



BENEFICIATION AND CHARACTERIZATION OF BARITE MINERAL ORE FOR INDUSTRIAL APPLICATION.

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ABSTRACT

Barite ore was beneficiated using jigging and froth flotation

techniques and then the beneficiated barite was also treated with sulphuric acid and NaOH separately.

Chemical analysis indicated presence of major elements Ba, S, O and other minor elements. During the jigging operation grinded barite was placed in the jigging machine with constant water supply and both the overflow and underflow are collected, the concentrate from the jigging machine

Introduction

Various research has shown that Nigeria is home to different industrial non-metallic minerals but a good number of these industrial non-metallic minerals have experienced very little or no exploration at all (Raw material research and development, 2010). Barite is one of these minerals with the lowest exploration, it is an important industrial raw material that has many uses which includes its usage as a pigment for making paints, as well as in paper coating applications, and most importantly in Nigeria it is used as a weighting agent during drilling for crude oil. Considering the continual economic activities in the oil and gas sector, there will always be a demand for barite and this is due to its high specific gravity value, barite helps to counteract pressure formation during drilling thereby giving the drilling bit the necessary density required (Searls 2003). Some embargoes were placed on the importation of barite by the Nigerian Government since the year 2003 just to improve the exploration of local barite in the country (Mills 2005). A petition on the relaxation of the embargo placed on imported barite was made by the International Oil Companies (IOCs) operating in Nigeria in year 2013. The petition was on the basis that barite produced in Nigeria have lower quality (specific gravity) compared to the imported ones.



is passed into the froth machine, the collector, the frother and the depressant were added to the pulp after mixing in the froth machine the concentrates are collected and analyzed. The XRF characterization carried out on the raw sample shows the elemental composition of Ba is 47.231, S is 9.192, and O is 3.567, and after beneficiation Ba increased to 76.1639, S decreased 4.703, but after treatment with sulphuric acid there was a significant improvement of Ba to 89.5034 than when it was treated with NaOH 85.9736. FTIR analysis was also carried out on the barite samples to determine the functional groups present in barite. The specific was also calculated using a pycnometer, the specific gravity of the raw sample is evaluated as 3.521, after beneficiation process the specific gravity slightly increased to 3.704, after treating with base and acid separately, the specific gravity was calculated to be 3.906 and 4.102 respectively.

Keywords: Barite, Beneficiation, froth flotation, Jigging operation, XRF, Specific gravity

Majority of the barite minerals being used in Nigeria, are imported. This is due to the challenge encountered by the ones being processed in Nigeria, and the major challenge is the low specific gravity values which is below the minimum industrially accepted standard of 4.2 for barite (Mgbemere *et al.*, 2018). This is mainly due to the fact that a lot of impurities are associated with the barite ores, these impurities can be in the form of silicate oxide, iron, magnesium, aluminum oxide and a whole lot of other debris all of which can hamper the quality of the barite mineral. In order to improve the quality of the barite, the barite has to undergo a process called beneficiation, which includes techniques and processes that are used to improve the purity and quality of minerals from their ores.

There are various beneficiation processes used in mineral purification and improvement, they include magnetic separation, gravity separation, tubular separation, flotation process, jigging operation, froth flotation and many other processes. Jigging and froth flotation operations involve washing the grinded barite mineral in the jigging machine and frother at a particular constant speed, then the collector and depressant is added before the treated sample is collected and analyzed (Raju *et al.*, 2004). The beneficiated barite can then be characterized to determine its elemental composition, atomic and molecular structure of the barite.

This research is focused on the use of the jigging and the froth flotation to beneficiate Barite ore to improve its quality (Barite composition) and also increase its specific gravity



value. And also, how treatment of barite with acid or base affects its specific gravity and reduces the silicate present in the barite mineral.

Experimental Methodology

Sample Preparation and Beneficiation

The collected samples were mixed, broken manually with a sledge hammer to provide a required size (60mm) acceptable to laboratory jaw crusher. The samples were crushed, ball-milled, then coned and quartered to yield a representative sample.

500g of the sample was obtained from the head sample form the feed. Jig machine was rinsed with clean water. The spigot hutch compartment was placed firmly with the rubber cork and filled with water to cover the ragging in the feed compartment. The feed was fed into the jig and the process was allowed for 10 minutes. The spigot opened and the products collected as the underflow while the material through the feeding compartment referred to as overflow. The two products dewatered, dried, weighed and recorded.

A 12D Denver laboratory type flotation machine was used to produce the mineral concentrate. The Impeller speed of the machine was kept constant at 1500 rpm for both conditioning and flotation. The pulp was mixed for three minutes prior to addition of collector (Oleic acid) and after an additional mixing of three minutes, the frother (Pine oil) was added Depressant (Sodium silicate) also added. After three-minute mixing period, air was introduced into the cell and the froth products collected for five minutes (Wang *et al.*, 2014).

Acid leaching test performed on final flotation concentrate using concentrations of Sulphuric acid (97.99%). 50 g portions of concentrate were taken in to Pyrex glass beakers (500 mL), treated with 100 mL diluted of acid, with constant stirring on hot plate at 95 °C to boiling temperature for 1 h with speed of 10 rpm. These were cooled, filtered and washed with distilled water until free of acid. The residues left on filter paper were dried for 20 minutes in electric oven at 110 °C and the final product examined using XRF machine for Barite content. (Achusim-Udenko *et al.*, 2011). Alkaline treatment also performed on final flotation concentrate using concentrations of Sodium hydroxide (50%). 50 g portions of concentrate were taken in to Pyrex glass beakers (500 mL), treated with 100 mL diluted of Base, with constant stirring on hot plate at 95 °C to boiling temperature for 1 h with speed of 10 rpm. These were cooled, filtered and dried. the final product examined using XRF machine for Barite content.

Relative density bottle (Pycnometer) and weighing balance method was used in the determination of the specific gravity of the material. The specific gravity bottle was weighed and the value recorded as W_1 , then partly filled with barite sample and the value recorded as W_2 . Water was added to the sample to fill the bottle and the value recorded as W_3 . If W_4 is the weight of the water in the bottle, then the specific gravity of the test material is given as:

$$SG = \frac{W_2 - W_1}{(W_2 - W_1) - (W_3 - W_4)}$$



Sample Characterization

A) X-RAY Fluorescence (XRF)

This method operates on the principle of atomic physics and quantum chemistry. The specimens were exposed to the entire spectrum of photons consisting of primary radiations emitted from a standard X-ray tube. These irradiated specimen causing the elements in it to emit secondary fluorescence with their characteristics X-ray line spectra. The technique is carried out through the following steps:

1. Crushing of each sample with an electric crusher and then pulverized for 60 seconds using Herzog Gyro-mill (Simatic C7-621).
2. Pellets were prepared from the pulverized sample, first by grinding 20g of each sample with 0.4g of stearic acid for 60 seconds. After each grinding, the Gyro-mill was cleansed to avoid contamination.
3. 1g of stearic acid was weighed into an aluminium cup to act as binding agent and the cup was subsequently filled with the sample to the level point.
4. The cup then taken to Herzong pelletizing equipment when it was passed at a pressure of 200KN for 60 seconds.
5. The 2mm pellets were added into a sample holder of the x-ray equipment (Phillips PW1800) for analysis.

B) Fourier Transform Infrared Spectroscopy (FT-IR)

This analysis was performed in all samples isolated to have a prompt result regarding the bio mineral. A few crystals were mixed with KBr (Merck for spectroscopy) and pulverized in an agate mortar to form a homogenous powder from which, under a pressure of 7 tons, the appropriate pellet was prepared. All spectra were recorded from 4000 to 400 cm^{-1} using the Perkin Elmer 3000 MX spectrometer. Scans were 32 per spectrum with a resolution of 4 cm^{-1} . The IR spectra were analysed using the spectroscopic software Win-IR Pro Version 3.0 with a peak sensitivity of 2cm^{-1}

Discussion of Results

Table 3.1: Table of raw barite composition

Sample Elements	Concentration
O	30.567
Mg	0.237
Al	0.862
Si	7.130
S	9.192
K	0.304
Ca	1.222
Ti	1.673
Fe	0.312
Zn	0.010
Ba	47.231
Pb	0.010



Table 3.2: Table of composition of beneficiated barite treated with acid and then base

Elements	Composition after Beneficiation	Composition after treatment	Composition after Acid treatment	Composition after base treatment
Mg	0.0031	< LOD		< LOD
Al	1.0014	0.0341		0.1196
Si	16.0624	8.2819		6.9703
S	4.7031	2.0624		6.0211
K	0.0211	< LOD		< LOD
Ca	0.7543	0.0051		0.0089
Ti	0.0251	< LOD		< LOD
Fe	0.0811	0.0032		0.0028
Ag	0.0002	< LOD		< LOD
Zn	0.0003	< LOD		< LOD
Ba	76.1639	89.5034		85.9736
Pb	< LOD	< LOD		< LOD

< LOD= Low Detection

Table 3.3: Results of Specific gravity

Parameter	Raw Sample	Beneficiate Conc.	d	Leaching Prod. By Acid	Leaching Prod. By Alkaline
W₁	23.3	23.3		23.3	23.3
W₂	28.3	28.3		28.3	28.3
W₃	75.18	75.25		75.36	75.32
W₄	71.6	71.6		71.6	71.6
Specific Gravity	3.521	3.704		4.102	3.906

Figure 3.1: FTIR result of raw barite ore

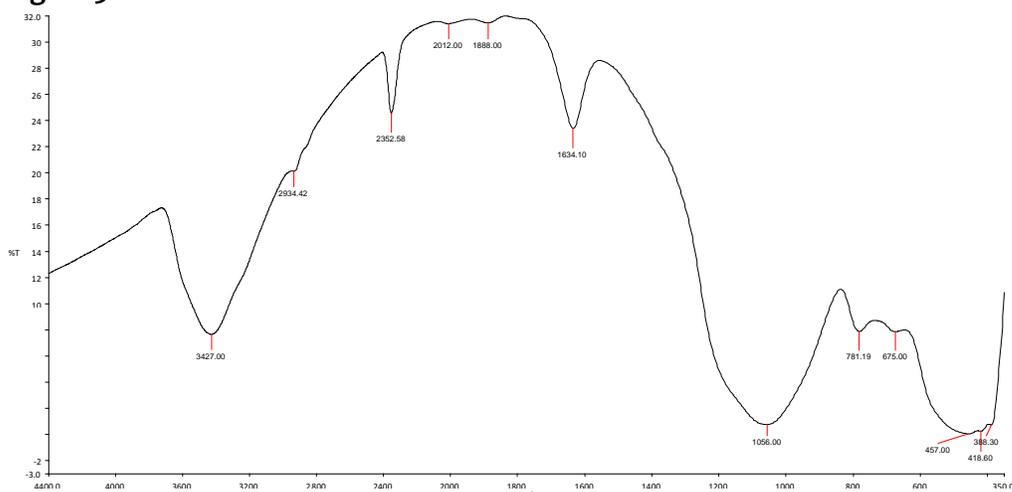




Figure 3.2: FTIR result of base treated barite

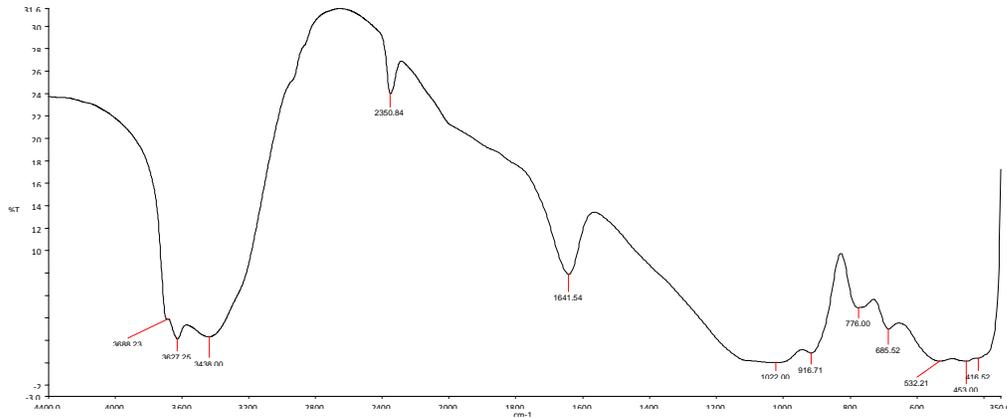
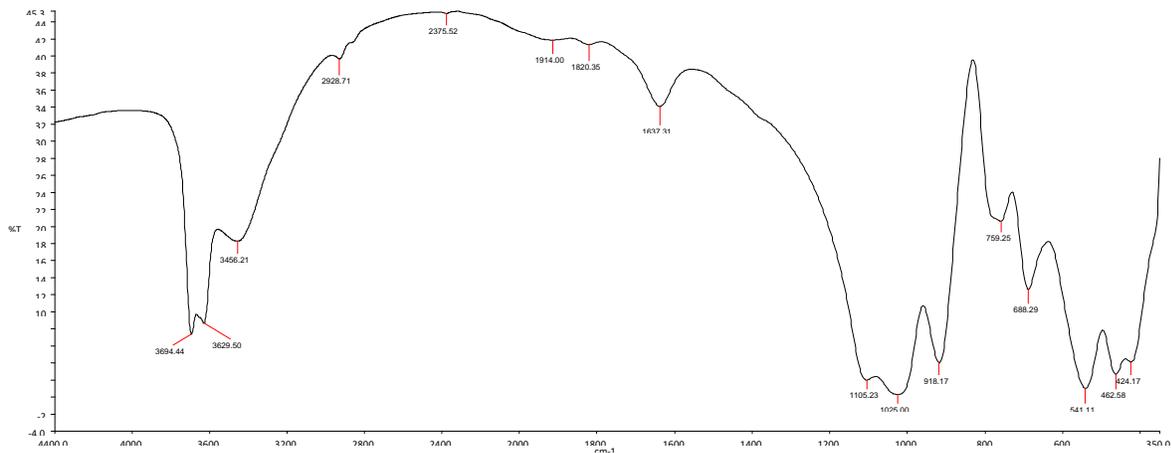


Figure 3.3: FTIR result of acid treated barite



Barite characterization

A) XRF analysis

Barite was characterized by XRF. This technique is used to determine the elemental compositions contained in the barite sample. Table 3.1 shows the results of the chemical composition analysis for barite sample before the beneficiation process. The barite ore is associated with a lot of impurities and an analysis of the ore using X-ray Fluorescence indicated that apart from the major elements namely; barium and sulphur, about 10 other elements were observed. While table 3.2 shows the results of the chemical composition analysis for barite sample after beneficiation and further treatment with acid and base. Alongside the major element barium were other elements which are the minor elements and impurities. From table 3.1 it can be observed that the raw analysis of the barite mineral gave a barium composition of 47.271, and after beneficiation it increased to 76.1639. However, as shown in table 3.2



on further treatment of the beneficiated barite with acid, the barium increased to a significant 89.5034.

B) FTIR analysis

Barite was also characterized by FTIR. This technique could be used to identify the major functional groups consisting barite. From the wave number of the molecular vibrations modes, a good explanation of the chemical structure could be obtained. As illustrated in Figure above, the characteristic IR band of montmorillonite appeared at $3,412\text{ cm}^{-1}$. This band represents the fundamental stretching vibrations of different –OH groups present in Mg–OH–Al, Al–OH–Al, and Fe–OH–Al units in the octahedral layer. The strong peak appears at $1,012\text{ cm}^{-1}$ is related to the stretching vibrations of Si–O groups, while the bands at 525 and 466 cm^{-1} are due to Ba–O–Si and Si–O–Ba bending vibrations, respectively. The peak at 878 cm^{-1} is assigned to O–Si–O asymmetric stretching, while the peak at 406 cm^{-1} is due to O–Si–O bending mode. On the other hand, the peak at $1,650\text{ cm}^{-1}$ is for H–O–H bending whereas the peak at 878 cm^{-1} is due to OH bending bounded to Fe $3p$ and Al $3p$.

Figure 3.2 gives strong indication that the aim of purification process is achieved. According to this figure, the intensity of absorbance at $1,012\text{ cm}^{-1}$ peak, which relates to Si–O groups, decreased after purification. This can be explained by the fact that the SiO 2 amount decreases after purification process.

From fig 3.1, deformation relative to the OH hydroxyl groups are presented. The vibration bands of valence at 3400 and 3500 cm^{-1} and the deformations at 1619 and 1641 cm^{-1} are characteristic of the vibration of the hydroxyl function of the water of hydration of the barite 420. The Si-O group band, intense centered around 1139 , 639 and 485 cm^{-1} are ascribed to binding valence vibration in barite minerals 420. The vibrational band of valence at 2039 cm^{-1} is attributed to the characteristics of the CO stretching, which confirms the presence of carbonates and other organic impurities in the barite.

Specific Gravity Calculation

Specific gravity of the test material is given as:

$$SG = \frac{W_2 - W_1}{(W_2 - W_1) - (W_3 - W_4)}$$

W_1 = weight of empty density bottle, W_2 = weight of empty density bottle + sample

W_3 = weight of empty density bottle +sample+ water.

W_4 = weight of density bottle + water.

The specific gravity values of the barite before and after the beneficiation process is shown in Table 3.3 as seen above. The raw barite head ore has a specific gravity value of 3.521. This value is low compared to the minimum industrially accepted value of 4.2. The



reason for this can be traced to the high level of impurities observed from the chemical analysis.

From table 3.3 the specific gravity of the barite after beneficiation process is 3.704, which is a slight increase from the value of the raw barite, but after treating the beneficiated barite with sulphuric acid, the specific gravity was now calculated to be 4.102. which is a massive increase from the value after beneficiation.

Conclusion

Jigging, Froth Flotation and acid/base treatment have been used to increase the concentration of Ba which is the major element in barite mineral. It is also clear that using only beneficiation process may not be very effective in removing as many impurities as is necessary to obtain an industrially acceptable specific gravity value. from the result discussed earlier 47.271 was the composition of Ba in the raw sample and after it was beneficiated through jigging and froth flotation it increased to 76.1639, however further treatment with acid the composition improved to 89.5034, and treatment with NaOH it also increased to 85.9736 but it is not up to the composition when treated with acid, hence it can be concluded that to improve barite concentration, jigging and froth flotation should be carried out and then treated with sulphuric acid. From the specific gravity results it can be concluded that there was an increase of the specific gravity after treatment with acid, the specific gravity increased from 3.704 after beneficiation to 4.102 after treatment with sulphuric acid. However, the specific gravity after treatment with NaOH was calculated to be 3.906, with this information barite ore composition can be improved as well as its specific gravity through jigging and froth flotation beneficiation processes and then treatment of the beneficiated barite with sulphuric acid.

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