Froth Flotation Beneficiation A Sure Way to Value Addition to Arufu (Nigeria) Zinc Ore towards Smelting Grade Concentrate Production

Alabi Oladunni Oyelola, Bodeode Olanrewaju Rotimi, and Popoola Taiwo Paul

Abstract—Froth flotation of Arufu ore was carried out at varying particle sizes after characterization. Fifty (50) kilogram crude sample of the ore was sourced from Arufu zinc mine in Arufu town of Nassarawa state, Nigeria. The whole sample was crushed out of which five (5) kilogram was sampled out using random sampling method. One kilogram each of the resulting sample was then ground and sieved to three particle sizes viz; 63 µm, 90 µm and 125 µm. Chemical analysis of the representative sample of the sourced ore was carried out using Energy Dispersive X-Ray Fluorescence Spectrometer (ED – XRF). 250 grams of the 63 µm sample was charged into Froth flotation cell mixed with water at a ratio of 1:4 to form slurry. The Slurry formed was condition to a pH of 9, while other froth flotation reagents were added one after the other. This resulted in froth and depressed samples, which were dewatered after 24 hours and samples picked for compositional analysis.

The procedure was repeated for 90 µm and 125 µm. The characterization of head sample revealed that the ore contains predominantly 36.80%ZnO (26.29% Zn), 31.1 % SiO₂ alongside other trace mineral such as gangue in the ore. However, Froth flotation studies of the ore at varying particle size revealed that, appreciable amount of mineral of interest (ZnO) was lost to the tailing at sieve sizes 63 µm and 90 µm. This was attributed to over-grinding above the ore’s liberation size phenomenon which has been proven to have adverse effect on the mineral’s quality and overall separation efficiency. It was concluded that the froth flotation is best carried out at a particle size of 125 µm, pH of 9, using potassium ethyl xanthate (PEX) as frother to yield concentrate grade of 50.21 % ZnO (35.93 % Zn) at a recovery of 46.3%. This was re – cleaned to yield high grade of 82.36% ZnO (66.42% Zn). The re – cleaned concentrate produced falls within the standard requirement of 65 % Zn needed as a charge into the blast furnace for Zinc metal production.

Index Terms—Froth Flotation, Concentration, Smelting, Grade.

I. INTRODUCTION

Mineral deposits are frequently found in adequate concentration to enable the metals to be effectively recovered. These mineral deposits come to be due to action of some natural agencies which include magmatic, hydrothermal, sedimentary and geological events [1]. Several mineral resources are widely distributed all over Nigeria with over thirty four minerals commodities spread across the entire country, which include gold, coal, iron ore, tantalite ore, columbite ore, lead – zinc ore, barite, cassiterite, gemstones, limestone, gypsum, talc, feldspar and marble [2] [3].

A. Zinc Ore

Zinc ore is found in commercial quantity in various deposits such as Zamfara, Bauchi, Gombe, Nassarawa, Plateau, e. t. c. Ore is a natural occurrence of rock or sediment that contains sufficient minerals with economically important element typically metals that can be economically extracted from the deposit. No ore deposit consists entirely of a single mineral, there are always mixtures of valuable and non - valuable (gange) mineral collectively [4] [5]. However, for every mineral deposit there is a set of conditions, such as the level of concentrate and the size of the deposit that must be reached if the deposit is to be worked at a profit [6]. Hence, treatment of ores to get their metallic concentrate (beneficiation) into useful product (Concentrate) of smaller bulk and simultaneously to separate the worthless mineral (gange) into discarded waste (tailings) is required for materials manufacturing [7] [8].

B. Mineral Processing Operation

There are two fundamental operations in mineral beneficiate namely, the release or 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. Chemical and Mineralogical characterization of the ore comes ahead of and lead to determination of best beneficiation route for its constituent minerals or metals [9].

C. Froth Flotation

Froth flotation being the beneficiation method adopted in this research is a physicochemical beneficiation process which exploits the difference in surface properties of the concentrate and gangue. Such differences in mineral properties become apparent after conditioning with suitable reagent like depressants, activators, collectors etc. The clinging of the mineral to air bubbles is the most important mechanism and represents the majority of the particles which are recovered as concentrate [10] [11] and tailing as deposit or vice -verse.

D. Importance of Mineral Exploration and Exploitation

Federal Government of Nigeria is presently being confronted with huge financial burden partly caused by the reduction in revenues from crude oil. The reduction in global crude oil prices is not expected to reverse at least in the short run, thus the need to start thinking of
diversification in economic towards improving other sources of revenue. One key sector which offers great potential in achieving this is the solid mineral sector which is expected to contribute at least 10 % to the Gross domestic product (GDP) by 2025 but currently average only about 0.46 % [12] Nigeria is endowed with large number of zinc ore deposit, but gained only a little due to the sales of same mineral to the outside world in its crude after exploration and exploitation which is mostly done by the licensed and unlicensed artisanal miners. However, Nigeria stands to benefit more from processing its zinc mineral from zinc ore deposit and add more value to the mineral by extraction of metal from it before exporting [13] [14]. One must note that choice of process route should be that which has been proven to be scientifically and technically feasible with least possible time, energy and cost [15] [10].

E. Applications of Zinc
Zinc product finds its application in several major industries including construction (for galvanizing and steel manufacturing), transportation (wheel balance production), machinry manufacturing, electrical, chemical and other industries. Zinc oxide is well suited for use in rubber, paints, inks, dyes, oil additives, wood preservatives, fungicides and varnishes. Zinc oxide is used in these products because of its characteristics opacity to ultra – violet light and high refractory index, durability, and electrostatic properties [16]. As a result of the areas where zinc finds application and profit in which an individual or a nation stand to gain, there is need for the construction of zinc processing and smelting plant in the country. This is what propelled this research, thereby suggesting a suitable process route for concentrating Arufu zinc ore deposit, in order to obtain zinc mineral in smelting grade, a suitable beneficiation route needs to be arrived at to suit its attributes or peculiarity.

II. MATERIAL AND METHODS

A. Materials
Zinc ore used at the course of this research was sourced from Arufu town in the middle trough, Northern – Eastern, Nassarawa state, Nigeria. On a longitude 9° 10’ 0” - 9°20’0”E and Latitude 7°40’0” – 7°45’0”N. It is roughly about 200 – 300 m above sea level, with River Benue as the main river flowing through it, and dendritic type of drainage [17] Arufu is characterized by the tropical wet – dry climate and a rainfall which is generally moderate about 100 – 120 cm per annum, with annual temperature range from 30 - 35°C and relative humidity of between 30 – 60% [18]. The vegetation of Arufu is the savannah wood type, characterized by tall grass, shrub and few trees ranging in height of 3 - 6 meters [19] [20] worked in the upper, Middle and Lower Benue trough reported that the area consists of sediments of the cretaceous age (136±25 Ma) and surrounded by the Pan African Precambrian basement complex (600±150 Ma). He further observed that the region has three stratigraphical correlations; an upper or north – eastern region; middle Benue region or Lafia Muri area and the lower or south- western part of Makurdi. Arufu mines consist of zinc ore deposit in commercial deposit of over 2.6 million tons well spread under - ground at a depth of about 16 meters below the earth surface with quartz as over its burden. Other materials used are Collector (Potassium Ethyl Xanthate), Frother (Pine Oil), Activator (Copper Sulphate), pH regulator (Sodium hydroxide), and depressant (Zinc sulphate).

B. Methods - Sample Collection and Preparation
Crude zinc ore samples were collected from twenty (20) different pits on the Arufu mine site. These samples were homogenized after hammer crushed. Fifty Kilograms (50Kg) of the crude zinc ore was collected from the total homogenized ore using random sampling method followed by cut and quartering to represent the total lot on the mine site. Five (5kg) Kilograms was sampled out in the laboratory from the site lot; this was further prepared for the froth flotation process by comminution which consist of sequential crushing using Laboratory Denver jaw crusher (model D -12), and grinding using Denver ball millling machine model 69012A to a pulverized stage of 63 µm, 90 µm and 125 µm respectively ready for froth flotation. However, ahead of this representative sample of the ore has been collected for chemical analysis using Energy Dispersive X-ray Fluorescence Spectrometer (PANanalytical Minipal 7) at the National Metallurgical Development Centre, Jos Plateau state, Nigeria.

C. Froth Flotation of Arufu Zinc Ore
Froth flotation of Arufu zinc ore was carried out using three different particle sizes (63, 90 and 125 µm) at the Mineral processing Laboratory of the Federal University of Technology, Akure, Ondo State, Nigeria. 250 g of Arufu ore ground to 63 µm was charged into cell or tank of Denver froth flotation machine alongside 1000 cm² of water, mixed together to form slurry of pH 6.8 under the impeller at a clearance of 1 cm from the cell’s bottom. The mixture was agitated for 120 seconds after which the pulp pH was measured and adjusted to pH 9 using drops of Sodium hydroxide (NaOH) as the pH regulator. The constituent was agitated for another 120 seconds, after which 0.5 grams of zinc sulphate (ZnSO₄) was added to the pulp as depressant and agitated for another 120 seconds followed by addition of 0.5 gms of activator – copper sulphate (CuSO₄) . 0.5 gms of collector – Potassium Ethyl Xanthate (PEX) was added and agitated before 0.5 gms of frother – Pine Oil was added and agitated for another 120 seconds. Finally, the air tap was opened for aeration through the pulp and air bubbles introduced into the pulp. The froth formed was scooped out into a receiver for 300 seconds, followed by addition of water and more aeration until clear froth was observed. The froth and depressed were left to settle for 24 hours, decanted and filtered of water to form cake, which was oven dried at 105°C in an Endecott oven and then weighed and resulting samples were chemically analyzed using ED – XRF. This procedure was adopted and repeated for the 90 µm and 125 µm samples respectively.

III. RESULTS AND DISCUSSION
The result of the chemical analysis of the crude Arufu ore showing its compositions are presented in Table 1.
The result of the chemical characterization of the crude zinc ore shows that the run of mine contains 31.1% SiO$_2$, 24.9% SO$_2$, 2.58% Fe$_2$O$_3$, 36.8% ZnO, 0.81% PbO as major constituent with some others as minor constituents. However, Zinc oxide has the highest grade in the ore with equivalent Zinc of 29.56% (36.8% ZnO) with other impurities like silica in quartz form and iron which when processed out will improve the constituent of zinc in the ore, hence the need for beneficiation of Arufu zinc ore. Based on the composition of zinc oxide in the ore, it is evidenced that the ore met the typical standard feed grade of 1 – 10% Zn required to mine zinc from its deposit [21]. From this result it is evident that the zinc ore is notable a sulphide mineral based on the identified Sulphur oxide having 24.9% in the ore. It was therefore established that the Arufu zinc ore is a low grade sulphide mineral assaying 36.8% ZnO (29.56% Zn) and has been proven to be economically viable for mining.

<table>
<thead>
<tr>
<th>Sieve Sizes (µm)</th>
<th>Compd. Wt. of Feed (g)</th>
<th>Wt. of Conc. (% ZnO)</th>
<th>Grade of Concentrate (%)</th>
<th>Wt. of Tailing (g)</th>
<th>Grade of Tailing (%)</th>
</tr>
</thead>
<tbody>
<tr>
<td>63</td>
<td>250.0</td>
<td>109.31</td>
<td>49.65</td>
<td>140.69</td>
<td>40.28</td>
</tr>
<tr>
<td>90</td>
<td>250.0</td>
<td>62.88</td>
<td>46.50</td>
<td>187.12</td>
<td>42.83</td>
</tr>
<tr>
<td>125</td>
<td>250.0</td>
<td>84.83</td>
<td>50.21</td>
<td>165.17</td>
<td>4.49</td>
</tr>
</tbody>
</table>

From Table II: it was observed that at 63 µm has the highest weight of concentrate of 109.31 g at a grade of 49.65% ZnO, tailing 140.69 g at 40.28. When the sieve size was increased to 90 µm there was a drop in weight of 62.88 at 46.50% ZnO, Tailing 187.12 g at 42.83% ZnO and a further increase in the sieve size to 125 µm lead to increase in weight of concentrate of 84.83 g at a grade of 50.21% ZnO with its tailing at 165.17 g at 4.49% ZnO. It was observed that the maximum yield was observed at concentrate at optimized case of 63 µm, with fairly high grade. It can be deduced that appreciable amount of the mineral of interest (Zn) was lost to tailing at sizes 63 µm and 90 µm due to the high zinc oxide content of their tailings. This phenomenon is attributed to over – grinding which has been proven to pose adverse effect on liberation and overall enhancement of the ore [22]. In other word, over – grinding directly affects the quality and efficiency of mineral separation [21][22]. However, effective liberation of the mineral of interest (ZnO) was more pronounced at sieve size of 125 µm due to the markedly difference observed between the essay of the concentrate and tailing obtained at this size. Therefore, processing at this particle size is advised to annul any effect of over grinding. It is worthy to note that 125 µm has a fairly increased weight, with the highest ZnO grade, but did not meet up to the required 65% Zn (80.5% ZnO) grade needed as a charge for pyrometallurgical extraction plant of Zinc. Hence the need to re – clean the product of this sieve size – 125 µm that resulted in best grade.

Metallurgical accounting parameter is one of the crucial feature of a successful metallurgical operation which determines the distribution of products of a concentrate and the value inherent in them, also to make decisions about the operation from the value of recovery and grade obtained from the accounting procedure. Table III present the metallurgical accounts of the process by considering value of Concentrate assay, Recovery, Concentration ratio and Enrichment ratio at each sieve sizes. It was deduced that at 63 µm, the concentrate obtained assayed 49.65 at recovery of 58.99%, with calculated Concentration ratio of 2.29 and enrichment ratio of 1.35. at 90 µm, the concentrate obtained assayed 46.50 at recovery of 31.78%, with calculated Concentration ratio of 3.98 and enrichment ratio of 1.26 and at 125 µm, the concentrate obtained assayed 50.21 at recovery of 46.30%, with calculated Concentration ratio of 2.95 and enrichment ratio of 1.36. It can be seen that 125 µm gave the highest concentrate assay while processing at 63 µm gave the highest recovery. However, according to Wills (2006) a low grade concentrate may achieve greater recovery of the values, but incur greater smelting and transportation cost due to the included gangue mineral. Processing of Arufu zinc ore at 125 µm with pH of 9 using potassium ethyl xanthate as collector was established as the best condition at this stage, yielding concentrate grade of 50.21 % ZnO (36.93% Zn) at recovery of 46.30%, concentration ratio of 2.95 and enrichment ratio of 1.36 from a feed of 36.80% ZnO (29.53%Zn).

<table>
<thead>
<tr>
<th>Sieve size (µm)</th>
<th>Wt. of Feed (g)</th>
<th>Wt. of Conc. (% ZnO)</th>
<th>Grade of Conc. (%)</th>
<th>Wt. of Tailing (g)</th>
<th>Grade of Tailing (%)</th>
</tr>
</thead>
<tbody>
<tr>
<td>125 (First Run)</td>
<td>250.0</td>
<td>36.80</td>
<td>84.83</td>
<td>50.21</td>
<td>165.17</td>
</tr>
<tr>
<td>125 (Re– Cleaning)</td>
<td>84.83</td>
<td>50.21</td>
<td>21.36</td>
<td>82.36</td>
<td>64.47</td>
</tr>
</tbody>
</table>

Re - cleaning is aimed at obtaining better result compared to the first. Table IV revealed the level at which the grade of zinc ore increased from 50.21% ZnO to 82.36% ZnO (66.41% Zn) when subjected to re – cleaning which is above the standard grade of 80.5%ZnO (65%Zn) needed for pyrometallurgical extraction purpose. It clearly revealed that the grade of the ZnO improved from the initial best to much better when subjected to re – cleaning process.
IV. CONCLUSION AND RECOMMENDATION

A. Conclusion

The research has been carried out to prove the importance of Arufu zinc ore deposit on the development of Arufu community if explored and exploited. The research has been able to establish the following:

i. chemical characterization of the ore has established that Arufu Zinc ore assays 36.80% ZnO (26.56% Zn) which proves that the mine is viable to explore and exploited,

ii. best processing route is at a sieve size 125 µm to increase the grade of ZnO to 50.21% ZnO at recovery of 46.3%, Concentration ratio of 2.95 and Enrichment ratio of 1.36, and;

iii. Re - cleaning of the best processing route to yield an added value of 82.36% ZnO (66.41% Zn) above the 65% Zn needed as a charge.

However, it is worthy to note that loss of material of interest to tailing at sieve sizes 63 and 90 µm was due to over grinding, a phenomenon which adversely affects the mineral quality and overall separation efficiency. It is therefore worth that froth flotation of Arufu zinc be carried out at other separation methods such as magnetic, gravity separation and froth flotation should be carried out at varying parameters such as pH, types of collectors, activator, depressor, and so on.

B. Recommendation

Processing of Arufu Zinc ore should be carried out using other processing methods such as magnetic, gravity and leaching methods in order to compare and contrast the effect of the processes on the ore.

ACKNOWLEDGMENT

The Authors wish to show our profound gratitude to the management of the Federal University of Technology, Akure and National Metallurgical Development Centre, Zaria Road, Jos Plateau State, Nigeria; for allowing the utilization of their facilities - laboratories and Library. These in has led to the success of this research.

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