

# Determination of liberation size of Akiri copper ore, Nasarawa state, north-central Nigeria

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**Abstract** - This research work focused on the Determination of Liberation Size of Akiri Copper Ore in Nasarawa state. The techniques involved in the study were sample collection and preparation, sieve analysis and chemical analysis of the head sample. The mesh of grind was found to be 210  $\mu\text{m}$  and the liberation size was established to be 125  $\mu\text{m}$  sieve size having the highest percentage of copper of 11.9 and 14.9% copper oxide. The study recommended further investigation on the separation of the copper ore using 125  $\mu\text{m}$  sieve size.

## I. INTRODUCTION

The enormous growth of industrialization from the eighteenth century onward led to dramatic increases in the annual output of most mineral commodities, particularly metals. Copper output grew by a factor of 27 in the twentieth century alone (Wills, 2006). Minerals by definition are natural inorganic substances possessing definite chemical compositions and atomic structures. Many minerals exhibit isomorphism, where substitution of atoms takes place without affecting the atomic structure (Wills, 2006). Other minerals exhibit polymorphism; different minerals having the same chemical composition but markedly different physical properties due to a difference in crystal structures.

There are two fundamental operations in mineral processing namely; the release or liberation of the valuable minerals from their waste gangue minerals and separation of these values from the gangue, this latter process being known as concentration. Therefore, the liberation of the valuable minerals from the gangue is accomplished by comminution and one of the major objectives of comminution is the liberation or release of the valuable minerals from the associated gangue minerals at the coarsest possible particle size, if such an aim is achieved, then not only is energy saved by the reduction of the amount of fines produced but any subsequent separation stages become easier and cheaper to operate. If high – grade solid products are required then good liberation is essential, (Wills and Atkinson, 1993).

Copper as in the case of most minerals, occurs finely disseminated and intimately associated with gangues, which they must be initially “unlocked” or “liberated” before separation can be undertaken. This is achieved by comminution, in which the particle size of the ore is progressively reduced until the clean particles of mineral can be separated by such methods as are available (Knecht, 1994).

Comminution in the mineral processing plant or “mill” takes place as a sequence of crushing and grinding processes. Crushing reduces grinding which can be carried out until the mineral and gangue are substantially produced as separate particles (Flavel, 1978).

Crushing is the first mechanical stage in the process of comminution in which the main objective is the liberation of the valuable minerals from the gangue (Lewis et al, 1976). Crushing is accomplished by compression of the ore against rigidly constrained motion path. This is contrasted with grinding which is accomplished by abrasion and impact of the ore by the free motion of unconnected media such as rods, balls or pebbles.

Grinding is the last stage in the process of comminution, in this stage the particles are reduced in size by a combination of impact and abrasion, either dry or in suspension in water. It is performed in rotating cylindrical steel vessels which contain a charge of loose crushing bodies. (The grinding medium) which is free to move inside the mill, thus comminuting the ore particles. According to the ways by which motion is imparted to the charge, grinding mills are generally classified into two types: tumbling mills and stirred mills (Wills 2006). Size analysis of the various products after comminution of a concentrator constitutes a fundamental part of laboratory testing procedure. It is of great importance in determining the quality of grinding and in establishing the degree of liberation of the values from the gangue of various particle sizes (Barber, 1972).

This is done, primarily to establish that particle size(s) at which the various grains in an ore happened to be “freest” of each other, thus setting the stage for effective and efficient separation process. This particular particle size is referred to as liberation size, which also should be at the coarsest possible size to avoid energy waste through over grinding.

Most minerals are finely disseminated and intimately associated with gangue; they must be intimately unlocked or liberated before separation can be undertaken. Therefore, the study is geared towards establishing the liberation or release of the ore from the associated gangue minerals at the coarsest possible particle size. If such an aim is achieved, then not only is energy saved by the reduction of the amount of fines produced, but any subsequent separation stage become easier and cheaper to operate.

The research study will establish the particle size at which the various grains in an ore happened to be free of each other, thus setting the stage for effective and efficient separation process.

Liberation size of an ore is very significant component in any process design as it gives the operators a clear view of the sieve size, the grinding operation should target. This information is of great importance as it avoid over grinding and hence saves a great deal of cost.

## II. LOCATION OF THE STUDY AREA

The study area is Akiri copper ore deposit in Azara in Awe local government area of Nasarawa State. Akiri copper ore deposit is located between the longitudes of N 08° 22' 783" and E 009° 21' 537". The deposit is located in Akiri, which is 110km from Lafia 3km away from Emo Asharpura Energy and mining company limited of Azara, Nasarawa State of Nigeria. Nasarawa state is well accessible due to its largest boundary with the federal capital territory (F.C.T.) Abuja, but the terrain from Lafia to Akiri is rough (Aderotoye, 1998).

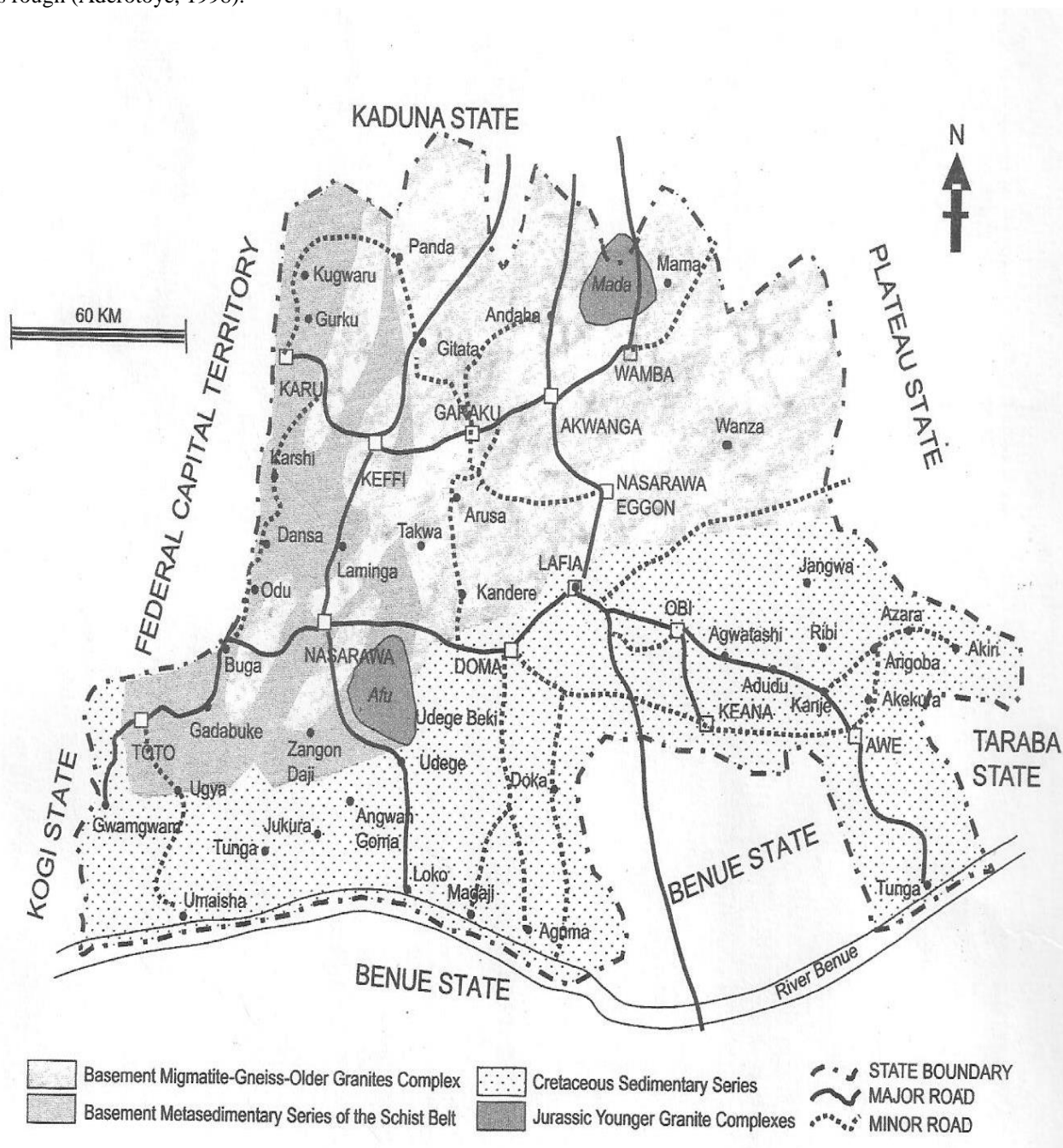


Figure 2.1: Map of Nasarawa State showing Akiri Village

## III. GEOLOGY OF THE STUDY AREA

Akiri is located in Awe local government area in Nasarawa State. It is made up of sedimentary rocks, Benue trough is a zone of sedimentary accumulated formed when track of basement subsided to form a rift valley in the early cretaceous time. The major rock form within the area of rocks was of sedimentary origin.

The sedimentary that were accumulated in the trough are sequences of sandstone, clays, shale's and limestone. The sediment have been affected by trough both burial incident by earth movement, high heat flow accompanying igneous intrusion and hydrothermal vein emplacement, it was probably during the migration of the oceanic or continental brume (Hydrothermal processes) that the ores were deposited with the sedimentary rock (Adetoro, 1998).

#### IV. COMMINATION

Because most minerals are finely disseminated and intimately associated with gangue, they must be intimately “unlocked” or “liberated” before separation can be undertaken. This is achieved by comminution in which the particle size of the ore progressively reduced until the clean particles of mineral can be separated by such methods as are available.

Comminution in its earliest stages is carried out in order to make the freshly excavated material easier to handle by scrapers, conveyors, and ore carriers, and in the case of quarry products to produce materials of controlled particle size. Explosives are used in mining to remove ores from their natural beds, and blasting can be regarded as the first stage in comminution, (Wills, 2006).

Comminution in the minerals processing plant or “mill” takes place as a sequence of crushing and grinding process. Crushing reduces the particle size of run-of-mine ore to such a level that grinding can be carried out until the mineral and the gangues are substantially produced as separated particles.

Crushing is accomplished by compressing of the ore against rigid surface, or by impact against surface in a rigidly constrained motion path. This is contrasted with grinding which is accomplished by abrasion and impact of the ore by the free motion of unconnected media such as rods, balls or pebbles.

Crushing is usually a dry process, and is performed in several stages, reduction ratio being small, ranging from three to six in each stage. The reduction ratio of a crushing stage can be defined as the ratio of maximum particle size entering to maximum particle size leaving the crusher, although other definitions are sometimes used.

Tumbling mills with steel rods or balls, sized ores as the grinding media are used in the last stages of comminution. Grinding is usually performed “wet” to provide a slurry feed to the concentration process. Dry grinding has limited applications. There is an overlapping size area where it is possible to crush or grind the ore. From a number of case studies, it appears that at the fine end of crushing operations equivalent reduction can be achieved for roughly half the energy and costs required by tumbling mills (Flavel, 1978).

##### *Principles of Comminution*

Most minerals are crystalline materials in which the atoms are regularly arranged in three dimensional arrays. The configuration of atoms is determined by the size and types of physical and chemical bonds holding them together. In the crystalline lattice of minerals, these inter-atomic bonds are effective only over small distances, and can be broken if extended by a tensile stress. Such stresses may be generated by tensile or compressive loadings. (Figure 2.2below):

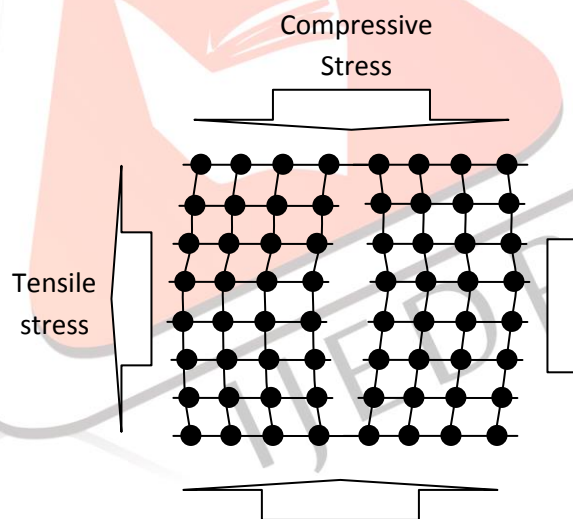


Figure 2.2: Strain of a crystal lattice resulting from tensile or compressive stresses

Even when rocks are uniformly loaded, the internal stresses are not evenly distributed, as the rock consists of a variety of minerals dispersed as grains of various sizes. The distribution of stress depends upon mechanical properties of the individual minerals, but more importantly upon the presence of cracks or flaws in the matrix, which act as sites for stress concentration (Figure 2.3).



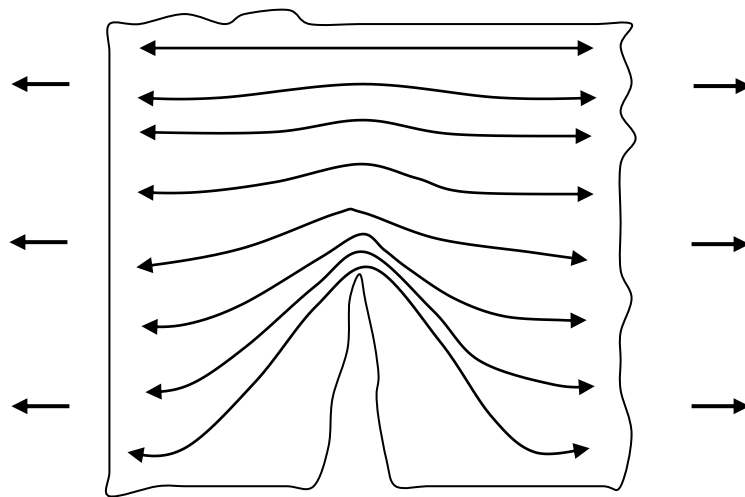


Figure 2.3: Stress concentration at a crack tip

Wills (2007) found in (Inglis, 1913) that the increase in stress at such a site is proportional to the square root of the crack length perpendicular to the stress direction. Therefore, there is a critical value for the crack length at any particular level of stress at which the increased stress level at the crack tip is sufficient to break the atomic bond at that point. Such rupture of the bond will increase the crack length, thus increasing the stress concentration and causing rapid propagation of the crack through the matrix, thus causing fracture.

Although the theories of comminution assume that the material is brittle, crystals can, in fact, store energy without breaking, then release the energy when the stress is removed. Such behavior is known as elastic. When fracture does occur, some of the stored energy is transformed into free surface energy, which is potential energy of atoms at the newly produced surfaces. Due to this increase in surface energy, newly formed surfaces are often more chemically active, and are more amenable to the action of flotation reagents, as well as oxidation more readily.

### Particle Size Analysis

Particle size analysis of the various products of a concentrator constitutes a fundamental part of laboratory testing procedure. It is of great importance in determining 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 used to determine the optimum size of the feed process for maximum efficiency and to determine the size range at which any losses are occurring in the plant, so that they may be reduced. It is essential, therefore, that methods of size analysis must be accurate and reliable, as important changes in plant operation may be made on the small amounts of material are used in the sizing test, it is essential that sample is representative of bulk material and the same care should be taken over sampling for the size analysis as for assay.

### Sieve Analysis

Sieve analysis is one of the oldest methods of size analysis and is accomplished by passing a known weight of sample material successively through fine sieve weight the amount collected on each sieve to determine the percentage weight in each sieve fraction sieving is carried out with wet or dry materials and the sieve are usually agitated to expose all the particle to openings.

Sieving when applied to irregular shape particles, is complicated by the fact that a particle with a size near that of the nominal aperture of the test sieve may pass only when presented in a favourable position. As there is inevitable a variation in the size of sieve apertures, due to irregularity of weaving, prolonged sieving will cause the larger apertures to exert an unduly large effect on the sieve analysis. Given time, every particle small enough could find its way through a few such holes. The procedure is also complicated in many cases by the presence of near size –particle which cause blinding, or obstruction of the sieve apertures, and reduce the effective area of the sieving medium. Blinding is most serious with test sieve of very small aperture size.

The process of sieving may be divided into two stages: first, the elimination of particle considerably smaller than the screen apertures, which should occur fairly rapidly and the second, the separation of the so-called “near-size” particles which is a gradual process rarely reaching final completion. Both stages require the sieve to be manipulated in such a way that all particles have opportunities for passing the apertures and so that any which blind an aperture may be removed from it. Ideally, each particle should be presented individually to an aperture, as is permitted for the largest aperture sizes, but for most sizes this is impractical.

The effectiveness of a sieving test depend on the amount of materials put on the sieve (the “Charge”) and the type of movement imparted on to the sieve, a comprehensive account of sampling techniques for sieving is given in BS 1017 – (Anon.,1989). Basically, if the charge is too large, the bed of material will be too deep to allow each one a chance to meet an aperture in the most favourable position for sieving in a responsible time. The charge, therefore, is limited by a requirement the end of the sieving appropriate to the aperture size. On the other hand, the sample must contain enough particles to be representative of the bulk, so a minimum size of sample will have to be subdivided into number of charges if the requirement for preventing over-loading of the sieves is to be satisfied.

### Liberation

One of the major objectives of comminution is the liberation, or release of the valuable minerals from the associated gangue minerals at the coarsest possible particle size. If such an aim is achieved, then not only is energy saved by the reduction of the amount of fines produced, but any subsequent separation stages become easier and cheaper to operate. If high-grade solid products are required, then good liberation is essential; however, for subsequent hydrometallurgical processes, such as leaching it may only be necessary to expose the required mineral.

In practice complete liberation is seldom achieved, even if the ore is ground down to the grain size of the desired mineral particles. This is illustrated by Figure 2.5, which shows a lump of ore which has been reduced to a number of cubes of identical volume and of a size below that of the grain of mineral observed in the original ore sample. It can be seen that each particle produced containing mineral also contains a portion of gangue; complete liberation has not been attained; the bulk of the major mineral - the gangue - has, however, been liberated from the minor mineral - the value.

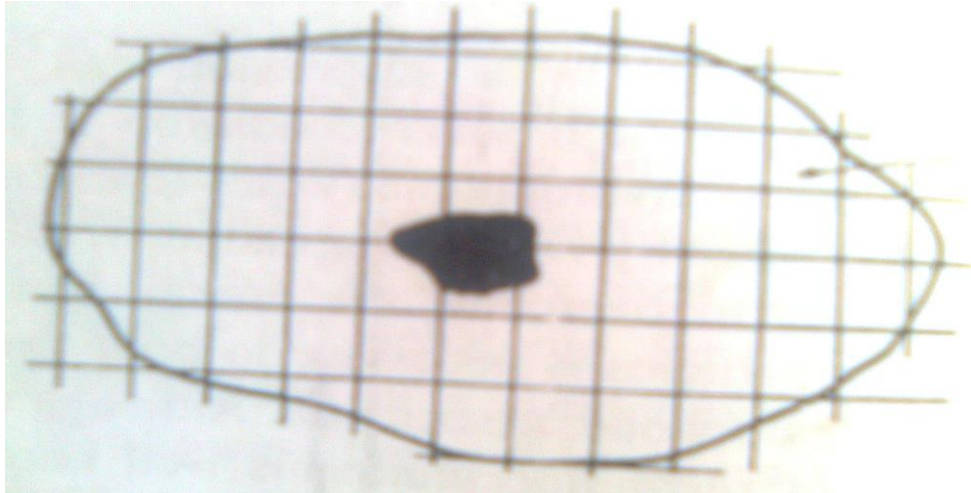


Figure 2.5 “Locking” of mineral and gangue

The particles of “locked” mineral and gangue are known as middlings and further liberation from this fraction can only be achieved by further comminution.

The “degree of liberation” refers to the percentage of the mineral occurring as free particles in the ore in relation to the total content. This can be high if there are weak boundaries between minerals and gangue particles, which is often the case with ores composed mainly of rock-forming minerals, particularly sedimentary minerals. Usually, however the adhesion between mineral and gangue is strong and, during comminution, the various constituents are cleft across. This produces much middlings and a low degree of liberation. New approaches to increasing the degree of liberation involve directing the breaking stresses at the mineral crystal boundaries, so that the rock can be broken without breaking the mineral grain (Wills and Atkinson, 1993).

Many researchers with a view to predicting the behavior of particles in a separation process (Barberry, 1991). The first attempt at the development of a model for the calculation of liberation was made by Gaudin (1939; King (1982) developed an exact expression for the fraction of particles of a certain size that contained less than a prescribed fraction of any particular mineral. These models, however, suffered from many unrealistic assumptions that must be made with respect to the grain structure of the minerals in the ore, in particular that liberation is preferential, and in 1988 (Austin and Luckie) concluded that “there is no adequate model of liberation of binary systems suitable for incorporation into a mill model”. For this reason, liberation models have not found much practical application. However, some fresh approaches by Gay, allowing multi-mineral systems to be modeled (not just binary systems) free of the assumptions of preferential breakage, have recently demonstrated that there may yet be a useful role for such models (Gray, 2004a,b). The quantification of liberation is now routinely possible using the dedicated scanning electron microscope MLA and QEMSCAN systems mentioned earlier, and concentrators are increasingly using such systems to monitor the degree of liberation of their processes.

It should also be noted that a high degree of liberation is not necessary in certain processes, and, indeed, may be undesirable. For instance, it is possible to achieve a high recovery of values by gravity and magnetic separation even though the valuable minerals are completely enclosed by +gangue, and hence the degree of liberation of the values is zero. As long as a pronounced density or magnetic susceptibility difference is apparent between the locked particles and the free gangue particles, the separation is possible by intensive fine grinding, which may reduce the particles to such a fine size that separation becomes very inefficient. On the other hand, froth flotation requires as much of the valuable mineral surface as possible to be exposed, whereas in a chemical leaching process, a portion of the surface must be exposed to provide a channel to the bulk of the mineral.

In practice, ores are ground to an optimum grind size, determined by laboratory and pilot scale test work, to produce an economic degree of liberation. The concentration process is then designed to produce a concentrate consisting predominantly of valuable mineral, with an accepted degree of locking with the gangue mineral, and a middlings fraction, which may require further grinding to promote optimum release of the minerals. The tailings should be mainly composed of gangue minerals.

Figure 2.5 is a cross-section through a typical ore particle, and illustrates effectively the liberation dilemma often facing the mineral processor. Regions A represent a valuable mineral, and region AA is rich in valuable mineral, ranging from fully liberated mineral and gangue particles, to those illustrated. Particles of type 1 are rich in mineral, and are classed as concentrate as they

have an acceptable degree of locking with the gangue which limits the concentrate grade. Particles of type 4 would likewise be classed as tailings, the small amount of mineral present reducing the recovery of mineral into the concentrate. Particles of types 2 and 3, however, would probably be classed as middlings, although the degree of regrinding needed to promote economic liberation of mineral from particle 3 would be greater than in particle 2.

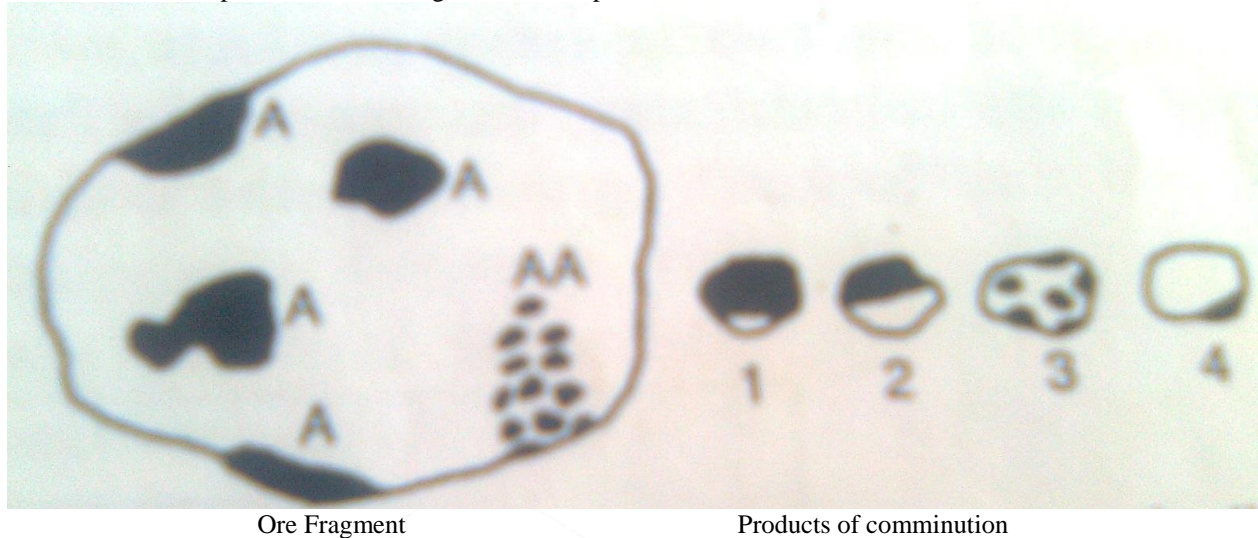


Figure 2.6 Cross-section of ore particles

During the grinding of a low-grade ore the bulk of the gangue minerals is often liberated at a relatively coarse size (see figure 2.6). In certain circumstances it may be economic to grind to a size much coarser than the optimum in order to produce in the subsequent concentration process a large middlings fraction and tailings which can be discarded at a coarse grain size. The middlings fraction can then be reground to produce a feed to the final concentration process.

This method discards most of the coarse gangue early in the process, thus considerably reducing grinding costs, as needless comminution of liberated gangue is avoided. It is often used on minerals which can easily be separated from the free gangue, even though they are themselves locked to some extent with gangue.

## V. METHODOLOGY

In order to determine the liberation size of Akiri copper ore the following methods were adopted:

1. Sample Collection
2. Sample Preparation
3. Sieve Analysis
4. Chemical Analysis
5. Instrumentation

### *Sample Collection*

In order to have a true fraction representation of copper ore from the bulk, samples were collected from different points at different depth ranges using the random sampling techniques within the study area.

### *Sample Preparation*

Since the sample from the field is usually bulky. It was necessary to reduce to smaller sizes. Therefore crushing and grinding was carried out followed by sampling with the aid of a sample splitter. The Jones riffle was used as a splitter. It is an open v-shaped box in which a series of chutes was mounted at right angles to the long axis to give a series of rectangular slots of equal areas alternatively feeding two trays placed on either side of the trough. The sample was poured into the chute and splitted into equal portions by the slots until after repeated cycles as sample of the desired size was obtained.

### *Sieve Analysis*

200g of the prepared sample was subjected to sieve analysis. The arrangement of the sieves were done using a sieve scale in which the ratio of the aperture widths of adjacent sieves is the square root of two [ $\sqrt{2} = 1.414$ ]. Sieve sizes from 250 $\mu$ m to 63 $\mu$ m were arranged in a stack with the coarsest sieve on the top and the finest at the bottom. A tight fitting pan was placed below the bottom sieve to receive the final undersize and a lid was placed on top of the coarsest sieve to prevent escape of the sample. The arranged sieves were placed in a sieve shaker which vibrates the material vertically. The duration of the screening was controlled to 30 minutes by an automatic timer.

During the shaking, the undersize material falls through successive sieves until it is retained on a sieve having apertures which were slightly smaller than the diameter of the particles. After a successful operation each size fraction retained on each sieve was collected weighed and value recorded.

### *Chemical analysis*



Mini Pal was used which is a compact energy dispersive x-ray spectrometer designed for the elemental analysis of a wide range of samples. The system is controlled by a PC running the dedicated mini analytical software.

The Mini pal 4 version in use is PW 4030 x-ray spectrometer, which is an energy dispersive microprocessor controlled analytical instrument designed for the detection and measurement of elements in a sample (solids, powders and liquids), from sodium to uranium.

The pellet is loaded in the sample chamber of the spectrometer and voltage (30kV maximum) and a current (1mA maximum) is applied to produce the x-rays to excite the sample for a preset time (10 mins. in this case). The spectrum from the sample is now analyzed to determine the concentration of the elements in the sample.



Figure 2.7 Plate 1: Laboratory Sieve Shaker

## VI. RESULTS

The results of various laboratory experiments and analysis carried out were presented and analyzed in this chapter, which include:

- i. Sieve test result
- ii. Results of the chemical analysis of the head sample

**Sieve Test Result**

Table 1: Result of Sieve Analysis

Size Range (µm)	Weight (g)	Weight (%)	Nominal (µm) Aperture size	Cumulative under size (%)	Cumulative over size (%)
+ 250	24.2	12.10	250	87.90	12.10
-250 + 180	35.0	17.50	180	70.40	29.60
-180 + 125	37.2	18.60	125	51.80	48.20
-125 + 80	35.4	17.70	80	34.10	65.90
-80 + 63	39.2	19.60	63	14.50	85.50
-63	29.0	14.50	-	14.50	100.00

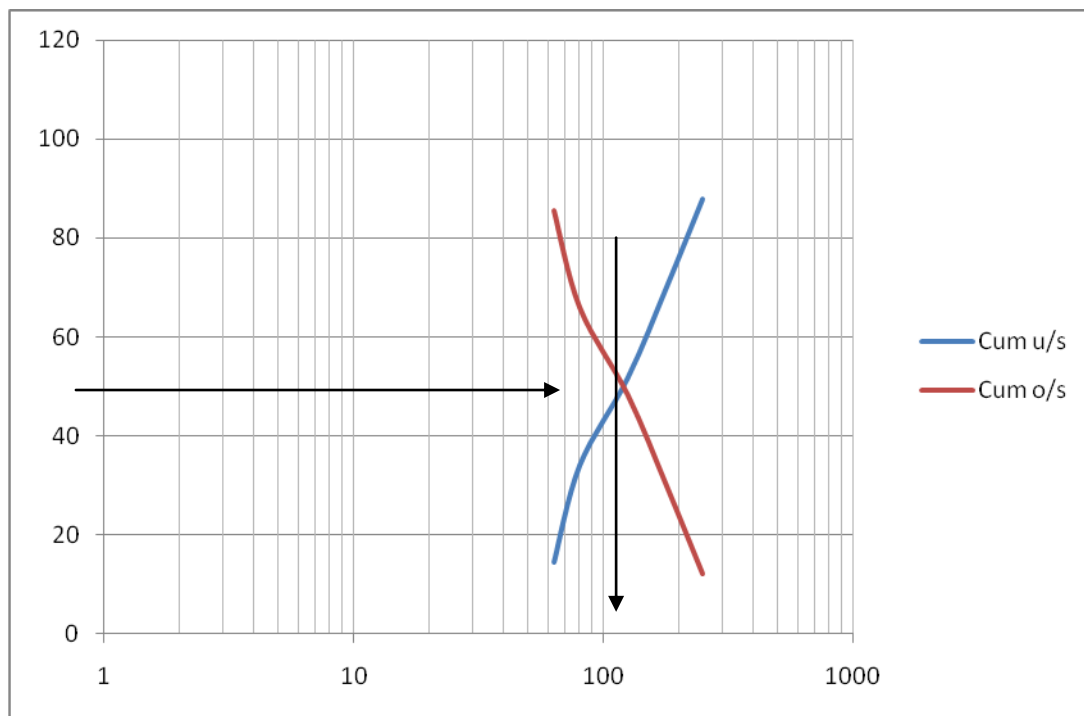


Fig. 4.1: Particle Size Distribution Graph

Table 4.3: Result of the Chemical Analysis of Head Sample in Element form

Aperture Size (µm)	Al	Si	S	K	Ca	Ti	Mn	Cr	Fe	Ni	Cu
250	4.6	11.3	6.7	0.48	2.04	0.24	0.18	0.03	23.80	0.068	9.20
180	3.7	11.7	7.1	0.33	1.49	0.12	0.16	0.03	24.11	0.077	10.54
125	3.7	11.7	7.1	0.33	1.00	0.10	0.16	0.04	22.57	0.102	11.90
80	3.8	12.0	6.8	0.44	1.96	0.19	0.15	-	22.36	0.079	10.14
63	4.0	11.8	6.8	0.44	1.12	0.13	0.13	0.03	22.92	0.060	11.02

Table 4.2: Result of Chemical Analysis of the Head sample in oxide form

Aperture Size(µm)	Al <sub>2</sub> O <sub>3</sub>	SiO <sub>2</sub>	SO <sub>3</sub>	K <sub>2</sub> O	CaO	TiO <sub>2</sub>	MnO	Cr <sub>2</sub> O <sub>3</sub>	Fe <sub>2</sub> O <sub>3</sub>	NiO	CuO
250	8.6	24.2	16.7	0.58	2.85	0.40	0.23	0.04	34.0	0.087	11.5
180	6.9	25.0	17.8	0.40	2.08	0.20	0.21	0.051	34.5	0.098	13.2
125	6.9	25.0	17.8	0.40	1.37	0.17	0.21	0.056	32.3	0.130	14.9
80	7.2	25.7	17.0	0.53	2.74	0.32	0.20	-	32.0	0.100	12.7
63	7.5	25.2	17.0	0.44	1.56	0.21	0.17	0.048	32.8	0.076	13.8

**Discussion of Results**

It was observed from the sieve test result presented (table 4.2) that 24.2g of the total weight (200g) was retained on the 250µm sieve size, 35.0g on 180µm, 37.2g on 125µm, 35.4g on 80µm and 39.2 on 63µm. A graph of cumulative weight, percentage undersize and cumulative weight percentage oversize was plotted against the particle size. From the graph, the mesh of grind was 210µm and the median size was 50%. The chemical analysis of the sample was conducted on all the five sieve sizes. The result in its elemental form indicated that 250µm contains 4.6%Al, 11.3%Si, 23.8%Fe and 9.2%Cu e.t.c., 180µm contains 3.7%Al,



11.7%Si, 24.11%Fe and 10.54%Cu. 125 $\mu$ m contains 3.7%Al, 11.7%Si, 22.57%Fe and 11.9%Cu. 80 $\mu$ m contains 3.8%Al, 12.0%Si, 22.36%Fe and 10.14%Cu, while 63 $\mu$ m contains 4.0%Al, 11.8%Si, 22.92%Fe and 11.02%Cu.

The result in its oxide form indicated that 250 $\mu$ m contains 8.6% Al<sub>2</sub>O<sub>3</sub>, 24.2% SiO<sub>2</sub>, 34.0% FeO and 11.5% CuO. 180 $\mu$ m contains 6.9% Al<sub>2</sub>O<sub>3</sub>, 25.0% SiO<sub>2</sub>, 34.5% FeO and 13.2% CuO. 125 $\mu$ m contains 6.9% Al<sub>2</sub>O<sub>3</sub>, 25.0% SiO<sub>2</sub>, 32.3% FeO and 14.9% CuO. 80 $\mu$ m contains 7.2% Al<sub>2</sub>O<sub>3</sub>, 25.7% SiO<sub>2</sub>, 32.0% FeO and 12.7% CuO, and 63 $\mu$ m contains 7.5% Al<sub>2</sub>O<sub>3</sub>, 25.2% SiO<sub>2</sub>, 32.8% FeO and 13.8% CuO. The result has confirmed 125 $\mu$ m to be liberation size of Akiri copper ore, upholding the conclusion by (Abubakar 2009), (Habila 2009) and Usaini (2010).

## VII. CONCLUSION AND RECOMMENDATION

### Conclusions

The research study established that:

1. The mesh of grind and the median size was 210 $\mu$ m and 50% respectively for Akiri copper ore.
2. The liberation size is 125 $\mu$ m sieve size having the highest recovery of copper in its elemental and oxide form, of 11.4 and 14.9 percent respectively.

### Recommendation

Base on the result of the research work, it is recommend that further investigation on the separation of the copper ore using 125 $\mu$ m sieve size.

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