Liberation Size and Beneficiation of Arufu Laed Ore, Nasarawa State, North-Central Nigeria

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Abstract -**The lead ore sample was obtained from Arufu mining site, Nasarawa State, Nigeria. The ore was pulverized, sieved to liberation size and beneficiated. The mesh of grind was found to be 198μm and liberation size established to be 63μm sieve size having the highest percentage of lead of 17.3% then upgraded to 53.3%Pb by froth flotation method and was assessed by using x-ray fluorescence (XRF).The lead concentrate by weight was obtained to be 65%, confirmed being of high economic grade having surpassed 60% wt Pb element in an ore. The study recommended further investigation to be done especially to establish the reserve estimate of the deposit.**

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I. INTRODUCTION

The Nigerian Minerals and Metals Policy produced by the Ministry of Mines and Steel Development (MMSD) reckons that it is a fact of modern international trade that developing countries such as Nigeria stand to gain more from processing their metal ore endowments into primary metal or even final products before sales rather than trading the crude ores. In view of this the focus in the case of lead and zinc ores should be the establishment of smelting plants to produce lead and zinc metals if possible to fabricate the metals into semi-finished/finished products (Anon, 2013).

Determining the liberation size is an intricate mineral processing research area that has been considered very relevant in the choice of processing route.

There are two fundamental operations in mineral processing namely; the release of liberation of the valuable minerals from their waste gangue mineral 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 mineral 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 becomes easier and cheaper to operate. If high grade solid products are required then good liberation is essential. (Wills and Atkinson,1993).

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

II. COMMINUTION

Comminution in the mineral processing plant or "mill" takes place as a sequence of crushing and grinding processes. Crushing reduced 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 objectives 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 vessel which contains 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 procedures. 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 particles sizes (Barber, 1972).

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

Most mineral 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 toward establishing the liberation size at which the valuable mineral is release from the associated gangue. 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.

Principles of Comminuation

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

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

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, the 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 surface. Due to this increase in surface energy, newly formed surface are often more chemically active, and are more amenable to the action of flotation reagents, as well as oxidation more readily.

III. PARTICLE SIZE ANALYSIS

Particle size analysis of the various product of a concentrator is a fundamental part of laboratory testing procedure. It is of great importance in determining the quality of grinding and in establishing the degree of libation of the values from the gangue at various particles sizes.

In the separation stage, size analysis of the product used to determine the optimum six 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.

IV. SIEVE ANALYSIS

Sieve analysis is one of the oldest methods of size analysis and is accomplished by passing a known weight of sample materials 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 particles to openings.

Sieving when applied to irregular shape particle, is complicated by the fact that a particle with a size near that of the nominal aperture of the test sieve may past only when presented in a favorable 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 ways through a few such holes. The procedures is also complicated in many cases by the present of near size particle which cause blinding, or obstruction of the sieve apertures, and reduces 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 considerable 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 individual to an aperture, as is permitted for the largest sizes this is impractical.

The effectiveness of a sieving test depends 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 materials 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 charger 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 chargers if the requirement for preventing over-loading of the sieves is to be satisfied.

V. CONCENTRATION

This is the second fundamental operation in mineral processing concerned with the separation of valuable minerals from the gangue. Once the comminution process is completed, the succeeding operations in mineral processing are taken over by what is known as separation. Regardless of the method or methods used, the aim is always the same to take a natural aggregate of minerals (an ore) and separate it into two or more mineral products. In general, the products of separation are (1) the concentrate which contains the valuable minerals; and (2) the tailings which contain primarily materials of little or no value. It may be borne in mind that minerals have been liberated, either by grinding or chemical means, must usually be 'sized' prior to their separation from each other because the efficiencies of most separation methods are improved when closely sized fractions are used.

A common feature of all separation processes is that they are imperfect – some of the materials of no value contaminate the concentrate to some extent, and some of the minerals of value are always present in the tailings in small amounts (Wills 2006).

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 as well beneficiate the ore by appropriate method of concentration. .

VI. METHODOLOGY

The following techniques were adopted in order to determine the liberation size and beneficiation of Arufu lead ore:
1. sample collection
2. sample preparation
3. sieve analysis
4. chemical analysis
5. froth flotation
6. instrumentation

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

Sample Preparation
The samples collected in lumps size were broken manually with sledge hammer to provide a required size acceptable to laboratory jaw crusher. The samples were crushed and pulverized, then coned and quartered to yield a representative sample

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 materials vertically. The duration of the screening was controlled to 30minutes 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 particle. 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 if PW x-ray spectrometer, which is an energy dispersive microprocessor controlled analytical instrument designed for the detection and measurement of element in a sample (solid, 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 present time (10 mins. In this case). The spectrum from the sample is now analyzed to determine the concentration of the element in the sample.

Concentration by Froth flotation
The following equipment and materials were used:
- Laboratory flotation machine
- Flotation cell
- Beakers
- Scraper
- Collector; sodium ethyl xanthate
- Frother; pine oil
- PH regulator; sodium carbonate(soda ash)
- PH meter

Procedure
a. 100g of the sample was obtained from the sieve size of $63\mu m$ being the liberation size.
b. The sample was empty into the flotation cell and $2cm^3$ of water added below the cell tip.
c. Impeller was start up for agitation with no air passing through the cell. Impeller speed was about 2000 rpm, having about 1cm clearance from the bottom of the cell.
d. The PH was checked and adjusted to 9.5.
e. 3 drops (0.0015kg/tonne) of collector was then added
f. Conditioning (without air) was done for about 10 minutes after collector was added
g. 3 drops (0.0015kg/tonne) of frother was added before the expiration of conditioning time.
h. Air was allowed to pass through the pulp at a reasonable rate.
i. The mineralized froth was skimmed until barren froth persists.
j. The froth and tailings were filtered, washed and dried then weighed.

VII. RESULTS

Results of various laboratory experiments and analysis carried out were presented and analyzed which include:
i. Sieve test result

      ii.   Result of the chemical analysis of the head sample
      iii.   Result of the froth flotation techniques

    i.   Sieve test result

Table 1: result of Sieve Analysis

Size range (μm)	Weight retained (g)	Weight retained (%)	Nominal (μm) Aperture size	Cumulative Weight retained (%)	Cumulative weight passing (%)
+250	26.48	13.24	250	13.24	86.76
-250+180	28.32	14.16	180	27.40	72.60
-180+125	30.02	15.01	125	42.41	57.59
-125+90	24.86	12.43	90	54.84	45.16
-90+63	35.60	17.80	63	72.64	27.36
-63	54.72	27.36	-	100	0.00

If 180μm =72.60
Xμm   = 80%
           = 180 x 80 / 72.60
           = 198.4 μm at 80%

Table 2: Result of the Chemical Analysis of Head Sample in Element form

Aperture size (μm)	Si	K	Ca	Ti	V	Mn	Fe	Ni	Cu	Zn	Ag	Au	Pb
250	5.67	1.12	0.14	0.18	0.02	1.00	20.10	0.02	0.13	1.10	1.9	0.01	9.30
180	5.90	1.35	1.12	-	0.04	2.10	18.11	0.03	0.15	1.14	2.5	0.03	10.55
125	5.73	1.70	0.80	0.10	0.01	1.11	19.10	0.01	0.12	1.14	2.6	0.11	11.8
90	6.21	2.41	0.31	0.15	0.04	1.12	17.12	0.08	0.19	1.20	2.1	0.10	14.9
63	4.92	1.93	0.62	0.11	0.09	1.51	13.30	0.10	0.04	1.11	3.8	0.08	17.3

Table 3: Result of the froth flotation method

Products	Weight (g)	Weight (%)	Assay Value (%)			
			Pb	**Fe**	**Zn**	**Si**
Feed	100	100	17.3	13.3	1.11	4.92
Concentrate	64.80	64.80	53.3	7.68	0.88	9.38
Tailings	33.20	33.20	23.7	10.0	0.59	21.1
Losses	2.00	2.00	-	-	-	-

Discussion of Results
Sieve size
   It was observed from the sieve test results presented (table 1) that 26.48g of the total weight (200g) was retained on the 250μm sieve size, 28.32g on 180μm, 30.02g on 125μm , 24.86g on 90μm and 35.60g on 63μm. From the table, the mesh of grind was calculated to be 198μm.
Chemical analysis
   The chemical analysis of the sample was conducted on all the five sieve sizes. The result in its elemental form indicated that 250μm contain 9.30%Pb, 1.10%,Zn and 5.67%Si etc, 180μm contains 10.55%Pb, 1.14%Zn, and 5.9%Si. 125μm contains 11.8%Pb, 1.14%Zn, and 5.73%Si. 90μm contains 14.9%Pb, 1.20%Zn,and 6.20%Si, while 63μm contain 17.3%Pb, 1.11%Zn, and 4.92%Si.
   The result has confirmed 63μm to be liberation size of Arufu lead ore.
Froth flotation
   The froth flotation results shows two products; tailings and the concentrate, having weight of 33.20g with assay value 23.7%Pb and weight of 64.80g with assay 53.3%Pb respectively.
   The result revealed that the ore has been upgraded from 17.3%Pb to 53.3%Pb.

VIII. CONCLUSION AND RECOMMENDATION

Conclusion
   From the foregoing, the research work established the mesh of grind to be 198μm for Arufu lead ore and liberation size to be 63μm sieve size having the highest recovery of lead in its elemental form. Also further work was done on the separation using 63μm sieve size (liberation size) by froth flotation method and the ore upgraded from 17.3% to 53.3%Pb assessed by using x-ray fluorescence (XRF) with 65 percentage by weight lead concentrate confirmed to be of economic grade having surpassed 60% wt Pb element in an ore (www.rosslynhillmining.com.au, 2013). It is hoped that this will enhance its development to meet both local and international demand for potential investment opportunities.

Recommendation

Further work need to be done especially to determine the reserve estimates of the deposit.

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