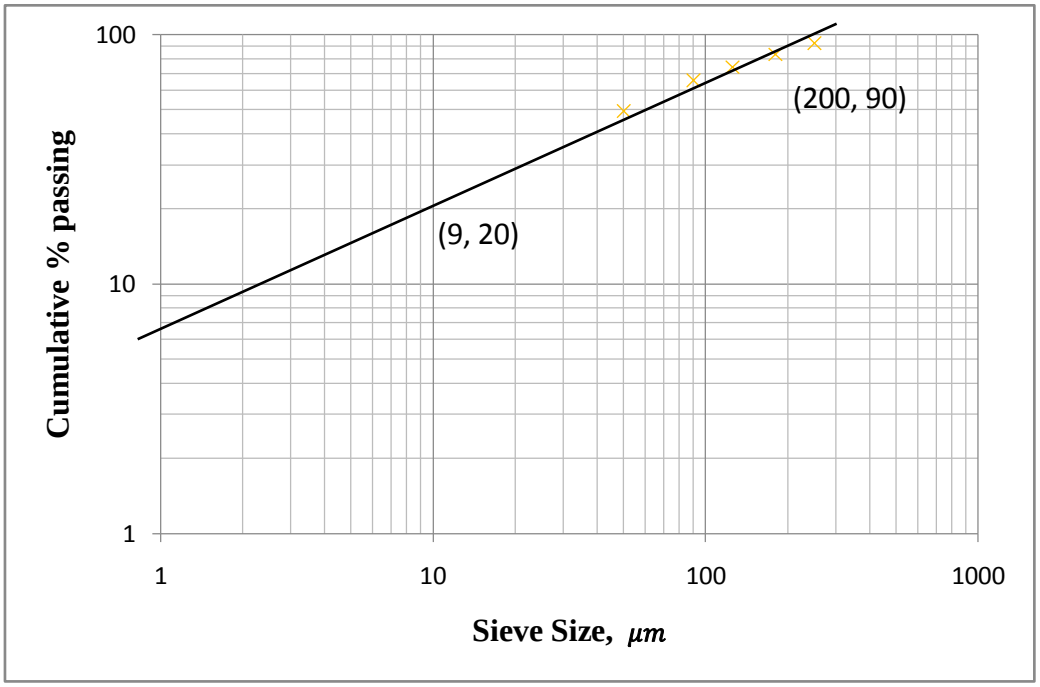



**Figure 4.7: Log-log plot of cumulative % passing (undersize) vs size, (Gaudin-Schuhman plot for test ore product)**

From figure 4.7,

$$\text{Slope} = \frac{\log 90 - \log 20}{\log 200 - \log 9} = 0.485$$

Using point (9, 20) in Gaudin-Schuhman logarithm equation, we have

$$Y = 0.485X + 0.8382$$

$$Where\ Y = \log y\ and\ X = \log x$$

Calculating 80% passing, gives

$$\log 80 = 0.485 \log x + 0.8382$$

$$x = 156.9\ \mu m$$

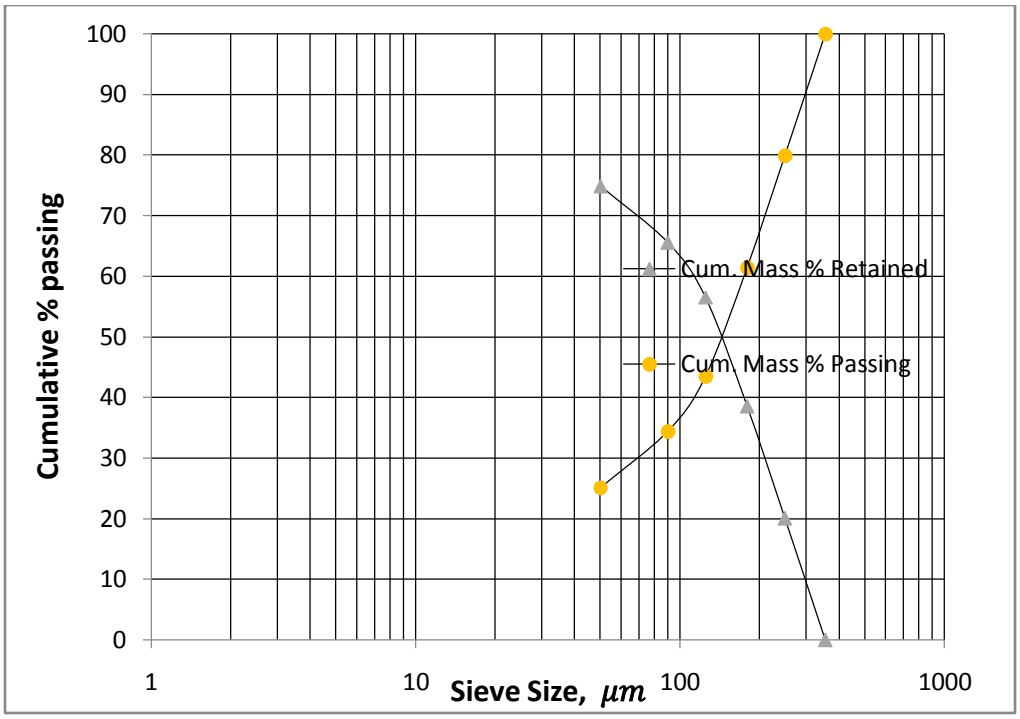



**Figure 4.8: Plot of cumulative % undersize (passing) and oversize (retained) for reference ore feed.**

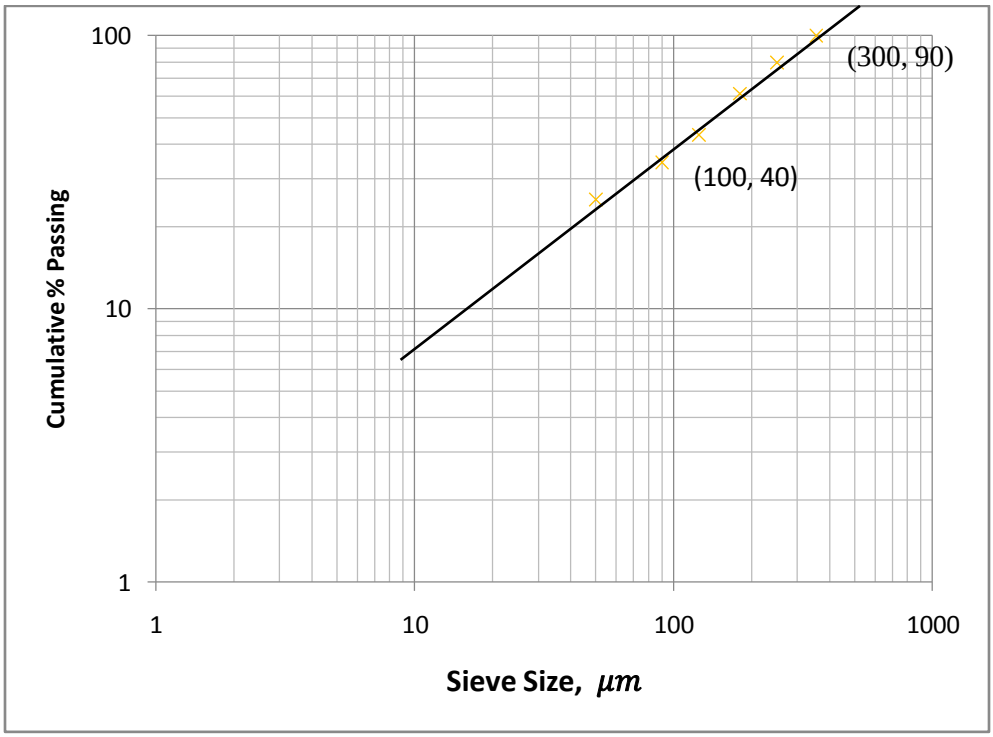



**Figure4.9: Log-log plot of cumulative % passing (undersize) vs size, (Gaudin-Schuhman plot for reference ore feed)**

From figure 4.9,

$$\text{Slope} = \frac{\log 90 - \log 40}{\log 300 - \log 100} = 0.738$$

Using point (100, 40) in Gaudin-Schuhman logarithm equation, we have

$$Y = 0.738X + 0.1261$$

$$Where\ Y = \log y\ and\ X = \log x$$

Calculating 80% passing, gives

$$\log 80 = 0.738 \log x + 0.1261$$

$$x = 255.8\ \mu m$$

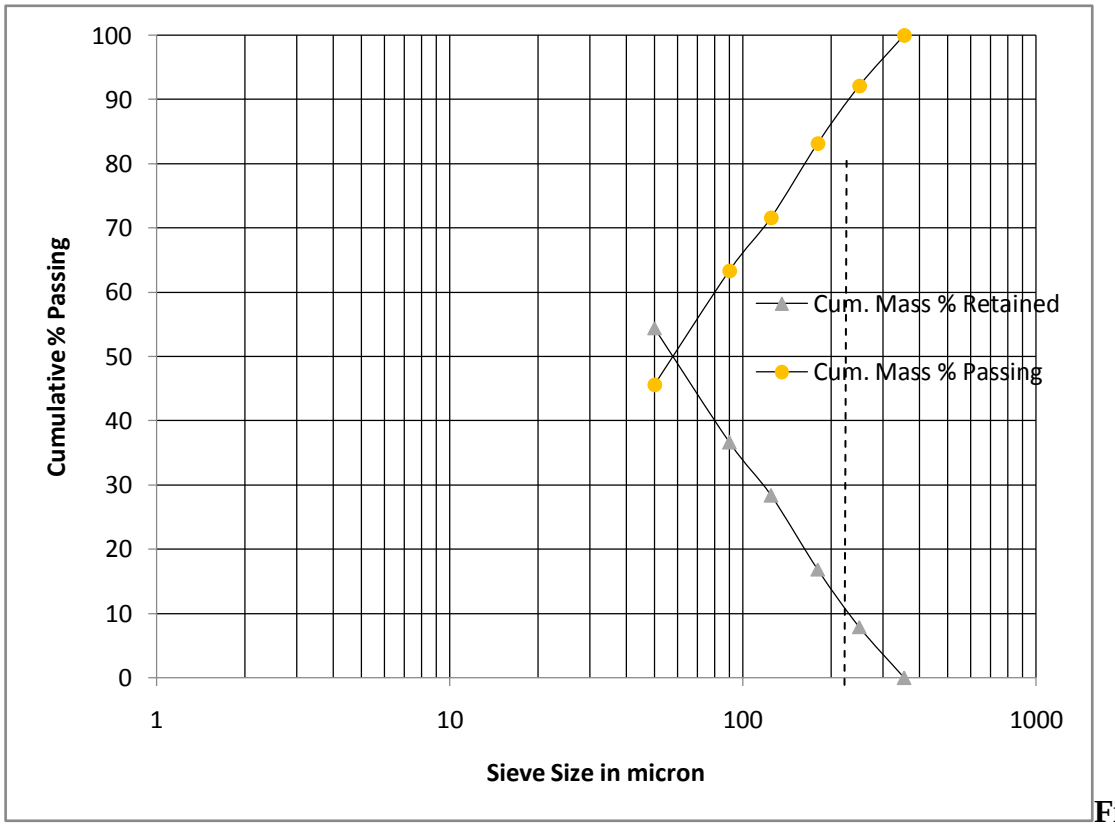



**Figure 4.10: Plot of cumulative % undersize (passing) and oversize (retained) for referenceore (product).**

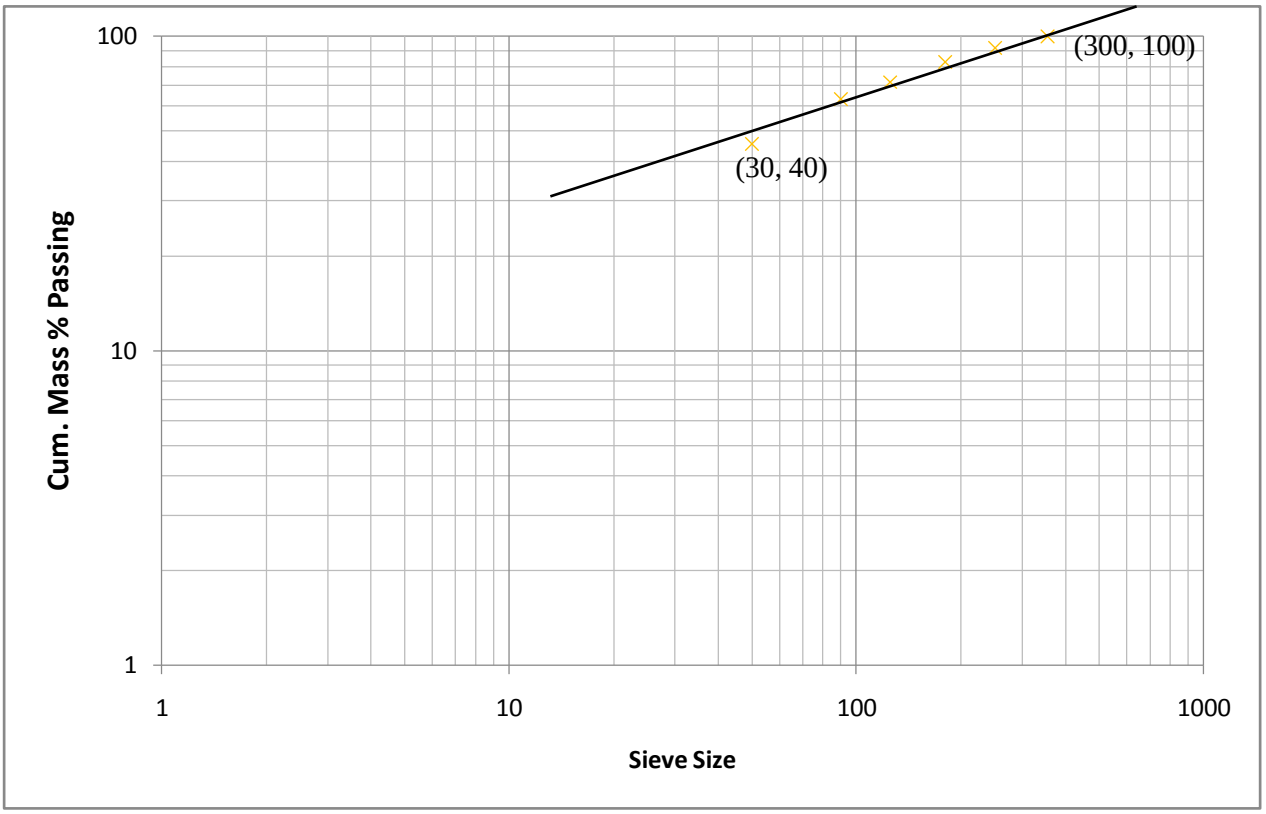



**Figure 4.11: Log-log plot of cumulative % passing (undersize) vs size, (Gaudin-Schuhman plot for reference ore Product)**

From figure 4.11,

$$\text{Slope} = \frac{\log 100 - \log 40}{\log 300 - \log 30} = 0.3979$$

Using point (100, 40) in Gaudin-Schuhman logarithm equation, we have

$$Y = 0.3979X + 1.0143$$

$$Where\ Y = \log y\ and\ X = \log x$$

Calculating 80% passing, gives

$$\log 80 = 0.3979 \log x + 1.0143$$

$$x = 171.3\mu m$$

Calculating the Work Index using Modified Berry and Bruce Method;

$$\text{Work Index} = 15.14\left(\frac{\frac{1}{\sqrt{171.3}} - \frac{1}{\sqrt{255.8}}}{\frac{1}{\sqrt{156.9}} - \frac{1}{\sqrt{249.3}}}\right) = 12.989\text{kWh/tonne}$$

Using the modified Bond's method of work index determination for the copper ore, it was found that the work index is 12.989kWh/tonne and this implies that about 12.989 kilowatts hour of energy is required to grind the ore from theoretical infinite size to 80 per cent passing in line with Bonds work index. This value obtained compared favourably with other work reported by Fuerstenau and Han, (2003). Using the Denver grindability test curves, the copper ore can be classified as a medium soft texture type (B) (Mathur, 1985).

# CHAPTER FIVE

## 5.0 CONCLUSION AND RECOMMENDATION

### 5.1 Conclusion

The characterization of Azara copper ore was carried out and the following conclusions are drawn:

i. the copper ore was found to be a metallic lustre, dark bronze with greenish stripe and has irregular or uneven breakages, hardness of 3.5 using Mohs hardness scale, the specific gravity of 4.40 and electrical conductivity of $8.096 \times 10^{-7}\Omega^{-1}m^{-1}$.

ii. the copper ore was found to be 3.3% Cu and contains anatase, zincite, chalcopyrite, hopeite, and tenrite as valuable mineral and kaolinite, hematite, goethite, calcite and pyrite as gangue. The copper bearing minerals are separated from other associated minerals in the ore by smooth grains boundaries and non-interlocking by another mineral, hence, making it easy to free from other associated minerals by comminution.

iii. the particle size analysis/ assay indicated that the size and mineral distribution of ore as follows: 1.85% Cu in $-355+250\mu m$, 0.43% Cu in $-250+180\mu m$, 2.35% Cu in $-180+125\mu m$, 1.9% Cu in $-125+90\mu m$, 6.53% Cu in $-90+50\mu m$ and 6.73% Cu in $-50\mu m$ with the liberation size of the ore possible below $50\mu m$.

iv. the work index of the ore was found to be 12.989kWh/tonne and graded as type B with soft medium texture.

Hence, the ore could be mined for its copper mineral as its parameters meet the 0.4 to 1.0% Cu cut-off grade required for mining copper mineral globally and also as a source of other alloying minerals since it contained reasonable amount of associated valuable minerals as gangue.

## 5.2 Recommendation

The beneficiation parameters of the ore should be investigated in order to develop the specific technology for the process route of the ore.

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## Appendices

### Appendix A: Equipment and Materials



Figure A1: Copper Ore from Azara



Figure A2: Pycnometer on a Scale



Figure A3: Sieve shaking machine



Figure A4: Ball Mill Machine



Figure A5: SEM Machine

## Appendix B: Chemical and Mineralogical Analyses

**Table B1: EDS of the Head Sample**

| Element Number | Element Symbol | Element Name | Confidence | Concentration (%) | Error |
|---|---|---|---|---|---|
| 38 | Sr | Strontium | 100.0 | 13.5 | 1.2 |
| 16 | S | Sulfur | 100.0 | 8.7 | 1.2 |
| 83 | Bi | Bismuth | 100.0 | 23.0 | 1.6 |
| 29 | Cu | Copper | 100.0 | 3.5 | 1.5 |
| 14 | Si | Silicon | 100.0 | 3.2 | 1.8 |
| 8 | O | Oxygen | 100.0 | 36.8 | 1.5 |
| 26 | Fe | Iron | 100.0 | 7.5 | 1.8 |

**Table B2: Chemical Analysis of various sieve size**

| Sieve Size/ %Element | $+355$ | $-355$ $+250$ | $-250$ $+180$ | $-180$ $+125$ | $-125$ $+90$ | $-90$ $+50$ | $-50$ |
|---|---|---|---|---|---|---|---|
| Fe | 14.52 | 17.12 | 18.12 | 24.54 | 27.35 | 25.93 | 23.43 |
| Cu | 3.34 | 1.85 | 0.43 | 2.35 | 1.90 | 6.53 | 6.73 |
| Pb | 1.52 | 0.46 | 0.16 | 0.55 | 0.65 | 1.09 | 1.08 |
| Mn | 0.11 | 0.16 | | 0.22 | 0.48 | 0.98 | 1.43 |
| Ca | | | | | 10.02 | | |
| Sn | 0.16 | 0.15 | 0.15 | | | | |
| Sr | | | | | 0.23 | | |
| Cr | | | | | | 0.36 | 0.40 |
| Zn | 0.08 | 0.03 | 0.03 | 0.07 | 0.13 | 0.18 | 0.14 |

| | | | | | | | |
|---|---|---|---|---|---|---|---|
| Zr | | | | 0.02 | 0.09 | 0.04 | 0.04 |
| Mo | 0.03 | | | | | | 0.02 |
| Ti | | | | | 1.88 | 0.52 | |
| Nb | 0.03 | | | | 0.04 | 0.05 | 0.03 |
| Sb | 0.21 | | | | | | |
| Ag | | | 0.23 | | | | |
| As | 0.28 | | | | | 0.23 | |
| Si | 23.37 | 20.46 | 19.29 | 21.37 | 22.61 | 19.38 | 14.66 |
| S | 1.01 | 1.06 | 1.20 | 1.59 | 1.65 | 1.97 | 2.51 |

**Table B 3: Metallurgical Balance for Copper Ore**

| **Sieve Size** $\mu m$ | **Weight** | **Assay of Cu (%)** | **Metal Content** | **Distribution (%)** |
|---|---|---|---|---|
| $-355+250$ | 14.42 | 1.85 | 26.68 | 2.70 |
| $-250+180$ | 18.31 | 0.43 | 7.87 | 0.80 |
| $-180+125$ | 18.75 | 2.35 | 44.07 | 4.46 |
| $-125+90$ | 17.08 | 1.9 | 32.44 | 3.28 |
| $-90+50$ | 32.45 | 6.53 | 211.89 | 21.42 |
| $-50$ | 98.99 | 6.73 | 666.20 | 67.35 |

**Table B 4: Excel Worksheet for Metallurgical Balance**

| | **A** | **B** | **C** | **D** | | | **E** |
|---|---|---|---|---|---|---|---|
| **1** | Sieve Size | Weight | Assay of | Weight Metal | Weight | Distribution | Distribution (%) |

| | | | Cu (%) | | Metal | | |
|---|---|---|---|---|---|---|---|
| **2** | – 355 + 250 | 14.42 | 1.85 | =B2*C2 | 26.68 | =D2*100/D$8 | 2.70 |
| **3** | – 250 + 180 | 18.31 | 0.43 | =B3*C3 | 7.87 | =D3*100/D$8 | 0.80 |
| **4** | – 180 + 125 | 18.75 | 2.35 | =B4*C4 | 44.07 | =D4*100/D$8 | 4.46 |
| **5** | – 125 + 90 | 17.08 | 1.9 | =B5*C5 | 32.44 | =D5*100/D$8 | 3.28 |
| **6** | – 90 + 50 | 32.45 | 6.53 | =B6*C6 | 211.89 | =D6*100/D$8 | 21.42 |
| **7** | – 50 | 98.99 | 6.73 | =B7*C7 | 666.20 | =D7*100/D$8 | 67.35 |
| **8** | | | | =SUM(D2:D7) | 989.15 | | |

**Table B 5: Excel Worksheet for Particle Size Analysis (Copper Ore)**

| | **A** | **B** | **C** | | **D** | | **E** | | **F** |
|---|---|---|---|---|---|---|---|---|---|
| **1** | **S/No** | **Sieve Size** | **Mass (g)** | | **Mass (%)** | | **Cum. Mass % Retained** | | **Cum. Mass % Passing** |

| | | | | | | | | | |
|---|---|---|---|---|---|---|---|---|---|
| **2** | 1 | 355 | 38.386 | =C2*100/C$8 | 19.193 | 0.00 | 0.00 | =D2+F3 | 100.00 |
| **3** | 2 | 250 | 29.998 | =C3*100/C$8 | 14.999 | =D2+E2 | 19.19 | =D3+F4 | 80.81 |
| **4** | 3 | 180 | 35.601 | =C4*100/C$8 | 17.8005 | =D3+E3 | 34.19 | =D4+F5 | 65.81 |
| **5** | 4 | 125 | 17.283 | =C5*100/C$8 | 8.6415 | =D4+E4 | 51.99 | =D5+F6 | 48.01 |
| **6** | 5 | 90 | 7.72 | =C6*100/C$8 | 3.86 | =D5+E5 | 60.63 | =D6+F7 | 39.37 |
| **7** | 6 | 50 | 71.012 | =C7*100/C$8 | 35.506 | =D6+E6 | 64.49 | =D7+F8 | 35.51 |
| **8** | 7 | pan | =SUM(C2:C7)) | | | =D7+E7 | 100.00 | | 0.00 |

**Table B6:Particle size analysis of feed samples (Copper Ore) for grindability test**

| S/No | Size Fraction ($\mu m$) | Sieves, ($\mu m$) | Mass (g) | Mass (%) | Cum. Mass % Retained | Cum. Mass % Passing |
|---|---|---|---|---|---|---|
| 1 | – 355 + 250 | 355 | 38.386 | 19.193 | 0.00 | 100.00 |
| 2 | – 250 + 180 | 250 | 29.998 | 14.999 | 19.19 | 80.81 |
| 3 | – 180 + 125 | 180 | 35.601 | 17.8005 | 34.19 | 65.81 |
| 4 | – 125 + 90 | 125 | 17.283 | 8.6415 | 51.99 | 48.01 |
| 5 | – 90 + 50 | 90 | 7.72 | 3.86 | 60.63 | 39.37 |
| 6 | – 50 | 50 | 71.012 | 35.506 | 64.49 | 35.51 |
| Total | | | 200 | | 100.00 | 0.00 |

**Table B7: Particle size analysis of ball mill discharge for the test ore (Copper ore)**

| S/No | Size Fraction ($\mu m$) | Sieves, ($\mu m$) | Mass (g) | Mass (%) | Cum. Mass % Retained | Cum. Mass % Passing |
|---|---|---|---|---|---|---|
| 1 | – 355 + 250 | 355 | 14.424 | 7.212 | 0.00 | 100.00 |
| 2 | – 250 + 180 | 250 | 18.312 | 9.156 | 7.21 | 92.79 |
| 3 | – 180 + 125 | 180 | 18.753 | 9.3765 | 16.37 | 83.63 |
| 4 | – 125 + 90 | 125 | 17.075 | 8.5375 | 25.74 | 74.26 |
| 5 | – 90 + 50 | 90 | 32.448 | 16.22 | 34.28 | 65.72 |
| 6 | – 50 | 50 | 98.989 | 49.4945 | 50.51 | 49.49 |
| Total | | | 200 | | 100.00 | 0.00 |

**Table B8: Particle size analysis of feed (reference ore - granite) for grindability test**

| S/No | Size Fraction ($\mu m$) | Sieves, ($\mu m$) | Mass (g) | Mass (%) | Cum. Mass % Retained | Cum. Mass % Passing |
|---|---|---|---|---|---|---|
| 1 | – 355 + 250 | 355 | 40.099 | 20.0495 | 0.00 | 100.00 |
| 2 | – 250 + 180 | 250 | 36.959 | 18.4795 | 20.05 | 79.95 |
| 3 | – 180 + 125 | 180 | 36.007 | 18.0035 | 38.53 | 61.47 |
| 4 | – 125 + 90 | 125 | 18.098 | 9.0490 | 56.53 | 43.47 |
| 5 | – 90 + 50 | 90 | 18.567 | 9.2800 | 65.58 | 34.42 |
| 6 | – 50 | 50 | 50.27 | 25.135 | 74.87 | 25.14 |
| Total | | | 200 | | 100.00 | 0.00 |

**Table B9:** Particle size analysis of ball mill discharge for the reference ore (granite)

| S/No | Size Fraction ($\mu m$) | Sieves, ($\mu m$) | Mass (g) | Mass (%) | Cum. Mass % Retained | Cum. Mass % Passing |
|---|---|---|---|---|---|---|
| 1 | – 355 + 250 | 355 | 15.772 | 7.886 | 0.00 | 100.00 |
| 2 | – 250 + 180 | 250 | 17.95 | 8.975 | 7.89 | 92.11 |
| 3 | – 180 + 125 | 180 | 23.075 | 11.5375 | 16.86 | 83.14 |
| 4 | – 125 + 90 | 125 | 16.519 | 8.2595 | 28.40 | 71.60 |
| 5 | – 90 + 50 | 90 | 35.542 | 17.77 | 36.66 | 63.34 |
| 6 | – 50 | 50 | 91.142 | 45.571 | 54.43 | 45.57 |
| Total | | | 200 | | 100.00 | 0.00 |

**Table B10: EDS of the copper ore sample**

| Element Number | Element Symbol | Element Name | Confidence | Concentration | Error |
|---|---|---|---|---|---|
| 38 | Sr | Strontium | 100.0 | 13.5 | 1.2 |
| 16 | S | Sulfur | 100.0 | 8.7 | 1.2 |
| 83 | Bi | Bismuth | 100.0 | 23.0 | 1.6 |
| 29 | Cu | Copper | 100.0 | 3.5 | 1.5 |
| 14 | Si | Silicon | 100.0 | 3.2 | 1.8 |
| 8 | O | Oxygen | 100.0 | 36.8 | 1.5 |
| 26 | Fe | Iron | 100.0 | 7.5 | 1.8 |
| 7 | N | Nitrogen | 100.0 | 3.8 | 4.7 |

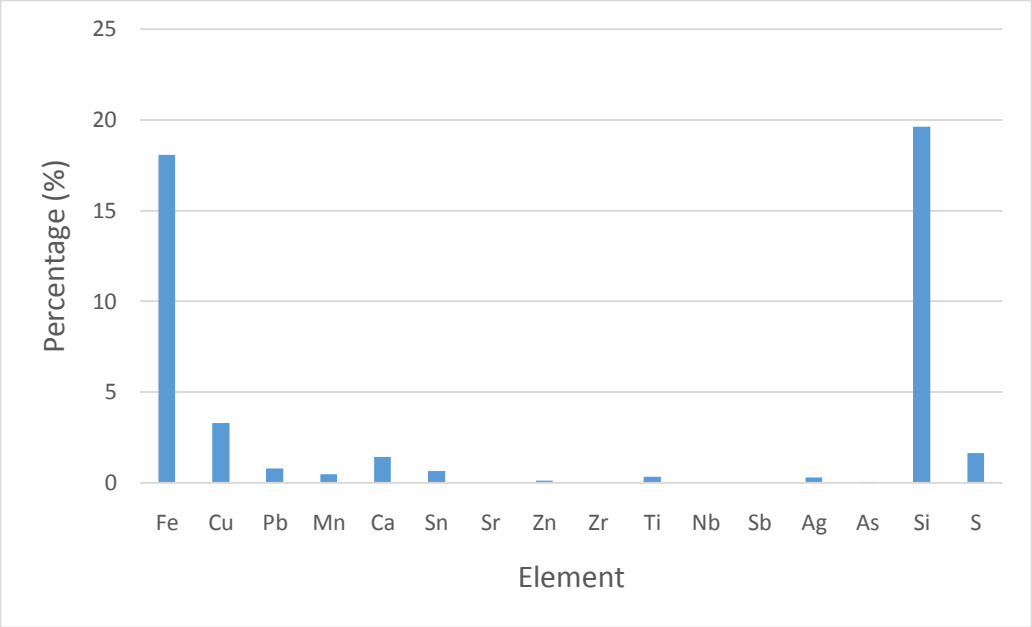



**Figure B1: The Chemical Analysis of the Head Sample (Azara Copper Ore)**

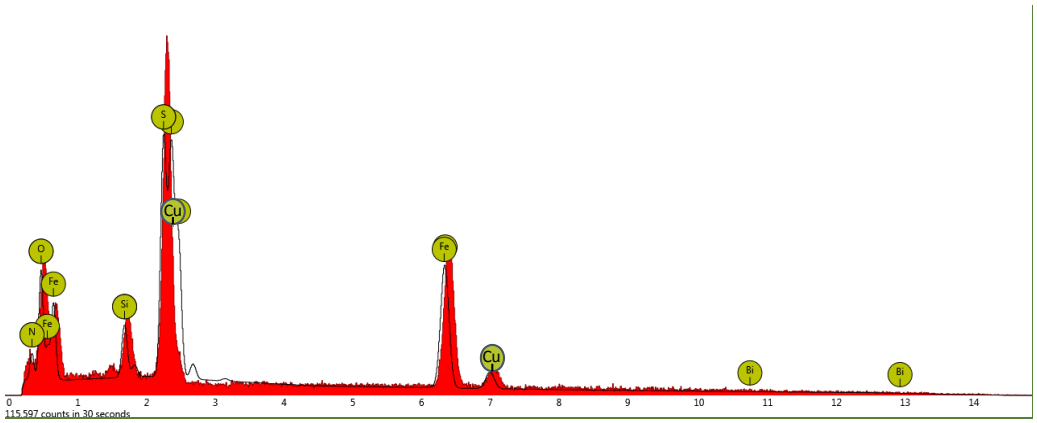



**Figure B2: Diffractogramof Head Sample from the SEM/EDS**

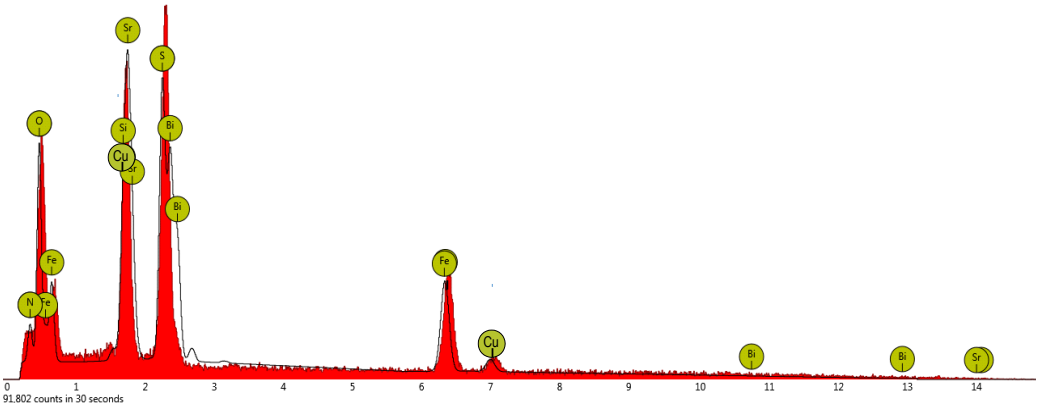



**Figure B3: Diffractogram of −50µm Sample from the SEM/EDS**

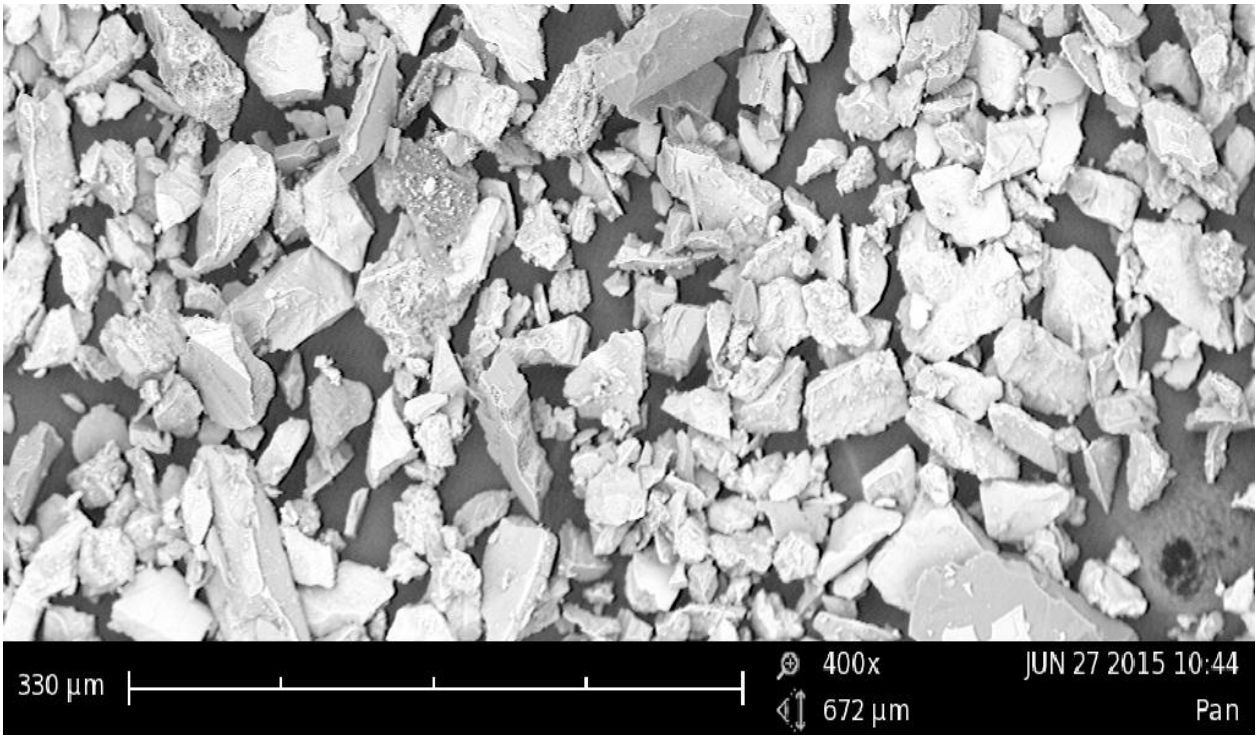



**Plate B1: SEM image of the copper ore sample (Pan)**

**Table B11: EDS Result of the Copper Ore Sample (Pan)**

| Element Number | Element Symbol | Element Name | Confidence | Concentration | Error |
|---|---|---|---|---|---|
| 26 | Fe | Iron | 100.0 | 16.5 | 1.3 |
| 83 | Bi | Bismuth | 100.0 | 29.1 | 1.1 |
| 29 | Cu | Copper | 100.0 | 6.7 | 1.5 |
| 16 | S | Sulfur | 100.0 | 9.4 | 1.1 |
| 8 | O | Oxygen | 100.0 | 29.2 | 1.8 |
| 14 | Si | Silicon | 100.0 | 2.2 | 2.7 |
| 7 | N | Nitrogen | 100.0 | 3.8 | 4.8 |

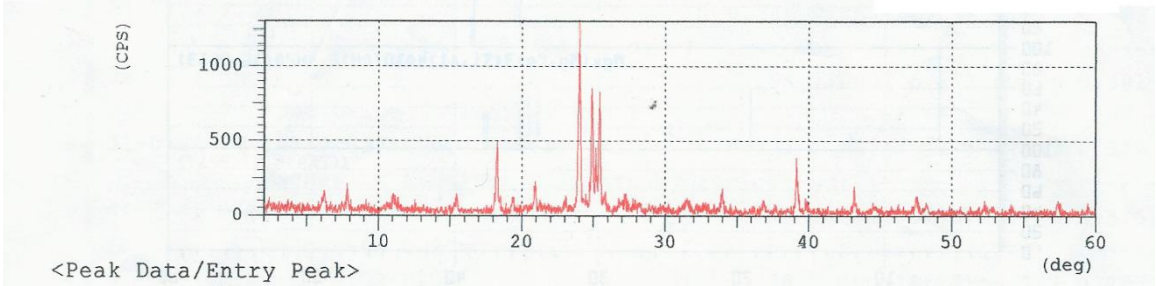
## Appendix C: XRD Result of Head Sample

********** SEARCH / MATCH RESULT **********

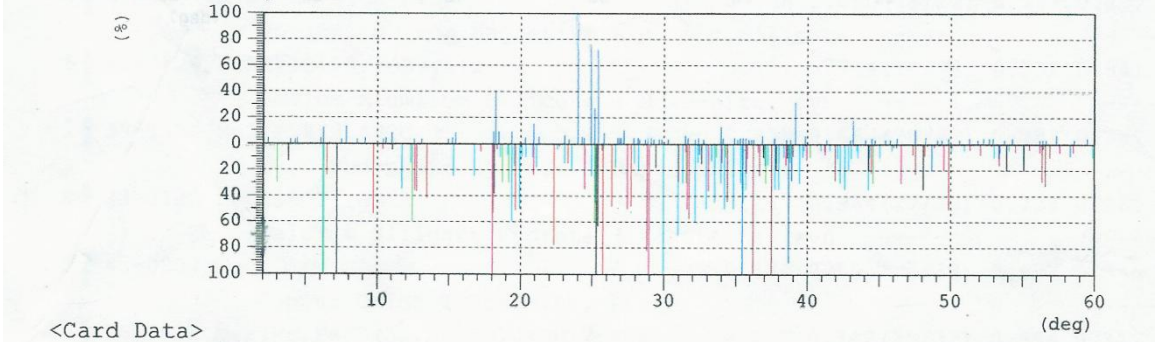
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Data Name : HEAD SAMPLE
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Sample Name :
Comment : GREYISH
Date & Time : 08 - 17 - 15 12:56:33

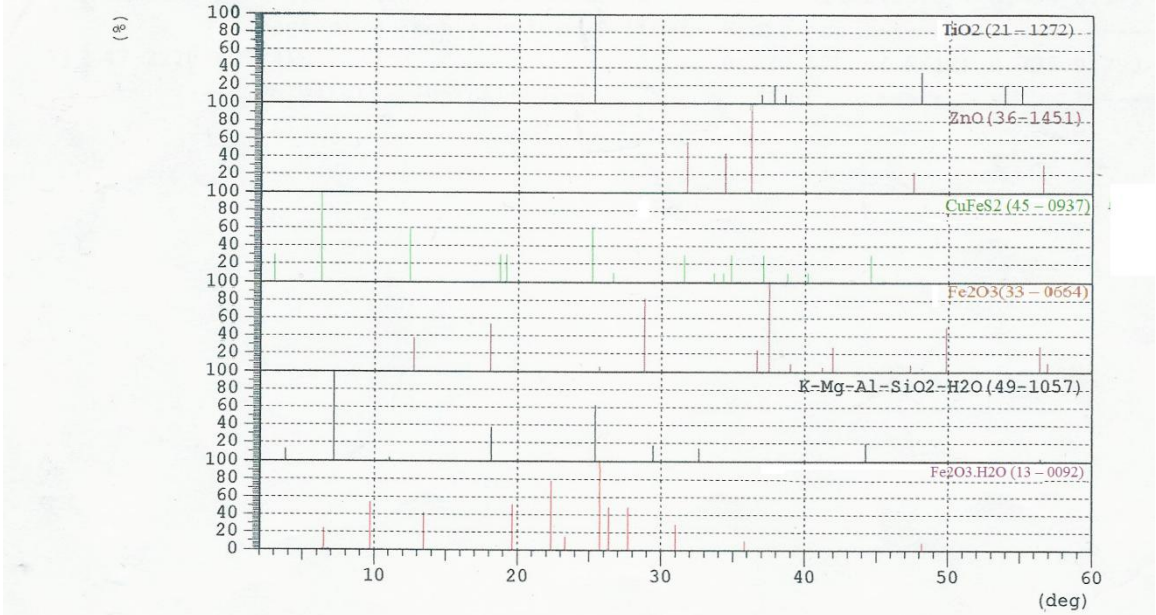
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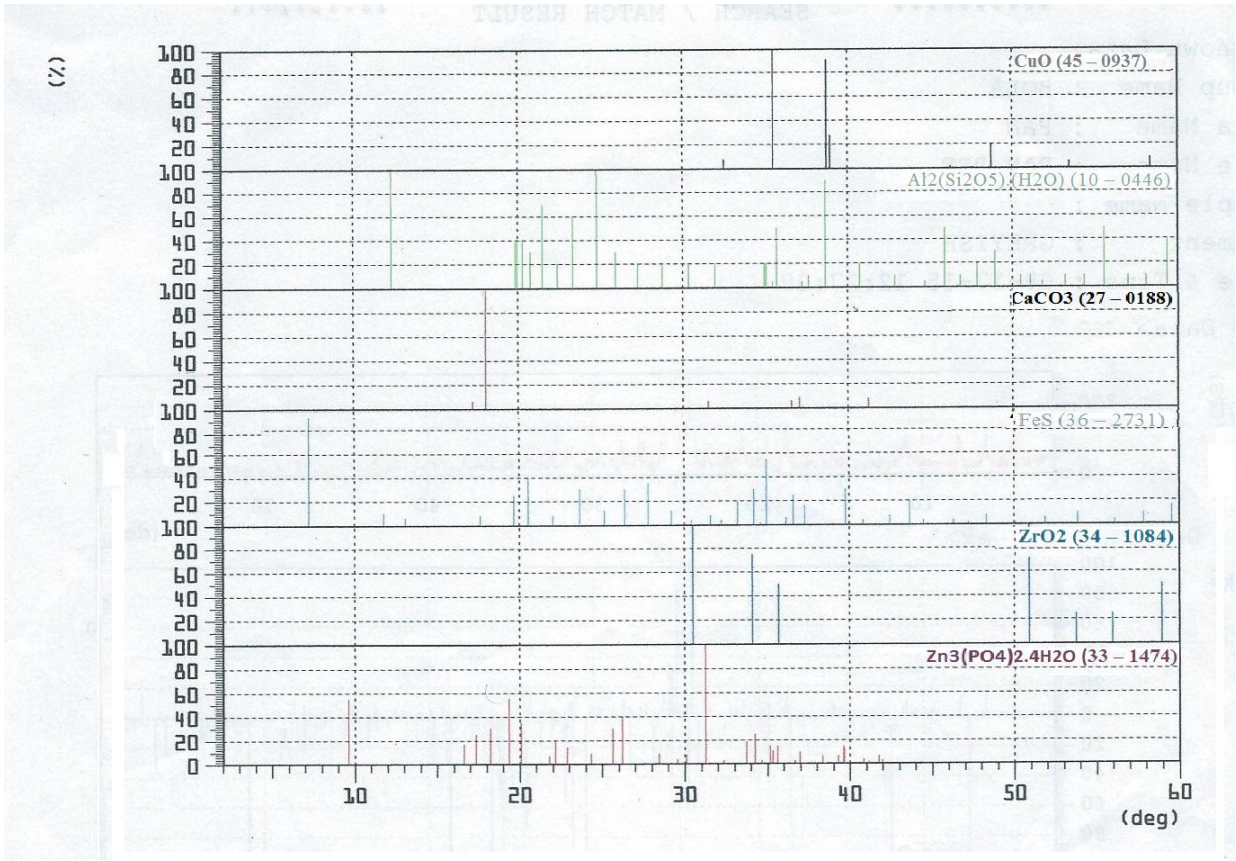



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(%)
CuO (45 – 0937)
Al2(Si2O5)(H2O) (10 – 0446)
CaCO3 (27 – 0188)
FeS (36 – 2731)
ZrO2 (34 – 1084)
Zn3(PO4)2.4H2O (33 – 1474)
(deg)