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# Gravity Separation and Leaching Beneficiation Study on Azara Nassarawa Barite Mineral Ore

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**Abstract-** The comparative study for the recovery of Azara barite mineral ore found in Nassarawa State, Nigeria using jigging and tabling gravity separations and leaching (with HCl and H<sub>2</sub>SO<sub>4</sub> Acids) processes of concentrates was investigated. The microstructural, chemical composition and physical properties of the as-mined sample of Azara barite mineral ore were analysed. The sample was then concentrated using the gravity separation and leaching processes. The microstructural and chemical composition analyses of the products of concentration were carried out to establish the effectiveness and efficiency of the methods for the recovery of Azara barite mineral ore. Xray Diffraction (XRD), Xray Fluorescence (XRF), Scanning Electron Microscope (SEM) and Energy Dispersive Spectrometry (EDS) tests; were used to carry out the morphology and chemical analysis of the Azara barite ore.

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# Gravity Separation and Leaching Beneficiation Study on Azara Nassarawa Barite Mineral Ore

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**Abstract-** The comparative study for the recovery of Azara barite mineral ore found in Nassarawa State, Nigeria using jigging and tabling gravity separations and leaching (with HCl and H<sub>2</sub>SO<sub>4</sub> Acids) processes of concentrates was investigated. The microstructural, chemical composition and physical properties of the as-mined sample of Azara barite mineral ore were analysed. The sample was then concentrated using the gravity separation and leaching processes. The microstructural and chemical composition analyses of the products of concentration were carried out to establish the effectiveness and efficiency of the methods for the recovery of Azara barite mineral ore. Xray Diffraction (XRD), Xray Fluorescence (XRF), Scanning Electron Microscope (SEM) and Energy Dispersive Spectrometry (EDS) tests; were used to carry out the morphology and chemical analysis of the Azara barite ore. From the results, the Azara barite ore contains approximately 36.2% barium (Ba) based on XRF and 50.5% barium (Ba) based on EDS tests with a specific gravity of 3.85. A random sieve size of -355+250 $\mu$ m was selected for jigging and tabling of the Azara barite ore. The EDS result of the products of jigging and tabling shows that jigging had a recovery of 69.8% Barium with an average specific gravity of 4.28 while tabling had a recovery of 69.5% Barium with an average specific gravity of 4.18. Leaching of the barite ore was done using 0.5 and 1.0 molar concentration solution of HCl and H<sub>2</sub>SO<sub>4</sub>. The results of the leaching process show that the super concentrates have average specific gravities of between 2.36 to 4.46 for different temperatures and molar concentrations of HCl and H<sub>2</sub>SO<sub>4</sub>. The best specific gravities obtained are 4.29, 4.39 and 4.46 at 30 minutes with 0.5 and 1.0 molar concentration of HCl and H<sub>2</sub>SO<sub>4</sub> respectively. Therefore, the results of this research work have established that the Azara barite ore found in Nassarawa state, Nigeria is suitable for oil and gas applications.

## I. INTRODUCTION

Barite or Baryte is a non-metallic mineral with an incredible specific gravity and it consists of barium sulphate (BaSO<sub>4</sub>) which belongs to the variety of caulk rock. The *barite group* consists of barite, celestine, anglesite and anhydrite. Barite is generally white or colourless, and is the main source (the primary-ore) of barium metal. Barite and celestine form a solid solution (Ba.Sr) SO<sub>4</sub>. The radiating form, sometimes referred to as *Bologna Stone*, attained some notoriety

among alchemists for the phosphorescent specimens found in the 17th century near Bologna by Vincenzo Casciarolo (1). A mineral is a naturally occurring inorganic substance possessing definite chemical compositions and ordered atomic structures. It is different from a rock (2). Minerals have played a major role in human life and have raised the standard of living which made them become increasingly important and we depend in countless ways. However, it is widely recognized that few countries can achieve any measure of meaningful industrialization and economic growth without a planned development with available mineral resources. Taking a closer look at solid mineral sector, it would not be an exaggeration to say that the industry is fully developed and can be an equal substitute to the oil and gas industries in terms of foreign exchange capacity. This assertion is based on the fact that this nation is endowed with untapped resources in commercial quantities which can be exploited economically at profit (3). Barite is a hydrothermal deposit which originated from hot aqueous solution in joint fault, permeable rock formation and fractures within the Benue trough of Nigeria, notably in Benue, Taraba, Adamawa, Gombe, Plateau, Nassarawa, Ebonyi and Cross-River states (3). Barites bearing veins are found mostly in the area geologically referred to as Middle Benue Trough. Nigeria has an estimated reserve of over two million (2,000,000) metric tons of barite ore scattered in different parts of the country to include Benue, Nasarawa, Plateau, Taraba and Cross River states (4). Barite ore occurs and has been mined at the following major locations in large quantity:

- Nasarawa State: Azara, Keana, Kuduku, Alosi all at Keana LGA
- Benue State: Gboko, Guma, Gwer, Ushongo, Makurdi, Konshisha
- Gombe and Adamawa: Gban and Mayo-Kpoki; Gombe and Liji hills
- Cross Rivers State: Obubra, Ikom, Akpet central, and Ogoja LGA
- Plateau State: Yelwan Shendam LGA
- Taraba state: Serikinkudu Akire LGA (4).

The mining of minerals in Nigeria accounts for only 0.3% of its GDP, due to the influence of its vast oil

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resources. The domestic mining industry is underdeveloped, leading to Nigeria having to import minerals that it could produce domestically, such as salt or iron ore. Rights to ownership of mineral resources is held by the Nigerian government, which grants titles to organizations to explore, mine, and sell mineral resources. About 34 minerals deposits have been identified in Nigeria and one of such is barite ore. Barite has many applications; the most significant of it is its usage by the oil companies when drilling for crude oil or petroleum. Other importance of barites is highlighted below:

- Barite is used in the manufacturing of drilling mud without which petroleum prospecting will be impossible. In fact, the demand for Barite by the oil companies is more than the demand for water by human beings but the supply is very low because only a very few individuals are aware of this business opportunity.
- Barite is used by chemical industries in the manufacturing of Barium compounds such as chloride, nitrate, carbonate and hydrate.
- Barite is used as drilling fluids in oil and petroleum industries and in paper and plastics productions.
- It is used by metallurgical industries for brass melting, textiles industries as weighting materials and used in manufacturing glass, paints etc.

The demand for barite by the oil and gas industries is practically more than the demand for water by humans. Despite intense extraction of barite ore in Azara, Nassarawa state over the years; production has remained low. The need for the most efficient method of processing the barite mineral, to boost the local supply due to its enormous applications is the reason for this study since there is steady and increasing demand for the product because of the numerous industrial uses of barite ore. This will facilitate a better exploitation of the resources which will in the long run sustain its enormous application towards the satisfaction of local content. This will go a long way to promote the development of the area in the form of revenue generation, provision of superstructure and infrastructural development. The quality of the Nigerian Barites is moderate to high. It is often associated with fluorite, calcite, dolomite, quartz, etc. The major impurities are quartz, iron oxide (goethite), and carbonates of iron, calcium and magnesium. These impurities tend to increase the ore volume, suppress and reduce the specific gravity of the unprocessed barites to about 2.0 – 4.0. The cost of processing is increased and the oil mills wear out rapidly. The goethite and silica impurities can be removed by magnetic and gravity separation. Once processed the specific gravity of the Nigerian barite increases and meets the 4.2 – 4.5 specified value (5).

## II. EXPERIMENTAL

The gravity separation of the sample of Azara barite was carried out using Jigging and Shaking Table processes with Jigs and Shake tables respectively. The barite was initially crushed and ground, sieved to a particle size of  $-355+250\mu\text{m}$  and subjected to Jigging and Tabling respectively. After the separation processes, jigging had Underflows (Concentrates) and Overflows (Tailings) while tabling yielded Concentrates, Middlings and Tailings. All the samples were simultaneously placed in a Gen Lab Drying Oven with a temperature of about  $120^{\circ}\text{C}$  and left for an average time of 2 hours to dry. Apparent density / specific gravity (after gravity separation) were got for all the samples: concentrates, middlings and tailings; which were compared with the required standard. The microstructure and elemental / chemical composition analysis was carried out on the concentrate samples to check for the composition of the barite and any possible impurity that may still be present in the concentrates after the gravity separation processes using Scanning Electron Microscope (SEM) and Energy Dispersive X-ray Spectrometry (EDS) tests.

New set of samples (6 samples at 10g each) were measured out from both gravity separation concentrates (Jigging and Tabling) and subjected to Leaching processes / methods to achieve super concentrates (a total of 12 samples).

40g of both Sodium Carbonate ( $\text{Na}_2\text{CO}_3$ ) and Potassium Carbonate ( $\text{K}_2\text{CO}_3$ ) were weighed and half the quantity arranged in a crucible forming a 6mm deep layer. 10g of the Barite sample ( $\text{BaSO}_4$ ) was also weighed and poured into the crucible; the other half of the above was placed also in the crucible, on top of the barite and stirred. The mixture in the crucible was covered and crucible placed in a furnace raised to  $950^{\circ}\text{C}$ , which is about 60% of the melting point temperature of barium metal; until fusing was achieved (varying fuse time). The crucible was allowed to cool after taken out of the furnace; it was rotated during cooling so that the fused mass solidifies into a thin layer.

The fused mass was leached out with about 250ml of hot distilled water with crucible placed into a 500ml glass beaker with thorough stirring. It was filtered through a whatman '41' filter paper and residue washed several times (about 10 to 12 times) by decantation with hot distilled water; The sample was washed on the filter paper to remove the sulphates.

50ml of dilute Hydrogen Chloride acid, HCl (of a particular molar concentration) and about 250 ml of hot distilled water were used to dissolve the residue from the filter paper respectively, catching the solution in a 500ml glass beaker. 10g of Ammonium Chloride ( $\text{NH}_4\text{Cl}$ ) was added into the solution. The solution was neutralized with a quantity of Ammonium hydroxide

solution ( $\text{NH}_4\text{OH}$ ) using methyl red as an indicator. The solution was boiled for about 5 minutes and filtered through the filter paper. The residue was dissolved with hot distilled water for about 5 to 6 minutes and filtered through the filter paper. Again, the residue was washed 4 to 5 times with hot distilled water by decantation. It was taken into a 500ml glass beaker and few drops of methyl red were used as an indicator. The solution was neutralized with 50ml of dilute Hydrogen Chloride acid, HCl (of the same molar concentration) and 250ml of hot distilled water was added.

It was boiled for about 10 minutes and 50ml of hot ammonium sulphate solution ( $(\text{NH}_4)_2\text{SO}_4$ ) was dropped with constant stirring to prevent coprecipitation of calcium and magnesium particles. The solution was again boiled for about 5 minutes and the conical flask was brought down from the hot plate and allowed to cool / rest over night to precipitate. It was filtered the next morning with the filter paper and rinsed with 50ml of dilute Tetraoxosulphate VI acid,  $\text{H}_2\text{SO}_4$  (of the same molar concentration of the HCl) and then washed several times with hot distilled water by decantation; scrubbing off the precipitate until the solution was free from chlorides. The sample was then transferred to a plate and ignited in an oven to dry at an average temperature of about  $120^\circ\text{C}$ . It was then brought out of the oven, allowed to cool, weighed and packaged.

These processes were carried out for 0.5 and 1.0 molar concentrations of HCl and  $\text{H}_2\text{SO}_4$  acids with different fusing time (furnace hold time) variations of 30 minutes, 60 minutes and 90 minutes, on both gravity separation concentrates (Jigging and Tabling); bringing up a total summation of 12 test samples. Finally, the results of the different samples were analysed as their apparent density / final specific gravity (after the leaching process) were compared.

### III. RESULTS AND DISCUSSION

The XRD pattern in Fig 1 confirms the Barite ( $\text{BaSO}_4$ ) phase pattern in line with literature and therefore indicates that the Azara barite ore can be used industrially, especially in the oil and gas sections.

The XRF elemental composition of the as-mined Azara barite ore with a specific gravity of 3.85 shows that the ore contains 36.2% Barium, 34.4% Sulphur, 14.7% Titanium, 5.5% Vanadium, 1.8% Aluminium, and 1.5% Silicon, amidst others. This shows that the specific gravity value obtained confirms that the barite ore in Nigeria has a specific gravity between 3.0 to 4.0 and has low percentage contents of barium and sulphur elements in the presence of other elements (impurities).

Scan electron photomicrograph shows the fracture surface of barite crude that is representative of the barite ore deposit in Azara LGA of Nassarawa State, Nigeria (see Fig 2).

Fig 3 represents the Energy Dispersive Spectroscopy (EDS) peak intensities of elemental composition of the as-mined, showing the different peaks with barium element (metal) at the highest peak which gave the distribution of elements and their compositions, thus: 50.5% Barium, 12.6% Sulphur, 22.7% Oxygen, 5.0% Gold, 2.4% Molybdenum, 2.3% Lead, 1.6% Tungsten, 0.7% Sodium, 0.6% Zinc, 0.4% Niobium, 0.3% Copper and Silicon respectively, 0.2% Aluminium, Potassium and Rubidium respectively, amidst others; which confirms the XRF results of the as-mined Azara barite ore.

Table 1 shows the result of gravity separation concentrates using jigging operation. -350 + 250 $\mu\text{m}$  particle size of the feed material was used for the jigging operation. 76% of the feed was recovered as underflow (concentrates) and 8.4% of the feed was recovered as overflow (tailings) with 15.6% loss during the jigging operation process.

Table 2 shows the result of gravity separation concentrates using tabling operation. -350 + 250 $\mu\text{m}$  particle size of the feed material was used for the tabling method. 52.3% of the feed was recovered as concentrates, 40.3% as middlings, and 0.3 as tailings with 7.0% loss during the tabling operation process.

Fig 4 and Fig 5 represents the scan electron photomicrograph of the concentrates after jigging and tabling respectively. This shows a clearer SEM microstructure than that of the as-mined ore and similar to the microstructure of a standard barite ore.

Table 4.6a and 4.6b, Table A.3, Table A.4 and Table A.5 (Appendix A) respectively represents the specific gravities of the super concentrates samples of A to L, where Samples A, B, C and D fused by heating for 30 minutes in the furnace have higher specific gravities as compared to samples E, F, G, H, I, J, K and L heated for 60 minutes and 90 minutes respectively. It was noticed that Samples B and D leached with 1.0 Molar Concentration of HCl and  $\text{H}_2\text{SO}_4$  have higher specific gravities as compared to samples A and C leached with 0.5 Molar Concentration of the same acids.

Fig 6 and Fig 7 shows the graphical representations of specific gravity of the super concentrates on Y-axis against fusing time on X-axis for 0.5 and 1.0 molar concentrations respectively.

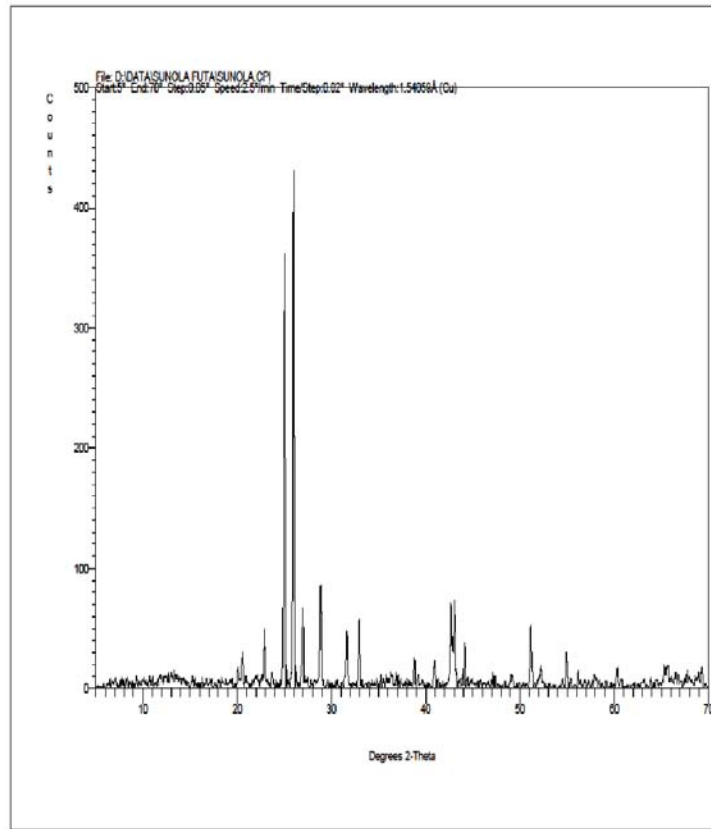


Fig. 1: Xray Diffraction (XRD) Phase Pattern of the As-mined barite ore

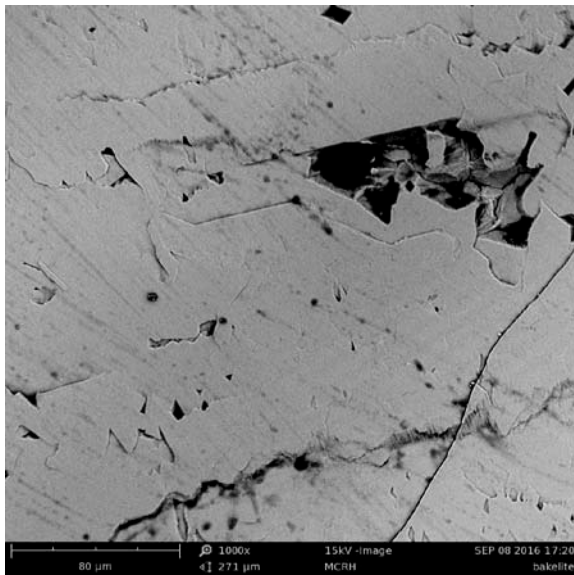


Fig. 2: SEM Microstructure of As-mined Azara barite

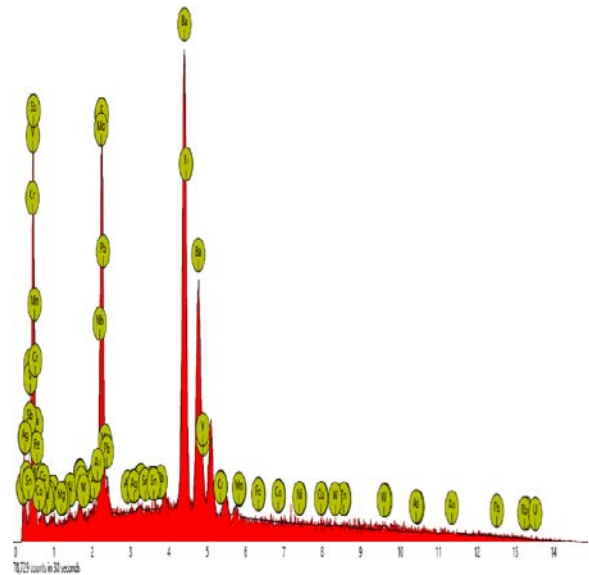


Fig. 3: EDS Elemental Composition of Azara barite



*Table 1:* Result of Concentration of Azara barite ore using Jigging method

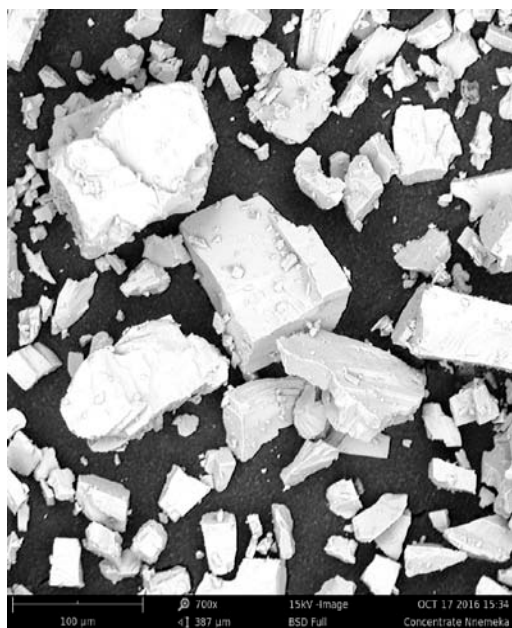
| SIEVE SIZE         | FEED | UNDER FLOW | OVER FLOW | LOSS | UNDER FLOW | OVER FLOW | LOSS  |
|--------------------|------|------------|-----------|------|------------|-----------|-------|
| -350 + 250 $\mu$ m | 500g | 380g       | 42g       | 78g  | 76%        | 8.4%      | 15.6% |

*Table 2:* Result of Concentration of Azara barite ore using Tabling method

| FEED | CONCENTRATE | MIDDLING | TAILING | LOSS | CONCENTRATE | MIDDLING | TAILING | LOSS |
|------|-------------|----------|---------|------|-------------|----------|---------|------|
| 600g | 314g        | 242g     | 2g      | 42g  | 52.3%       | 40.3%    | 0.3%    | 7.0% |



*Fig. 4:* SEM Microstructure of the Jigging underflow



*Fig. 5:* SEM Microstructure of the Tabling concentrates

*Table 3:* Specific Gravity (S.G) of the Theoretical Standard and the Super Concentrates of Azara barite ore using 0.5 and 1.0 Molar Concentrations (M) of HCl and H<sub>2</sub>SO<sub>4</sub>

| SAMPLES              | SPECIFIC GRAVITY |
|----------------------|------------------|
| THEORETICAL STANDARD | 4.20 – 4.50      |
| A                    | 4.29             |
| B                    | 4.46             |
| C                    | 4.19             |
| D                    | 4.39             |
| E                    | 3.92             |
| F                    | 3.85             |
| G                    | 2.69             |
| H                    | 2.65             |
| I                    | 3.51             |
| J                    | 3.45             |
| K                    | 2.68             |
| L                    | 2.36             |

Where:

- Sample A = Underflow, 30mins Fusing time, 0.5 Molar concentration
- Sample B = Underflow, 30mins Fusing time, 1.0 Molar concentration
- Sample C = Concentrate, 30mins Fusing time, 0.5 Molar concentration
- Sample D = Concentrate, 30mins Fusing time, 1.0 Molar concentration
- Sample E = Underflow, 60mins Fusing time, 0.5 Molar concentration
- Sample F = Underflow, 60mins Fusing time, 1.0 Molar concentration
- Sample G = Concentrate, 60mins Fusing time, 0.5 Molar concentration
- Sample H = Concentrate, 60mins Fusing time, 1.0 Molar concentration
- Sample I = Underflow, 90mins Fusing time, 0.5 Molar concentration
- Sample J = Underflow, 90mins Fusing time, 1.0 Molar concentration
- Sample K = Concentrate, 90mins Fusing time, 0.5 Molar concentration
- Sample L = Concentrate, 90mins Fusing time, 1.0 Molar concentration

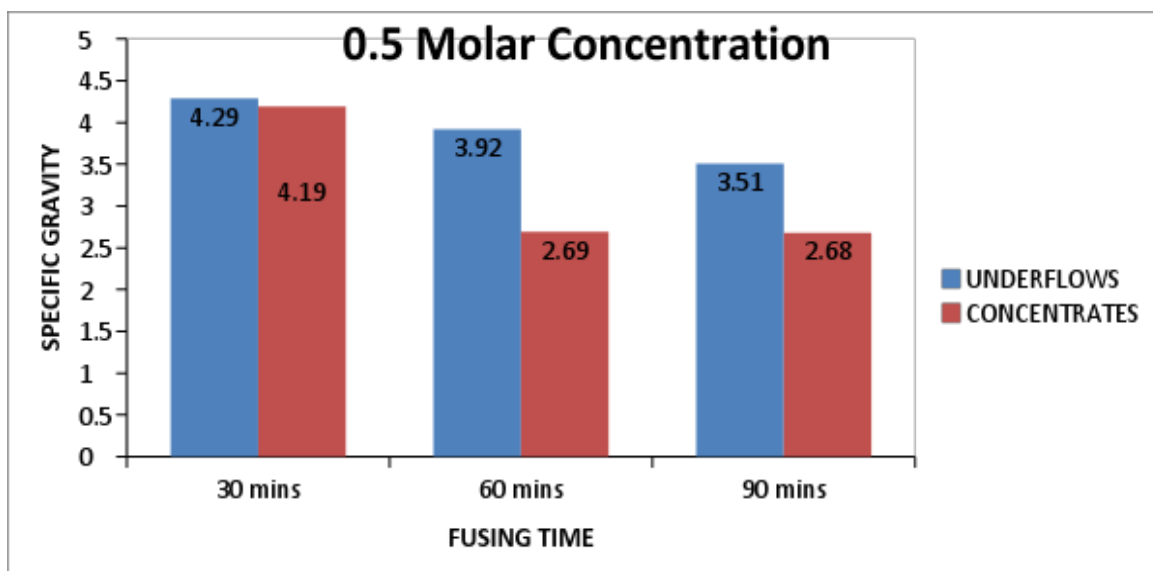


Fig. 6: Bar Chart of Specific Gravity of leached Azara barite ore using 0.5 Molar Concentration of HCl and H<sub>2</sub>SO<sub>4</sub>

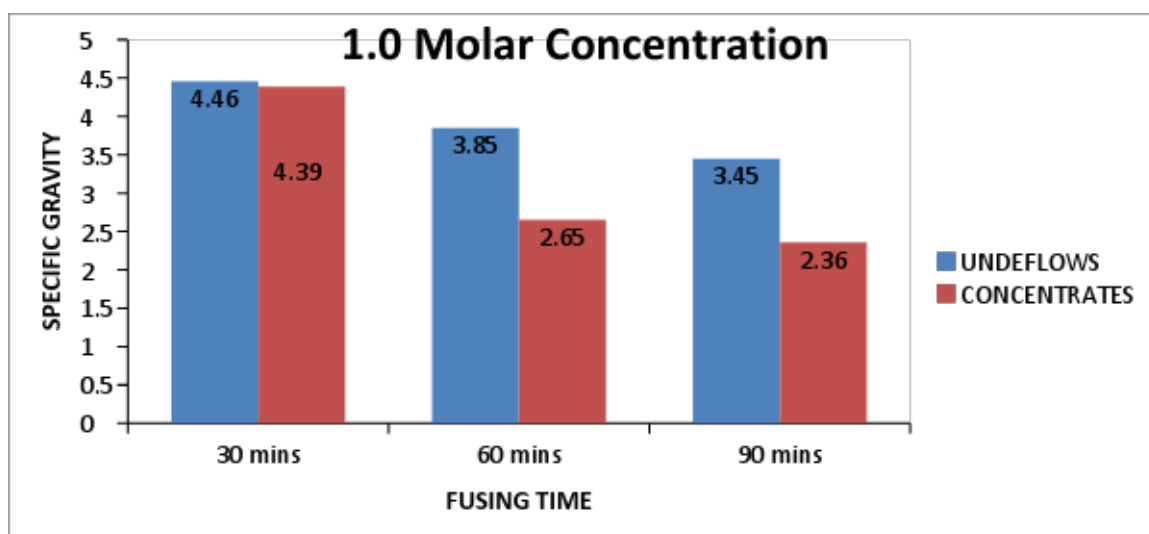


Fig. 7: Bar Chart of Specific Gravity of leached Azara barite ore using 1.0 Molar Concentration of HCl and H<sub>2</sub>SO<sub>4</sub>

#### IV. CONCLUSION

Azara barite contains about 36.2% to 50.5% barium metal, 12.6% to 34.4% sulphur and about 22.7% oxygen. Azara barite ore heat treated for 30 minutes and leached with 1.0 molar concentration of HCl and H<sub>2</sub>SO<sub>4</sub> gave the highest (best) specific gravity values of 4.46 and 4.39 of all the selected heat treatment (fusing) time as compared to the standard of 4.20 to 4.50 specific gravity required by the oil and gas industries. The specific gravity of Azara barite ore found in Nassarawa State, Nigeria has been upgraded to a range of 4.39 to 4.46 from the 3.85 specific gravity of the as-mined ore. Therefore, the results of this research work have established that the Azara barite ore found in Nassarawa state, Nigeria is suitable for oil and gas applications.

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