DETERMINING THE METAL CONTENTS OF LEACH LIQOUR FROM IHITUTU LEAD AND ZINC ORE, NIGERIA

Ajaka E. O., and Abiodun O. E.

Department of Mining Engineering, Federal University of Technology, Akure, Nigeria

ABSTRACT
This study characterized and recovered metals from Ihitutu lead-zinc sample Galena grains are interlocked with sphalerite and gangue minerals. Autogenous and media assisted grinding tests were studied in addition of difference and new mass charged modes. Gravity method for the production of a concentrate was also carried out using spiral separator with optimum recovery at +600 μm and highest loss at +125-53 μm. Also, the rate of leaching increased with time and an average of 88 g and 125 g was leached into the solution after three days for microwave pretreated and as-mined ore respectively. The final leachate contains 150 mg Pb/kg, 34 mg Zn/kg in aqua regia as-mined, 185 mg Pb/kg, 54.25 mg Zn/kg in aqua regia microwave pretreated, 137.20 mg Pb/kg, 21.50 mg Zn/kg in H2SO4 as-mined and 160.40 mg Pb/kg, 37.67 mg Zn/kg in H2SO4 microwave pretreated. The result from leaching revealed that aqua-regia has stronger ability for better yield than sulphuric acid in both microwave pretreated sample and as-mined sample respectively. Weight loss during roasting was 3.81%.

Correspondence:
 eoajaka@futa.edu.ng.

1.0 INTRODUCTION
The mining industries are now viewed as key drivers of economic growth and development of any nation (Bradshaw, 2005). Mining produces various minerals and materials for technological development. Thus mining, mineralogy and extractive metallurgy are key to the provision of the various metallic materials which drive nation and economic development (Bridge, 2008). Sphalerite (ZnS) and galena (PbS) are the major minerals of zinc and lead found in abundantly in Nigeria. They are also commonly associated with other minerals (metal). Sphalerite contains 67% Zn whilst galena contains 86.6% Pb. They often also include traces of sulphides of cadmium, gallium, germanium and indium in solution. Processing this ore to obtain the necessary values requires beneficiation, smelting and refining to prepare marketable lead or zinc product of high purity. Lead has found wide engineering applications in the electrical, mechanical, metallurgical, chemical, pharmaceutical, oil, medical industry. It is also used in the military, nuclear industry (Encyclopedia Britannica, 2010). Similarly, zinc products are used in several industries, including construction, transportation, machinery manufacturing, electrical, chemical and other industries (Egunlade and Jimoh, 2010). Recent works have revealed that heating can improve the liberation, grindability, and magnetic separation of valuable minerals from ores and has the potential to provide a new method for treating ores successfully (Omran et al., 2015; Song et al., 2013; Zhao et al., 2014).

Production of metals (including lead and zinc) from
their ores is usually done a number of metallurgical processing routes which beneficiation by processes such as flotation, gravity separation, hydrometallurgical processes (e.g. leaching) among other; this is followed by either electrolytic processing and smelting. Hydrometallurgical processing however, suffers a major drawback of inability to effectively dissolve sulphide ores due to their low solubility in many reagents and the complexities in their mineralogical associations (Olubambi et al., 2009): This drawback has led to the use of large volumes of concentrated solutions for dissolving these ores, thereby resulting in a high consumption rate of reagents that often cause environmental hazards. Leaching is the dissolution of a mineral in a solvent, while leaving the gangue (rock or mineral matter of no value) behind as undissolved solids. Oxidation-reduction reactions may also be involved (Geankoplis, 2007). In metal processing industries leaching has been extensively used to remove metals as soluble salts and is also referred to as a hydrometallurgical process (Petrucci, 2003). Due to the nature of the leaching process, most of the metals within the ore are dissolved during leaching, resulting in high acid consumption (Büyükakinci and Topkaya, 2009). The leaching is the first and the most important step of producing metal economically by using hydrometallurgical process (Abdel-Aal, 2000). The lead-zinc ore from Ihietutu in Ebonyi state which is used for this project contains other associated metals apart from the primary lead and zinc. To date, the ore is still being sold and exported as mined.

The aim of this research is to determining the metal contents of leach liqour with the specific objective to:

a) determine the chemical and mineralogical characteristics of the Ihietutu lead-zinc ore;
b) concentrate the ore; and
c) recover metals through leaching.

![Geologic Map of Nigeria Showing the Location of Study Area](image)

**Figure 1.1**

### 3.0 MATERIALS AND METHODS

#### 3.1 MATERIALS AND EQUIPMENT

The materials and equipment that were used for the execution of this study are; ore samples collected from a deposit at Ihietutu, operated by “Skyray Instrument” X-ray fluorescence spectrometer EDX3600B, laboratory Jaw Crusher GTEK PE - 100×100, Denver ball mill with steel balls, Riffle box, Denver sieve shaking machine, digital weighting balance - Labtech BL-20001, Set of sieves, Reagents (sulfuric acid, nitric and hydrochloric acid), Platinum microwave oven 7092970 (P90D25EL-G1), a hot plate with magnetic stirrer, furnace, Digital pH meter and Moulds for casting smelt. The materials and equipment were prepared and used according to the procedures for each experiment undertaken.

#### 3.1.1 Sample Collection

About 50 kg of the ore samples were collected from the deposit site at Ihietutu, using the grab and trench sampling techniques which involved collection at random manner. Samples were collected at GPS reading between latitudes 5°53’40"N and 5°56’46.7"N and longitudes 7°24’65"E and 7°29’02.3"E locations marked by Garmin 76S handheld GPS.

#### 3.2 METHODS

The composition of the ore samples was determined using X-ray fluorescence spectrometer (XRF), atomic absorption spectrometer and
petrographic analysis. Comminution and liberation analyses were carried out through crushing and grinding tests and these were followed by series of laboratory scale recovery tests using spiral separation techniques for concentration. Leaching and pyrometallurgical processes were used for recovery of the metals. Figure 2 is the general flow sheet for recovery process.

3.2.1 COMPOSITION ANALYSIS

Determination of some physical properties of the Ore

100 g of the dried ore was placed into 500 cm$^3$ of water in a graduated measuring cylinder. It was ensured that the ore particles were completely wetted and entrained air was allowed to escape. The volume of water which represents the volume of the ore was observed and recorded. The procedure was repeated two more times and the specific gravity of the ore was determined using Equation 1.

\[ S = \frac{w}{v} \]

Where, \( S \) is specific gravity, \( w \) is weight (g), and \( v \) is volume (cm$^3$) of the ore.

The as-mined colour of the sample was carefully observed and streak test was also carried out.

Chemical Compositional Analysis

The chemical analysis of the ore was carried out using X-ray fluorescence spectrometer - EDX3600B located at Engineering Materials Development Institute laboratory (EMDI), Federal Ministry of Science and Technology, Akure, Ondo State. The results of compositional analysis are presented in Table 1 and Figure 3.

Micrographic Analysis of Ore Samples

For micrographic analysis of the test samples, polished sections were prepared and examined under an optical (ore) microscope in cross nicol. This was used to characterize the ore as shown in Plate 2 and 3.

3.2.2 COMMINUTION AND LIBERATION TESTS

Crushing Test

The as-mined ore was sorted to into two portions containing 50mm and 80mm size ranges, and each size range was also divided into two portions for crushing in a laboratory jaw crusher GTEK PE - 100×100 at 10mm and 15mm set values. The 50mm ore samples were crushed at 10mm set after which each product was discharged and labeled appropriately. This was repeated for 15mm set size. The particle size analyses of the crushed products were done on both set sizes (10mm and 15mm) the laboratory sieve shaker for fifteen minutes. The sample retained on each sieve was weighed with a digital weighting machine Labtech BL-2000I and the cumulative per cent mass retained and passing each sieve size was calculated. The cumulative per cent weight was plotted against particle size to determine the 80% passing size. The procedure was also carried out for the 80mm feed sample. The aim of the crushing test was to prepare the ores for other downstream processes that were carried out in the course of this research.

Particle Size Analysis

Sieve analysis was carried out to determine the particle size distribution of the crushed ore in each sieve size fraction. The 10mm crushed ore was used for the analysis. The samples were thoroughly mixed using coning and quartering technique and riffle box splitting method for homogenization of the samples. 1kg of the product from the riffle box was sieved on a denver sieve shaking machine for 15 minutes with 10 selected sieves in two runs. Sieve aperture sizes ranging from 4750μm to 53μm were used. Each sieve size fraction obtained was weighed and recorded. This was repeated for accuracy.

Grindability Test

Each set of crushed samples was thoroughly mixed using coning and quartering technique and passed through a Jones riffling sampler for
homogenization after which it was used for the 
tests. Full autogenous and media-assisted (ball 
milling) grindability techniques were employed to 
compare the ease of grinding of the crushed sample. 
Both tests were carried out at 180 microns (180 \( \mu \)) 
using constant mass charge by addition of 
difference and new mass charge procedures. The 
media-assisted tests were carried out with 45% 
media (ball) charge. A constant mass of 200 g was 
used in all grinding tests.

Each measured batch of 200g material was ground 
for 200, 400, and 600 revolutions, in a Denver ball 
mill and the content of the mill was discharge and 
weighed after each run. The sieve size analysis of 
the product was carried out and the weight of 
material retained on each sieve was weighed with a 
digital weighing machine Labtech BL-20001 and 
the cumulative per cent mass passing each sieve 
size was calculated. The cumulative per cent mass 
passing each sieve size was plotted against particle 
size to determine the 80% passing size.

In the constant mass charge by addition of 
difference, the -180\( \mu \) fraction of the product was 
remove and new feed equivalent to its weight added 
for another run. But in the new mass charge, 200g 
of fresh feed was measured after each test and used 
in another run. Grindability was calculated using 
equation 2. Each result was reduced to Grindability 
in kilograms per tons per revolution (kg/ton/rev) 
and Bond's model (Equation 3) used to determine 
the grindability index value. Three runs were made 
for each batch of test, and the average value recorded.

\[
G_r = \frac{\text{Weight (W}_{-180})}{\text{Number of revolutions}} 
\]

Bond work index (BWI - \( w_t \)) was determined using 
the expression in Equation 3

\[
W_t = \frac{48.95}{D^{0.23}G^{0.82}10^{\frac{1}{(\sqrt{G_{80}})}}} 
\]

Where:

- \( F_{80} \) is Feed size (microns) through which 80% of 
  the feed passed
- \( P_{80} \) is Product size (microns) through which 80% of 
  the feed passed
- \( D \) is Aperture (microns) of the classifying screen
- \( G \) is Net weight (grams) of undersize (-180microns) 
  product per unit revolution of the mill,
- \( w_t \) is Bond work index in Kwh/t

After grinding, thorough mixing of the product and 
sieve analysis was carried out for classification 
according to their sieve sizes for further 
concentration processes.

**Liberation Size Analysis**
The liberation size of the minerals in the ore was 
determined from the results of photomicrographic 
analysis, and particle size analysis. The aim of the 
liberation test was to determine the particle size at 
which the valuable minerals are completely free 
from the gangue.

**3.2.3 CONCENTRATION TESTS**

**Determination of Concentration Criterion**
The specific gravities of the main minerals in the 
ore galena, sphalerite and quartz were used to 
determine the Concentration Criterion (CC) for 
determining the susceptibility of the ore to gravity 
separation process (La-Plante and Spiller, 2002). 
The concentration criterion is expressed in 
Equation (3.4)

\[
CC = \frac{(d_h - d_f)}{(d_l - d_f)} 
\]

Where; \( d_h \) is the specific gravity of the heavy 
mineral, \( d_l \) is the specific gravity of the light 
mineral, \( d_f \) is the specific gravity of the fluid,

**Spiral Separation**
Experimental concentration by spiral separation 
was conducted based on the difference in specific 
gravity of lead-zinc and gangue minerals using a 6-
bowl spiral separator. Based on liberation analysis, 
six particle size ranges of 125-53\( \mu \)m, 180-300\( \mu \)m, 
600\( \mu \)m, 850\( \mu \)m, 1180\( \mu \)m and 2360\( \mu \)m were used
separately in the experimental runs. Water was the fluid medium used; it was made to agitate continuously inside an agitator before adding 500g of each sieved sample into it to form a slurry of 40% solid content. The products (concentrate and tailing) were collected by discharging into separate bows. The tailings and the concentrate were dewatered and weighted after each operation, label and recorded. The separation process was repeated in two more stages with both concentrate and tailings, the final concentrates used in the metal recovery process.

![Diagram of Gravity Separation Process]

**Figure 1: Flowsheet for Gravity Separation**

### 3.2.4 METAL RECOVERY TEST

Two routes of leaching – electrowinning and roasting and smelting were used for the metal recovery process.

#### Microwave Pretreatment

300g of the representative sample was subjected to radiation for 60 seconds using a ceramics crucible. The crucible was placed in the central position of the microwave oven in order to minimize the effect of field pattern variation in the oven. Platinum microwave oven 7092970 (P90D25EL-G1) with radiation output of 2450 MHz. This was carried out to ascertain the influence of microwave pretreatment on leaching process and the final weight was measured.

#### Leaching

A batch of samples for the leaching experiment was pretreated by microwave irradiation and another set of samples were leached as-mined. An agitated leaching technique with sulphuric acid and 1:1 aqua regia leixvant were employed. A magnetic stirrer equipped with hot plate was used. 250g of sample was used in 500ml lixivant for each run and two sets of experiments were set up simultaneously for each sample type (as-mined and microwave pretreated). Leaching was carried out for four days and 20ml of the sample volume of leachate drawn after 1 day, 2 days and 3 days for analysis. Figure 2 shows the set-up of the leaching process.

**Electrowinning Process**

The final pregnant solution (leachate) from leaching was diluted. A simple electrolytic cell for the experiment was designed and constructed with the following features; anode (graphite rod), Cathode (zinc rod) and a cell of 300 mm x 100 mm x 100 mm electrolytic bath made of Polyvinyl- chloride material containing 1000 cm$^3$ of leached liquor. 200ml of the final pregnant solution was measured and used as electrolyte in the subsequent electrowinning processes. The electrowinning experiment was set up at room temperature (2°C). The initial weight of Zinc cathode was measured using the digital balance before the terminals of the DC power source were connected. The electro-winning cell bath was filled with the pregnant solution and organic additives. Gum-Arabic was added to improve on the throwing power of the electrolyte and the adhesion of zinc and lead metals to cathode. The Pb and Zn concentration in leached liquor (pregnant solution), spent (barren) solution were analyzed using atom absorption spectrometer.

**Figure 2: Flowsheet for Metal Recovery**
Plate 2: Iron Crucible Used for Smelting

3.0 RESULTS AND DISCUSSION
The result of all tests carried out in this research as described in chapter three and their implications on processing and metal recovery from the ore are presented in this section.

3.1 Chemical Composition
The ore was chemically characterised using EDX3600B X-ray fluorescence spectrometer and result presented in Table 1.

Table 1: Chemical analysis of the ore using X-ray fluorescence spectrometer

<table>
<thead>
<tr>
<th>Metals</th>
<th>Content (%)</th>
<th>Metals</th>
<th>Content (%)</th>
</tr>
</thead>
<tbody>
<tr>
<td>Al</td>
<td>0.8177</td>
<td>Fe</td>
<td>2.9927</td>
</tr>
<tr>
<td>Si</td>
<td>1.1768</td>
<td>Ni</td>
<td>0.0331</td>
</tr>
<tr>
<td>P</td>
<td>0.2035</td>
<td>Cu</td>
<td>0.0668</td>
</tr>
<tr>
<td>S</td>
<td>19.3696</td>
<td>Zn</td>
<td>8.5902</td>
</tr>
<tr>
<td>K</td>
<td>0.0026</td>
<td>As</td>
<td>2.2044</td>
</tr>
<tr>
<td>Ca</td>
<td>0.0260</td>
<td>Pb</td>
<td>54.6582</td>
</tr>
<tr>
<td>V</td>
<td>0.0049</td>
<td>W</td>
<td>6.7994</td>
</tr>
<tr>
<td>Cr</td>
<td>0.0020</td>
<td>Au</td>
<td>2.5523</td>
</tr>
<tr>
<td>Mn</td>
<td>0.1225</td>
<td>Ag</td>
<td>0.0007</td>
</tr>
<tr>
<td>Co</td>
<td>0.0024</td>
<td>Sn</td>
<td>0.2198</td>
</tr>
<tr>
<td>Mo</td>
<td>0.0634</td>
<td>Sb</td>
<td>0.0169</td>
</tr>
<tr>
<td>Cd</td>
<td>0.0017</td>
<td></td>
<td></td>
</tr>
</tbody>
</table>

3.2 Mineralogical Analysis
The photomicrographs from polished sections of the ore are presented in Plates 2 and Plate 3.

Figure 3: XRF Intensity Curve of Ihietutu Lead-Zinc Ore

Plate 2: Reflected Light Photomicrograph and Summary Ore Description of Polished Section (Objective Lens Magnification 10x) Sample A

Plate 3: Reflected Light Photomicrograph and Summary Ore Description of Polished Section (Objective Lens Magnification 10x) Sample B

The microphotographs are produced at 10x magnification and saved as produced without image adjustment. As seen in the microphotographs, there are dominant phases of dark influences which definitely represent in part, the presence of opaque minerals. The dominance of this region somehow contains the relative abundance of lead mineral over other minerals in the ore. The lighter grains or phases may represent...
light minerals such as quartz. Other grains showing different reflections are also observed which may suggest inclusion of other metals. Some of these inclusions are observed to be more dominant in the dark phases which may also be an indication that the secondary metals are mainly dissolved in the lead phase. The massive nature of the phase (especially lead phase) also implies that the ore is of high grade with respect to this phase and suggests that liberation from the quartz (gangue) will take place at some coarse particles size.

3.2 Grinding

Table 3 and Table 4 show the result of autogenous and media assisted grindability test of the lead-zinc ore described in section 3.2.2.

<table>
<thead>
<tr>
<th>No of Revolution (Nr)</th>
<th>(g)</th>
<th>(g/rev)</th>
<th>(kg/t/rev)</th>
</tr>
</thead>
<tbody>
<tr>
<td></td>
<td>New Charge (NC)</td>
<td>Addition of Difference (AD)</td>
<td>New Charge (NC)</td>
</tr>
<tr>
<td>200</td>
<td>9.97</td>
<td>10.07</td>
<td>0.04985</td>
</tr>
<tr>
<td>400</td>
<td>21.21</td>
<td>33.67</td>
<td>0.05303</td>
</tr>
<tr>
<td>600</td>
<td>43.27</td>
<td>55.03</td>
<td>0.07212</td>
</tr>
<tr>
<td>Average</td>
<td>24.82</td>
<td>32.92</td>
<td>0.05833</td>
</tr>
</tbody>
</table>

Table 4: Media Assisted Grinding Test

<table>
<thead>
<tr>
<th>No of Revolution (Nr)</th>
<th>(g)</th>
<th>(g/rev)</th>
<th>(kg/t/rev)</th>
</tr>
</thead>
<tbody>
<tr>
<td></td>
<td>New Charge (NC)</td>
<td>Addition of Difference (AD)</td>
<td>New Charge (NC)</td>
</tr>
<tr>
<td>200</td>
<td>10.15</td>
<td>10.97</td>
<td>0.05075</td>
</tr>
<tr>
<td>400</td>
<td>33.60</td>
<td>39.79</td>
<td>0.08400</td>
</tr>
<tr>
<td>600</td>
<td>59.03</td>
<td>64.57</td>
<td>0.09838</td>
</tr>
<tr>
<td>Average</td>
<td>34.26</td>
<td>38.44</td>
<td>0.07771</td>
</tr>
</tbody>
</table>

Figure 4: Grindability Test for Autogenous Grinding with Number of Revolution

\[
G = 3E + 0.5N_t^2 + 0.15N_t + 15.77
\]

\[
G = 0.0001N_t^2 - 0.025N_t + 9.55
\]

\[R^2 = 1\]
Table 3 and Table 4 present the grinding test results as measured for easy application in plant comminution analysis. The polynomial trend-lines of the plots of grindability with number of revolution which gave the best fits of the results of experimental runs are presented in Figures 4 and Figure 5. Addition of difference mode gave the best result more than new mass charged in the two test modes (addition of difference and new mass charge). This implies that addition of difference mode is applicable in continuous grinding operation where particles get weaker internally and at the peripheral after each revolution are already weakened by impact are easily removed by any small impact, even small interaction between particles while the new mass charge is intended for use in batch grinding. Although the tests were carried out at 180 microns, the results can be applied to grinding to finer sizes, especially in regrind mills for recovery by leaching.

As expressed by Equation 1, the Grindability of a material varies with the number of revolution; and as grinding continues, grindability approaches a constant value characteristic of the material. The equation shows grindability value as dependent on the number of mill revolution which in turn depends on the mill duration. Grindability values increased sharply with increasing number of revolution. This trend is the same for grinding test types and modes. It is also observed from the result that grindability values are higher for addition of difference mode than in new charge mode per revolution for both grinding tests. Although grindability value are generally higher in media assisted runs than in autogenous grinding test, the reason for maximum values between autogenous and media assisted grinding in the two test modes may be due to a number of factors which were likely to involve feed properties in addition to the mechanism of breakage.

3.3 Gravity Concentration

The calculation of Concentration Criterion of Ihietutu lead-zinc ore is shown below using Equation 5 for determining the susceptibility of the ore to gravity separation process.

\[
CC_{\text{of Ishiagu ore}} = \frac{(D_g - D_w)}{(D_s - D_w)}
\]

Thus, for the Ishiagu lead-zinc ore,
\(D_g\) = the specific gravity of galena (PbS) = 7.6,
\(D_s\) = the specific gravity of sphalerite (ZnS) = 4.0,
\(D_q\) = the specific gravity of quartz (SiO2) = 2.65,
\(D_w\) = the specific gravity of tungsten disulfide (WS2) = 19.25
\(D_{au}\) = the specific gravity of gold (Au) = 19.32; and,
\(D_{wa}\) = the specific gravity of water = 1.0.
Table 5: Concentration Criterion Table

<table>
<thead>
<tr>
<th></th>
<th>PbS</th>
<th>ZnS</th>
<th>WS₂</th>
<th>Au</th>
<th>SiO₂</th>
</tr>
</thead>
<tbody>
<tr>
<td>PbS</td>
<td>x</td>
<td>2.2</td>
<td>2.77</td>
<td>2.78</td>
<td>4.00</td>
</tr>
<tr>
<td>ZnS</td>
<td>2.2</td>
<td>x</td>
<td>6.08</td>
<td>6.11</td>
<td>1.82</td>
</tr>
<tr>
<td>WS₂</td>
<td>2.77</td>
<td>6.08</td>
<td>x</td>
<td>1.00</td>
<td>11.06</td>
</tr>
<tr>
<td>Au</td>
<td>2.78</td>
<td>6.11</td>
<td>1.00</td>
<td>x</td>
<td>11.10</td>
</tr>
<tr>
<td>SiO₂</td>
<td>4.00</td>
<td>1.82</td>
<td>11.06</td>
<td>11.10</td>
<td>x</td>
</tr>
</tbody>
</table>

The gravity concentration criteria shown in Table 5 show that quartz can be separated efficiently from galena, and from gold and tungsten mineral if these are not in solid solution in the galena or lead phase. The table also indicates that both galena (lead) and sphalerite (Zinc) (CC = 2.2) may be separated with a little difficulty while it will be very difficult to separate galena and quartz (CC = 1.82). If the tungsten mineral and gold are in solid solution in the lead phase, then separating galena and sphalerite will imply that both gold and tungsten will go with the lead phase. Even if they are not in solution in the lead phase but exist as free and liberated components, both will still go with the galena phase in any gravity separation process. Separation of galena and sphalerite by gravity method will therefore require very efficient control of the process otherwise, separation will be very poor. If this method must be used, a multistage process will be effective.

3.4 Beneficiation Studies using Spiral Separation

Table 6 and Table 7 show the result of Beneficiation Studies of Ihietutu lead-zinc ore using spiral separation.

Table 6: Beneficiation Studies of Ihietutu Lead-Zinc Ore using Spiral Separation.

<table>
<thead>
<tr>
<th>Sieve Size (μm)</th>
<th>First Run</th>
<th>Second Run</th>
<th>Average Concentrate</th>
<th>Average Tailings</th>
</tr>
</thead>
<tbody>
<tr>
<td></td>
<td>Feed</td>
<td>Conc. C₁</td>
<td>Tailings T₁</td>
<td>Feed</td>
</tr>
<tr>
<td>+ 2360</td>
<td>500</td>
<td>212.6</td>
<td>273.6</td>
<td>486.2</td>
</tr>
<tr>
<td>+ 1180</td>
<td>500</td>
<td>257.7</td>
<td>231.5</td>
<td>489.2</td>
</tr>
<tr>
<td>+ 850</td>
<td>500</td>
<td>350.1</td>
<td>145.6</td>
<td>495.7</td>
</tr>
<tr>
<td>+ 600</td>
<td>500</td>
<td>381.8</td>
<td>104.7</td>
<td>486.5</td>
</tr>
<tr>
<td>+ 300-180</td>
<td>500</td>
<td>311.3</td>
<td>185.6</td>
<td>496.9</td>
</tr>
<tr>
<td>+ 125-53</td>
<td>500</td>
<td>196.5</td>
<td>221.9</td>
<td>418.4</td>
</tr>
</tbody>
</table>

Table 7: Beneficiation Studies of Ihietutu Lead-Zinc Ore using Spiral Separation for Tailings Preconcentration.

<table>
<thead>
<tr>
<th>Sieve Size (μm)</th>
<th>First Run</th>
<th>Second Run</th>
<th>Average Concentrate</th>
<th>Average Tailings</th>
</tr>
</thead>
<tbody>
<tr>
<td></td>
<td>Feed</td>
<td>Concentrate C₄</td>
<td>Tailings T₃</td>
<td>Feed</td>
</tr>
<tr>
<td>+ 2360</td>
<td>273.6</td>
<td>131.45</td>
<td>139.15</td>
<td>202.2</td>
</tr>
<tr>
<td>+ 1180</td>
<td>231.5</td>
<td>137.98</td>
<td>86.52</td>
<td>252.5</td>
</tr>
<tr>
<td>+ 850</td>
<td>145.6</td>
<td>103.11</td>
<td>35.6</td>
<td>321.6</td>
</tr>
<tr>
<td>+ 600</td>
<td>104.7</td>
<td>76.82</td>
<td>23.88</td>
<td>373.6</td>
</tr>
<tr>
<td>+ 300-180</td>
<td>185.6</td>
<td>131.32</td>
<td>36.28</td>
<td>324.4</td>
</tr>
<tr>
<td>+ 125-53</td>
<td>221.9</td>
<td>96.82</td>
<td>86.08</td>
<td>189.3</td>
</tr>
</tbody>
</table>

The result of beneficiation studies of the ore using spiral separation and the polynomial trend-line shown in Figure 7 and Figure 8. Based on the result of determination of concentration criteria, it is obvious that lead and zinc may be separated from each other with a little difficulty. Thus, separation to concentrate will include all heavy components while silica and other lighter components reported to tailings. The highest recovery was observed at +600 μm for both runs of the concentrate which gives more recovery of the primary metal. Moreover, more weight loss was observed at +125 – 53 μm, this implies that gravity separation is not effective for the recovery of the ore below -180 μm.
Table 8: Beneficiation Studies of Ihietutu Lead-Zinc Ore using Spiral Separation for Tailings Preconcentration (Percentage Weight of Ore to Concentrate /Tailing).

<table>
<thead>
<tr>
<th>Sieve Size (µm)</th>
<th>1st Run</th>
<th>2nd Run</th>
<th>Average</th>
<th>Average</th>
</tr>
</thead>
<tbody>
<tr>
<td></td>
<td>Concentrate (%)</td>
<td>Tailing (%)</td>
<td>Concentrate (%)</td>
<td>Tailing (%)</td>
</tr>
<tr>
<td>+ 2360</td>
<td>48.41</td>
<td>51.59</td>
<td>41.22</td>
<td>58.78</td>
</tr>
<tr>
<td>+ 1180</td>
<td>60.9</td>
<td>39.1</td>
<td>58.35</td>
<td>41.65</td>
</tr>
<tr>
<td>+ 850</td>
<td>73.49</td>
<td>26.51</td>
<td>72.88</td>
<td>27.12</td>
</tr>
<tr>
<td>+ 600</td>
<td>77.19</td>
<td>22.81</td>
<td>71.24</td>
<td>28.76</td>
</tr>
<tr>
<td>+ 300-180</td>
<td>73.45</td>
<td>26.55</td>
<td>78.07</td>
<td>21.93</td>
</tr>
<tr>
<td>+ 125-53</td>
<td>61.21</td>
<td>38.79</td>
<td>57.58</td>
<td>42.42</td>
</tr>
</tbody>
</table>

*Figure 6: Flowsheet Spiral Separation*

The highest grade for the reprocessed tailing was also obtained at +600 µm (77.19 %) and +300-180 µm (78.07 %) respectively for the concentrates. This implies that feed particle size is one of the factor that mostly contribute to the recovery of Pb and Zn. Although, Wills, (2006) claims that the feed particle size is a variable that slightly influence separation mechanisms when compared to other concentration processes.

3.5 METAL RECOVERY TESTS
The results presented here are those of leaching and smelting tests carried out as described in section

![Graph](image1.png)

*Figure 7: Ore Concentrate Variation with Particle Size after Beneficiation using Spiral Separation*

3.5.1 Ore Pretreatment by Microwave
300g of the representative samples was used and after microwave induction for 60 seconds weight was observed to be 293g and 295g after being weighed and 2.33% and 1.66% weight loss.

**Leaching**
Table 9 and Table 10 show the result of leaching tests on the sample.
Figure 8: Tailing concentration Variation with Particle Size after Beneficiation using Spiral Separation

Table 9: Result of Leaching the Ihitutu Lead-Zinc Ore

<table>
<thead>
<tr>
<th></th>
<th>As-mined Sample</th>
<th>Microwaved Sample</th>
</tr>
</thead>
<tbody>
<tr>
<td></td>
<td>$H_2$SO$_4$</td>
<td>Aqua Regia (1:1)</td>
</tr>
<tr>
<td>Weight of Ore (g)</td>
<td>250</td>
<td>250</td>
</tr>
<tr>
<td>Weight of Solvent (g)</td>
<td>820</td>
<td>610</td>
</tr>
<tr>
<td>Total Weight of Solution (g)</td>
<td>1060</td>
<td>855</td>
</tr>
<tr>
<td>Weight of Residue after leaching (g)</td>
<td>130</td>
<td>120</td>
</tr>
<tr>
<td>Weight of Metal Leached into the Solution (g)</td>
<td>120</td>
<td>130</td>
</tr>
</tbody>
</table>

Table 10: Atomic Absorption Spectrometer Analysis of the Leachate

<table>
<thead>
<tr>
<th>Sample</th>
<th>ARAM$_1$</th>
<th>ARAM$_2$</th>
<th>ARAM$_3$</th>
<th>ARM$_1$</th>
<th>ARM$_2$</th>
<th>ARM$_3$</th>
</tr>
</thead>
<tbody>
<tr>
<td>Pb (mg kg$^{-1}$)</td>
<td>12.60</td>
<td>34.40</td>
<td>150.50</td>
<td>4.50</td>
<td>24.03</td>
<td>185.20</td>
</tr>
<tr>
<td>Zn (mg kg$^{-1}$)</td>
<td>0.11</td>
<td>6.50</td>
<td>34.00</td>
<td></td>
<td>10.00</td>
<td>54.25</td>
</tr>
</tbody>
</table>

<table>
<thead>
<tr>
<th>Sample</th>
<th>HSAM$_1$</th>
<th>HSAM$_2$</th>
<th>HSAM$_3$</th>
<th>HSM$_1$</th>
<th>HSM$_2$</th>
<th>HSM$_3$</th>
</tr>
</thead>
<tbody>
<tr>
<td>Pb (mg kg$^{-1}$)</td>
<td>10.40</td>
<td>52.20</td>
<td>137.20</td>
<td>6.34</td>
<td>34.52</td>
<td>160.40</td>
</tr>
<tr>
<td>Zn (mg kg$^{-1}$)</td>
<td>0.60</td>
<td>5.53</td>
<td>21.50</td>
<td>0.43</td>
<td>7.49</td>
<td>37.67</td>
</tr>
</tbody>
</table>

* AR – Aqua-Regia  * HS – $H_2$SO$_4$  * AM – As-mined  * M – Microwave  * Day

Figure 9: AAS Analysis of the Leachate
Figure 9 shows that aqua regia has stronger ability for better yield than sulphuric acid, also the highest value (185.20 mg kg⁻¹ Pb and 54.25 mg kg⁻¹ Zn) in the microwaved ore and as-mined because of the radiation from the microwave that has broken the bonds between individual mineral in the ore and helping to free the metals for better yield during leaching shown in Figure 10 and Figure 11. Metal solution are withdrawn on daily basis and most of the metals dissolved in the acids, dissolving of metals led to the gradual increase in the concentration of the metals in the ore shown in Figure 4.23 and increased to the maximum level after day 3. This was most probably due to the precipitation of lead in solution i.e., reversion of reaction. A similar kind of increase in lead recovery with decreasing reaction duration was also observed by other researchers (Nunez, 1985). To get a maximum yield generally researchers should have leached for days instead of the instant leaching that is done for some hours. This type of leaching will show that metals are still in the solution. There is also a need for microwave pretreatment which gives a greater yield than the unmicrowaved ore. The daily withdrawal observation has shown that the release of metal has been gradual likely due to different bonding of the metals to the ore. Moreover, the bond between Pb and Zn in the ore greatly influenced the result, the increase in the recovery per day is due to the stronger bond that exist between the ore. This implies that the day 1 samples are of stronger bond than day 2 and day 3 respectively.
3.6 Roasting – Smelting
Roasting released about 3.81% volatile matters (Table 11). The result revealed that the process is efficient since most previous reports by Egunlue et al., (2006); Oluwaseyi and Egunlue, (2007) on sulphide roasting varied from 2.85 – 3.85% weight loss after roasting. The reducing furnace allowed high oxidation with the addition of 50 g of baking flour thoroughly mix with the ore. Three possible chemical reactions occurred during roasting operation which are oxidizing roasting - the burning out of sulphur from sulphide ore which are then replaced wholly or in part by the oxide (Equation 5); sulphaing roast - the conversion of certain metallic sulphides to Sulphate (Equation 6); and volatizing roast - which eliminates all other elements with volatile oxides (As$_2$O$_3$) as represented in equation 7. The roasting temperature 700 ± 50 °C was found suitable for Pb and Zn dissolution.

\[
2\text{PbS}_{(s)} / 2\text{ZnS}_{(s)} + 3\text{O}_2(g) = 2\text{PbO}_{(g)} / 2\text{ZnO}_{(g)} + 2\text{SO}_2(g)
\]

(5)

\[
\text{PbS}_{(s)} / \text{ZnS}_{(s)} + 2\text{O}_2(g) = \text{PbSO}_4_{(g)} / \text{ZnSO}_4_{(g)}
\]

(6)

\[
2\text{As}_{(s)} + \frac{3}{2}\text{O}_2 (g) = \text{As}_2\text{O}_3
\]

(7)

The reduction of PbO and ZnO was done by smelting at 1200 ± 50°C which was found suitable with the addition of 50 g of reducing agents each (charcoal and Calcium Carbonate). The matte sank to at the bottom of the crucible while the slag layer was on top. The total time taken for the process was 2 hours 30 minutes.

<table>
<thead>
<tr>
<th>Particle Size</th>
<th>Initial Weight (g)</th>
<th>Final Weight (g)</th>
<th>Weight Loss (g)</th>
<th>% Volatile Matter</th>
</tr>
</thead>
<tbody>
<tr>
<td>+2360 – 53 μm</td>
<td>400</td>
<td>385.5</td>
<td>14.5</td>
<td>3.63</td>
</tr>
<tr>
<td>+2360 – 53 μm</td>
<td>400</td>
<td>390.1</td>
<td>9.9</td>
<td>2.43</td>
</tr>
<tr>
<td>+2360 – 53 μm</td>
<td>400</td>
<td>379.9</td>
<td>20.1</td>
<td>5.03</td>
</tr>
<tr>
<td>+2360 – 53 μm</td>
<td>400</td>
<td>384.8</td>
<td>16.4</td>
<td>4.1</td>
</tr>
<tr>
<td>Average</td>
<td>400</td>
<td>384.8</td>
<td>15.2</td>
<td>3.81</td>
</tr>
</tbody>
</table>

4.0 CONCLUSION
The ore deposit is predominantly made up of lead and zinc in the forms of galena (PbS) and sphalerite (ZnS) assaying 54.66 % Pb, 8.59 % Zn, 6.80 % W and 2.55 % Au on the. The photomicrograph from the mineralogical analysis revealed galena is found as interlocked form with sphalerite and gauge minerals. The grinding test revealed that media assisted grinding produced more size reduction than that of autogenous grinding. Also, the addition of difference mode is applicable in continuous grinding operation where grains get weaker internally and at the peripheral after each revolution are already weakened by impact are easily removed by any small impact, even small interaction between grains while the new mass charge is intended for use in batch grinding. Based on the result of determination of concentration criteria, it is obvious that lead and zinc cannot be separated from each other. Thus, separation to concentrate will include all heavy components while silica and other lighter components reported to tailings. The result from leaching revealed that aqua-regia has stronger ability for better yield and in the digestion of metals than sulphuric acid in both microwaved and as-mined ore. Based on this, irradiation from the microwave has broken the bonds between individual mineral in the ore. Also, the rate of leaching increases with decreasing number of days. The result from Table 14 revealed that 3.81% volatile matter and 14.5 g weight loss was observed during the roasting-smelting operation. The weight of slag was 231.9 g and melt was 153.6 g after smelting operation.

4.1 RECOMMENDATIONS
1. The processing and recovery of Nigeria ore should be done locally to reduce the dependence on imported material.
2. The Federal Government should as a matter of urgency, enact a deliberate policy that will compel all
investors in the mining industry to add value to minerals in the country before they are exported.

REFERENCES


