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Beneficiation of Low-grade Barite ore from Dadin Kowa, Gombe State, Nigeria

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ABSTRACT

The challenge with using most Barite ores from Nigeria is usually their low specific gravity values as a result of impurities contained in them. This research investigates the beneficiation using froth floatation process of low grade Barite ore from Dadin Kowa in Gombe State, Nigeria. The concentration of the floatation chemicals was varied from 0 to 0.5 mol. X-ray fluorescence (XRF), microscopy, X-ray diffraction (XRD) and specific gravity (S.G) methods were used to analyse the constituents, morphology, phases and concentration of the barite respectively. The results show that 80% improvement of the barite ore concentration was achieved (53.6% to 78.6%) with 0.1 mol of frother and collectors. The morphology of the samples revealed decreased impurity and porosity after beneficiation. X-ray diffraction exposed the barite structure and shows that increasing concentration of reagents resulted in increased peaks intensity. More than 32% increase in specific gravity was observed in the barite ore which rose from 3.22 to 4.27 in the barite beneficiated with 0.5 mol of the chemicals. The results indicate that locally available barite with low concentration of barite can be beneficiated to levels where they can be used industrially.

1. INTRODUCTION

Barite which has the chemical formula $BaSO_4$, is a naturally occurring mineral often found in sedimentary rock as well as in other rock formations. It can be found in different parts of Nigeria especially in the Benue trough (Oden 2012; Ekwueme et al., 2013). Its chemical inertness, insolubility in many solvents makes it attractive for a lot of industrial applications. It is used for making paints, manufacturing plastics, glass, ceramics, and in drilling where more than 80% of the mineral is applied. It is more commonly used as a component of the fluids used in the drilling for oil and gas purposes because it helps to reduce high pressure formation, prevent possible blowouts and elongate the lifespan of the drilling bits. It is also noted for its non-interference with magnetic measurements during logging operations while drilling (Oahimire et al., 2021).

In its natural state, barite is usually associated with compounds like silicon oxide, iron oxide, magnesium oxide etc. (Naseem et al., 2011). These constituents act to lower the effective specific gravity values of most barite ores. In order for these barite ores to be acceptable for applications like drilling, paint making, etc., the American Petroleum Institute (API) has specified a minimum industrially acceptable standard for barite to have specific gravity values of 4.2 and above (Ene et al., 2012). The world's energy demand level has greatly increased in the last couple of years leading to higher demand for barite in drilling applications.

The suitability of many barite ores from Benue State has been investigated for their conformity to API specification using XRD and XRF based on field and laboratory studies. It was discovered that while some ores met the criteria for use in offshore drilling, others didn't (Oahimire et al., 2021). Barite ores from Taraba and Nasarawa States as well as a commercially available barite have also been investigated and characterized with the results showing that some of the barite ores met the API specification for use while others didn't and will need to be beneficiated (Otoijamun et al., 2021). Physical and chemical beneficiation techniques are usually employed to increase the concentration of $BaSO_4$ from their ores. Some of these methods include screening, log washing, jigging, heavy media separation, magnetic separation, froth flotation etc. The flotation technique is one of the most widely used methods because of its time-proven effectiveness not just for barite but for a lot of other minerals (Kecir and Botula 2015). The use of gravity preconcentration as well as flotation technology involving roughing, cleanings and scavenging steps have been used to enhance the recovery of low grade barite ores from China (Zhao et al., 2014). A comparison of the beneficiation effectiveness using froth flotation and gravity concentration for low to medium grade barite ores from Baluchistan, Pakistan showed that the former gave better concentration results than the later (Bhatti et al., 2017). Leaching was therefore introduced to further improve the concentration process to attain industrial grade levels.

The effect of using sodium petroleum sulfonate (SPS) and sodium hexametaphosphate (SHMP) as collectors in the separation using flotation method of fluorite from barite ore has been investigated. In the pH range 7-11, not much separation was observed using SPS but the concentration was good even at temperatures as low as 5^oC. SHMP was able to depress the fluorite more than barite at pH of 11 (Chen et al., 2018). The dry/wet high gradient magnetic separation (HGMS) and bleaching methods has been used to investigate the rejection rate of colouring impurities from barite ore in paint making. The wet process was found to give better results than the dry process The acids, H_2SO_4 and HCl improved the quality of barite but did not meet the requirement (H_2SO_4) but 15% HCl was found to be more effective (Deniz and Guler 2018). Barite ore from El Cuervo Butte, New Mexico, USA were beneficiated using flotation and gravity concentration and the results show that flotation gave a higher concentration than gravity concentration (Harris 1988). Repeated flotation experiments are required to achieve high grade barite. The behaviour of barite was analysed using flotation with the effect of different process parameters in order to optimise them (Kecir and Botula 2015). A comparison of the barite concentration from a conventional cell and in a column shows that the latter leads to better concentration results.

Barite ore obtained from Azara area in Nasarawa State have been beneficiated through the process of leaching with HCl and HOCl, jigging, and froth flotation, and yielded concentrated barite ore of specific gravity ranging from 3.207 to 4.38 (Mgbemere et al., 2018). Modelling has been used to upgrade barite ore using direct and indirect flotation methods and magnetic separation. In view of direct and reverse flotation, it has been noted that flotation in comparison with wet high intensity magnetic separation (WHIMS) has delivered higher quality for products with specific gravity in the range of 4.35 and 4.44 (Molaei et al., 2018a). In addition, other barite ore beneficiation techniques like gravity separation (jigging) and froth flotation were used to beneficiate barite ore and the results show that flotation gave comparably better results than jigging (95 versus 90%, respectively) (Molaei et al., 2018b). Taguchi method was used to optimise the parameters for the beneficiation. Lu et al., (2020) investigated the effect of lauryl phosphate as a promising collector for the froth flotation process and the following parameters; micro-flotation, contact angle, zeta potential and molecular dynamics simulation were used to show that it has superior flotation efficiency over a range of pH. This collector has been used for materials like calcite, magnesite etc. In this research, low grade barite ore from Dadin Kowa in Gombe State has been beneficiated using magnetic separation and froth flotation. The objective is to improve the concentration of the ore to ensure that it meets the industrially accepted standard.

2. MATERIALS AND METHODS

2.1. Sample Collection and Beneficiation

The barite ore investigated in this study was sourced from Dadin Kowa area of Gombe State, Nigeria. The reagents used for the experiment include sodium silicate, pine oil as frother, oleic acid as collector and distilled water. The equipment include a gyratory crusher, ball milling machine (wet ball mill with ceramic balls), magnetic separator, flotation cell, pipette, beaker, spoon, pH meter, and digital weighing balance.

Figure 1: The picture of the barite ore from Dadin Kowa in Gombe State

The barite ore was initially crushed with a gyratory crusher and was further reduced in size using a ball mill (Metso 2006). A total of 6 kg of barite ore was introduced to the crusher after which 5.4 kg was recovered. The powdered sample was ball-milled for 2 h to reduce the particle size of the barite to less than 300 µm. Magnetic separation was carried out by feeding the barite ore to a magnetic separator. The machine hopper is opened to discharge at a mass flow rate of 1.5 kg/min, and then particles are collected at the various magnetic buckets while the iron-free particles are taken to the overflow end. The slow flow rate was to enable the machine to completely collect iron-containing materials. Magnets of various capacities were placed at different locations to divert the iron-containing materials into different containers. The amount of barite ore that was fed to the separator is 5.4 kg and at the end of the process, approx. 4.8 kg were recovered as over belt concentrate. The quantity of magnetite, hematite and paramagnetic particles collected were 80.9 g, 57.8 g and 102.9 g respectively.

The ground barite powder was transferred to a ball mill while adding 100 ml of distilled water and 1.6 g of Na_2SiO_3. The milling process was allowed to run for 2 h. The milled sample was washed with distilled water and transferred to the froth flotation cell and agitated for 5 min. The froth flotation process was carried out with 200 g of the concentrate for each of the experiments. The pH of the system was calibrated using a pH meter while agitating the solution for between 8-10 min. 0.1 ml each of the frother (pine oil) and collector (oleic acid) were added to the solution while the agitation was ongoing for 10 min. This process was repeated using 0.3 and 0.5 mol each of both the collector and frother respectively. The air nozzles were opened to float the concentrate into a container. When the process is completed, water was decanted from both the overflow (concentrate) and underflow (gangue) and dried.

2.2. Sample Characterisation

The barite ores' elemental composition was examined before and after beneficiation using an X-ray fluorescence analyser (ARL Quantx EDXRF 9952120). Since Barium is a heavy element, it was carried out in air atmosphere. To achieve this analysis, 2 g of the finely ground barites were weighed, poured into a polypropylene sample holder, and covered with cotton wool. The specific gravity values of the barite samples were calculated using Archimedes method. A pycnometer bottle was placed in a weighing balance and 10 g of the barite sample was measured with a weighing balance and poured into the pycnometer bottle. Distilled water was used to fill three-quarters of the pycnometer bottle and the formula used to calculate the specific gravity is given by

$$Gs = \frac{Wo}{Wo+(WA+WB)} \quad (1)$$

Where Gs is specific gravity, W_o = weight of an oven dry barite sample, W_A = weight of pycnometer filled with water, W_B = weight of pycnometer filled with water, and barite.

The X-ray diffraction experiment was conducted to determine the existing phases in the barite samples with the aid of an X-ray diffractometer (Bruker D8 Discover, Karlsruhe, Germany) having a Cu$K\alpha$ anode of λ = 1.5406 Å and was operated at voltage, and current of 40 kV, and 40 mA respectively. Grazing incidence diffraction mode with a source fixed at 5^{o}C, scanning speed of 3 s, and increment at 0.01^o intervals, and measurement range of 10 to 60^o are the parameters used. A crystallographic database was used to match the peaks obtained with the reference materials that exist in the literature. The morphology of the barite ore as well as the beneficiated samples were examined using a light microscope (Olympus BX 51, Hamburg, Germany. The frothed barite sample was placed on the glass sample holder and placed under the microscope. Images were acquired with the aid of the analysis software at different magnifications.

3. RESULTS AND DISCUSSION

The result of the qualitative analysis for the barite ore using X-ray fluorescence analyser is shown in Figure 2. The spectrum for the barite ore shows that the peaks of barium are the dominant ones with a few minor peaks attributed to elements like Fe and Sr. The XRF analysis was also carried out for the beneficiated barite samples and the quantitative chemical composition of all the barite samples is shown in Table 1. The chemical composition of the barite samples shows that in addition to the major constituents which are BaO and SO_3, the other compounds with significant amounts in the beneficiated barite are SiO_2 and TiO_2. On floating the barite samples with 0.1 mol, 0.3 mol and 0.5 mol each of pine oil and oleic acid, the concentration of the BaO and SO_3 increased slightly. The reagents are effective at improving the concentration of the desired compounds but are not very effective at removing SiO_2. When the concentration of barite ore used in this research is compared to other barite ore deposits in the country, it can be categorized as a low-grade (Abraham et al., 2021; Oahimire et al., 2021; Otoijamun et al., 2021). The beneficiated barite samples however indicate that the concentration obtained using similar techniques are not far off from literature reports (Ibrahim et al., 2017; Mgbemere et al., 2019).

The specific gravity values of the samples are shown in Figure 3. For the barite ore, the S.G value was calculated to be 3.2. The location and depth of the barite ore has been shown to significantly affect the concentration of the ore (Abraham et al., 2021). When 0.1 mol each of both pine oil and oleic acid were used for the floatation process, the S.G. value increased to 3.66. On increasing the concentration of the reagents to 0.3 mol, the S.G value increased to 3.82. Finally, when the quantity was increased to 0.5 mol, the S.G value increased to 4.27. The increase in the specific gravity values with increasing concentration of both the collector and frother is related to their ability to separate the impurities from the $BaSO_4$ which is the basis of froth flotation. The use of acids like HCl and other chemicals have also been reported to result in an improvement in the concentration and therefore the specific gravity value of barite (Khan and Khan 2002, Mgbemere et al., 2018). Increasing the concentration of HCl also increases the concentration of the obtained $BaSO_4$ (Khan et al., 2003).

The diffraction patterns of the barite ore and barite samples floated with different amounts of the flotation reagents are shown in Figure 4. The peak fitting of the barite ore was determined with the POW_COD database provided by Qualx 2 software (Altomare et al., 2015). The observed diffraction pattern for barite ore was compared with the reference material 00-901-4889 with composition $Ba_{0.986}Ca_{0.002}Mg_{0.002}O_4SSi_{0.007}$ and it matches very well. The peaks at 38^o, 52^o, 53^o and 58^o are not associated with barite and so could not be matched. The albite phase material matches well with the pattern which indicates the presence of $AlNaO_8Si_3$. A report in the literature showed that the diffraction pattern of the barite ore contains in addition to the barite phase, other structures like quartz, illite etc. (Demeekul et al., 2016).

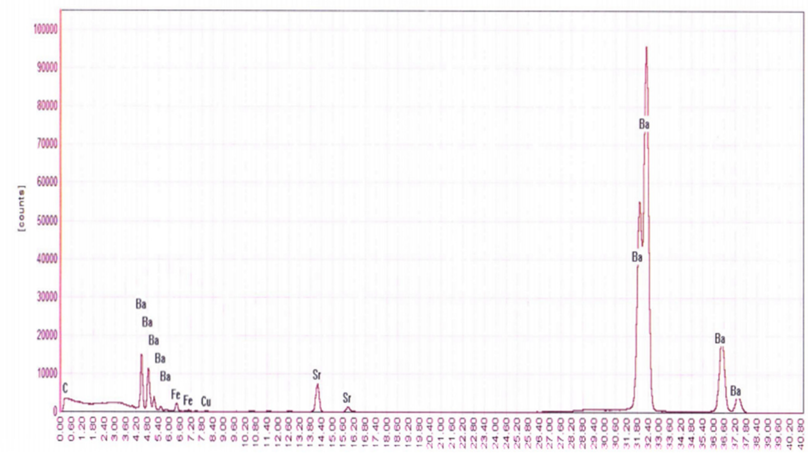

Figure 2: X-ray fluorescence analysis of the barite ore before the beneficiation process

Table 1: X-ray fluorescence analysis of the crude and froth-floated barite samples

Compound	Fe_2O_3 (wt.%)	SiO_2 (wt.%)	Al_2O_3 (wt. %)	SO_3 (wt.%)	TiO_2 (wt.%)	K_2O (wt.%)	CuO (wt.%)	BaO (wt.%)
Barite ore	1.15	7.398	0.736	15.71	8.466	0.138	0.106	37.926
Floated barite (0.1 ml)	0.21	7.855	1.053	19.245	10.59	0.071	0.084	59.346
Floated barite (0.3 ml)	0.21	6.443	0.968	20.904	11.1	0.068	0.082	49.229
Floated barite (0.5 ml)	0.21	7.801	1.095	18.767	10.006	0.119	0.078	51.922

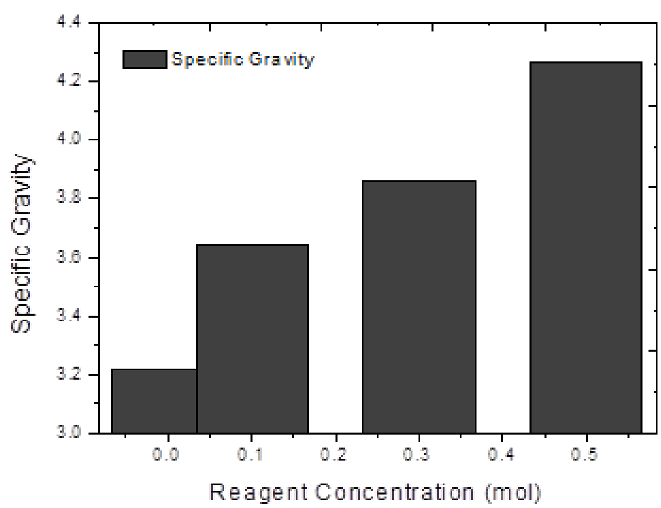

Figure 3: Specific gravity values of barite ores and floated barite samples (concentration of zero implies that the barite is in its natural state)

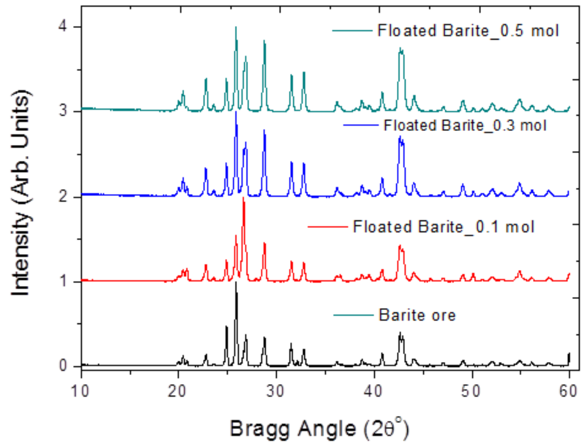

Figure 4: X-ray diffraction patterns of barite ore and floated samples with different concentration of chemicals

Compared to the Bragg angle of barite from the literature, some of the Bragg angles are either exactly at the expected positions or are slightly shifted from their positions (Bhatti et al., 2017). The intensity of the peaks was also close to the expected intensity although there are intensities that are either too high or low. As the reagents are introduced during the flotation process, the intensity of the diffraction patterns became sharper while the shape and position of the peaks slightly shifted to higher angles. The higher the amount of chemicals used in the flotation experiment, the higher the peak intensities. This implies that the level of impurities in the floated samples gradually lowered (Chen et al., 2018). This observation is in contrast to the diffraction pattern obtained when HCl and HOCl were used to improve the concentration of Barite (Mgbemere et al., 2018). In that case, it is believed that chemical reactions occurred leading to the formation of new phases.

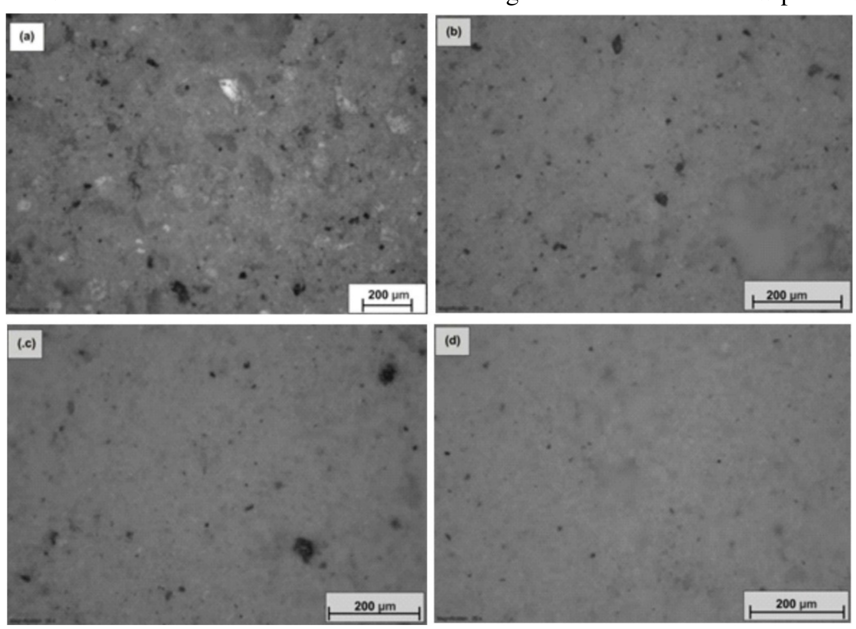

Figure 5: Light microscope images of (a) crude barite from Dadin Kowa (b) barite floated with 0.1 m of chemicals (c) barite floated with 0.3 m of chemicals and (d) barite floated with 0.5 m of chemicals

The light microscope images of the samples are shown in Figure 5. For the barite ore, three distinct morphologies corresponding to different constituents are observed on the surface of the micrographs. It is believed that the bright coloured shapes represent some of the impurities in the barite while the dark spots are basically the pores in the sample. The rest represent the $BaSO_4$ mineral. A similar microstructure has been observed for barite ore using light microscope where the bright and dark coloured shapes represent the quartz and barite respectively (Zhao et al., 2014). When the barite is floated with 0.1 mol of the reagents, what is observed are the dark pores and barite as well as the impurities which could not be removed through the flotation process. As the amount of reagent used in the froth flotation process increases, the amount of porosity decreased while uniformity in the microstructure improves. When the sample is floated with 0.5 mol of the reagents, the microstructure became more homogenous and the level of porosity decreases corresponding to the increased specific gravity value as shown in Figure 4.

4. CONCLUSION

This research has implemented froth flotation technique for the beneficiation of a low-grade barite ore sourced from Dadin-Kowa area in Gombe State, Nigeria. The initial chemical composition analysis shows that the concentration of the starting ore is low in $BaSO_4$ with a specific gravity value of 3.2. Magnetic separation on the barite ore helped to remove iron bearing particles thereby instantly improving the concentration. When froth flotation method was applied to the ore, the specific gravity values increased to 3.66, 3.82 and 4.27 for barite ores with frother and collector amounts of 0.1 mol, 0.3 mol and 0.5 mol respectively. The X-ray diffraction results showed that froth floatation helps to increase the intensity as the concentration of the floatation chemicals increases. The concentration of the $BaSO_4$ increased although the amount of SiO_2, and other impurities were still relatively high. The morphology of the samples indicates that the impurity level decreased with increasing concentration of the frothing chemicals.

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6. CONFLICT OF INTEREST

There is no conflict of interest associated with this work.

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