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# Beneficiation Study on Barite Ore of Duddar Area, District Lasbela, Balochistan Province, Pakistan

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**Abstract.** This study highlights the results of processing an indigenous low to medium grade barite ore of Duddar area, district Lasbela, Balochistan, Pakistan. The ore was characterized by x-ray diffraction technique. The gravity concentration and forth flotation technologies were employed to beneficiate the ore in order to achieve commercial grade barite concentrate with economical recovery. The results showed that flotation was the better method than gravity concentration to concentrate the barite mineral. A process flow sheet was designed in the light of these experiments. The flotation tests revealed that barite concentrate assaying 95.85% $BaSO_4$ could be obtained with recovery of 82.06% from an ore containing 76.04% $BaSO_4$. The flotation concentrate was leached to get rid of objectionable impurities. The final leached barite concentrate possesses 98.86% $BaSO_4$ content and conforms to the specifications of industrial grade barite concentrate.

**Keywords:** Duddar area, barite, beneficiation, tabling, flotation, leaching, recovery

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## Introduction

Barite ($BaSO_4$) also known as heavy spar is an exceptional nonmetallic mineral with an incredible high specific gravity of 4.5 which makes it suitable for a wide range of industrial applications. Barite is mainly used as weighting agent in oil-well and natural gas drilling mud to increase mud density up to 2.5 g/cm$^3$. It is estimated that more than 80% of the barite produced in the world is used only for drilling purpose (Scott *et al*., 2010). Barite is chemically inert and insoluble hence finds its use as additive in the manufacture of rubber, paints, enamels, plastics, paper goods, wallpapers, asbestos goods, leaded glass and ceramics. Moreover, it is used in radiology for x-rays of the intestines and to make high-density concrete resistant to nuclear radiation. A more recent application of barite is in the formulation of brake shoe linings, noise reduction in engine compartments and spark-plug alloys (Singh *et al*., 2007; Mills, 2006).

Barite is the principal mineral of barium. It is usually milky white but may have shade of other colours such as tinted yellow, brown, grey, red green and blue depending on the impurities trapped during the formation of crystals. It forms thin to thick tabular crystals. It usually occurs in sedimentary rocks especially disseminated in limestone, dolostone and sandstone as veins, lenses, fillings, concretions and replacements. It is also a common mineral in hydrothermal veins where it is a gangue mineral associated with various sulphide minerals of lead, zinc, silver, copper, iron, antimony, cobalt and nickel. In a few locations, barite is deposited as a sinter in hot springs. Barite commonly occurs with accessory minerals such as calcite, dolomite, siderite, aragonite, fluorite, gypsum, anhydrite, chert, quartz, apatite and metal sulphides (Naseem *et al*., 2011; Blackburn, 1988).

The total world's reserves of barite in all categories are about 2 billion tonnes, but only about 740 million tonnes resources are identified. China, India, USA, Morocco, Mexico, Kazakhstan, Iran and Turkey are the leading producers of barite (Table 1A). China alone shares more than 50% of the world's barite production. Pakistan exports limited quantity of high grade ore (Table 1B) ranging from 42 to 118 thousand metric tonnes (Mcrae, 2015; Miller, 2011). Pakistan possesses sizeable deposits of barite ore widely distributed in Balochistan and Khyber Pukhtunkhwa Provinces. Significant deposits are located at Monar Talar, Gunga, Surmai, Sekran, Malkhor and Ranj Laki (district Kuzdar), Duddar, Bankhri, Kundi-Dharn, Dham Jhal, Mithi-Arrara, Kanrachzi, Hunurki, (district Lasbela) in Balochistan and at Kohala, (district Abbotabad), Kacchi, Faqir Mohammad, Tipra (district Haripur), Besham (District Kohistan), Gera Banda

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(district Swat) in Khyber Pukhtunkhwa. These are medium to high grade ore deposits having $BaSO_4$ content ranging from 20 to 92%. The estimated reserves of barite in Pakistan are more than 30 million tonnes (Zaigham and Mallick, 2000; Ahsan and Quraishi, 1997; Ahmad and Siddiqi, 1992).

Among these, Duddar barite ore deposit is important which is located on the eastern flank of the Mor Mountain Range at latitude of 26°05'N and longitude of 66°50'E. The Duddar mineralization occurs in two separate exposures known as Duddar North and Duddar South. These exposures are 258 and 274 meters long with an average thickness of 10 meters. The mineralization is stratiform, hosted by interbedded siltstone, mudstone and shale sequence of Anjira formation (Naseem *et al*., 2011; Zaigham and Mallick, 2000).

During mining, high grade variety of barite having specific gravity ranging from 4.1 to 4.35 is taken separately for direct use after grinding. But low grade ore with specific gravity below 3.9 is rejected. These low grade ores contain various gangue minerals which need to be removed in order to obtain a product suitable for industrial applications. There are mainly two processes that are commercially used to beneficiate low grade barite ores. Gravity concentration techniques like jigging, tabling, spirals, and dense media separation are generally used for coarse grained ores. Direct or reverse flotation technology is applied on finely disseminated ores to separate the barite mineral from the associated gangue components. A large number of barite producers utilize only flotation to recover and improve the grade of barite (Raju *et al*., 2004; Day, 2002; Davis, 1985).

**Table 1A.** Barite production (thousand metric tonnes) country wise for the last 5 years

| Country | Year 2010 | Year 2011 | Year 2012 | Year 2013 | Year 2014 |
|---|---|---|---|---|---|
| Algeria | 60 | 40 | 60 | - | - |
| China | 3,600 | 4,100 | 4,000 | 4,000 | 4,100 |
| Germany | 75 | 70 | 70 | - | - |
| India | 1,000 | 1,350 | 1,400 | 1,740 | 1,600 |
| Iran | 250 | 350 | 350 | 270 | 270 |
| Kazakhstan | 100 | 200 | 200 | 250 | 250 |
| Mexico | 140 | 157 | 160 | 344 | 400 |
| Morocco | 460 | 600 | 650 | 1,000 | 1,000 |
| Pakistan | 45 | 58 | 60 | 118 | 75 |
| Peru | - | 87 | 90 | - | - |
| Russia | 65 | 62 | 60 | - | - |
| Turkey | 150 | 230 | 250 | 250 | 270 |
| United Kingdom | 50 | 50 | 50 | - | - |
| Vietnam | 90 | 85 | 85 | - | - |
| United States | 670 | 710 | 654 | 700 | 720 |
| Other Countries | 160 | 220 | 220 | 558 | 575 |
| Total | 6,900 | 8,370 | 8,400 | 9,230 | 9,260 |

Mcrae (2015).

The effects of important variables of flotation process on grade and recovery of barite concentrate were investigated. The rougher concentrate obtained was subjected to regrinding for further liberation of locked particles and two cleaning operations were employed in close circuit to achieve higher grade concentrate.

## Materials and Methods

**Sample preparation.** A representative sample of barite ore of Duddar area, weighing about 100 kg packed in five cloth bags was supplied by Pakistan Mineral Development Corporation (PMDC), Karachi, for beneficiation test work. The whole of the ore sample included drill core pieces cut into quadrants. Processing experiments were carried out on laboratory scale in Mineral Processing Research Centre (MPRC) PCSIR Complex, Lahore, Pakistan. The ore was mixed and crushed in laboratory jaw crusher where opening was set at 12 mm. The jaw product having size less than 12 mm was further crushed in secondary rolls crusher to obtain a product size of finer than 8 mm. The roll product was thoroughly mixed and a head sample for chemical and XRD analysis was prepared by using a sample splitter. It was ground to minus 200 mesh size with the help of disc pulverizer. The remaining crushed composite ore sample was riffled to make smaller samples weighing one kg each for beneficiation studies.

**Table 1B.** Barite production (thousand metric tonnes) in Pakistan and World for the last 10 years

| Barite production | 2005 | 2006 | 2007 | 2008 | 2009 | 2010 | 2011 | 2012 | 2013 | 2014 |
|---|---|---|---|---|---|---|---|---|---|---|
| Production by Pakistan | 42 | 45 | 44 | 43 | 42 | 45 | 58 | 60 | 118 | 75 |
| Total world production | 7,870 | 7,960 | 7,730 | 8,200 | 6,130 | 6,900 | 8,370 | 8,400 | 9,230 | 9,260 |

Mcrae (2015); Miller (2011).

**Chemical analysis.** The head sample was analyzed for $BaSO_4$ and the associated gangue components. The quantitative chemical analysis of the head sample was done by gravimetric, volumetric and instrumental analytical techniques. The finely ground ore sample was fused with AR grade sodium carbonate in platinum crucible at 900 °C in muffle furnace. The fused mass was leached in hot water and filtered. The volume of filtrate was made up to the mark with distilled water and this was named as 'Solution A', and used for the determination of soluble components i.e., silica, alumina, zinc and sulphur. The residue was dissolved in dilute hydrochloric acid and diluted with water to make the volume up to the mark. This was named as 'Solution B' and utilized for the determination of barium, calcium, magnesium, iron and lead. Barium, sulphur and silica were estimated by gravimetric methods (Jeffery, 1989; ASTM: E 247-01, 2007). Iron was found by oxidation reduction titration using standard solution of potassium dichromate (ASTM: E 246-05, 2007), while aluminum, calcium and magnesium were determined by complexometric titration using standard solution of EDTA (ASTM: E 738-05, 2007; Jeffery, 1989). The minor elements were determined by Atomic Absorption Spectrophotometer (Model: 8000, Hitachi, Japan). Another 1 g of sample was heated in electric oven for 1 h at 110 °C to find out moisture. The same sample was ignited in electric furnace at 950 °C for 1 h to determine the loss on ignition. The chemical analysis of the representative sample is given in Table 2. Barium sulphate content of processed products was also determined by the same method. The chemical analysis of flotation concentrate and final leached barite concentrate was carried out by same techniques as applied for head sample of ore.

**Table 2.** Chemical analysis of the composite sample of Duddar barite ore

| Constituents | Percentage |
|---|---|
| Barium sulphate ($BaSO_4$) | 76.04 |
| Silica ($SiO_2$) | 10.77 |
| Iron oxide ($Fe_2O_3$) | 4.85 |
| Aluminum oxide ($Al_2O_3$) | 2.31 |
| Calcium oxide (CaO) | 1.65 |
| Magnesium oxide (MgO) | 0.88 |
| Zinc (Zn) | 0.08 |
| Lead (Pb) | 0.06 |
| Sulphur (S) other than sulphate | 2.33 |
| Loss on ignition (LOI) other than sulphur | 0.95 |

**Mineralogy.** A qualitative scan was performed by x-ray diffractometer (Model: D-5000, Siemens, Germany) in order to find out different mineral phases present in the composite ore sample. X-ray diffraction was performed @ 0.02 steps/sec. The scan angle was ranged from 5°-80° to get all the important peaks. X-ray diffractogram of the ore is illustrated in Fig. 1, while 2 θ, d values and relative intensities of various peaks are given in Table 3. The diffractogram obtained was matched with standard data provided with x-ray diffractometer to identify the minerals by search/match procedure. The major and minor minerals found in the ore are given in Table 3.

**Gravity concentration tests.** Wilfley shaking table (Mine & Smelter Supply Company, Serial No.13, USA) was used for gravity concentration tests. It has a flat deck (55 cm × 110 cm) having cleats on it which form riffles. It moves with a horizontal to and fro motion of 300 cycles per minute. Each cycle consists of a slow

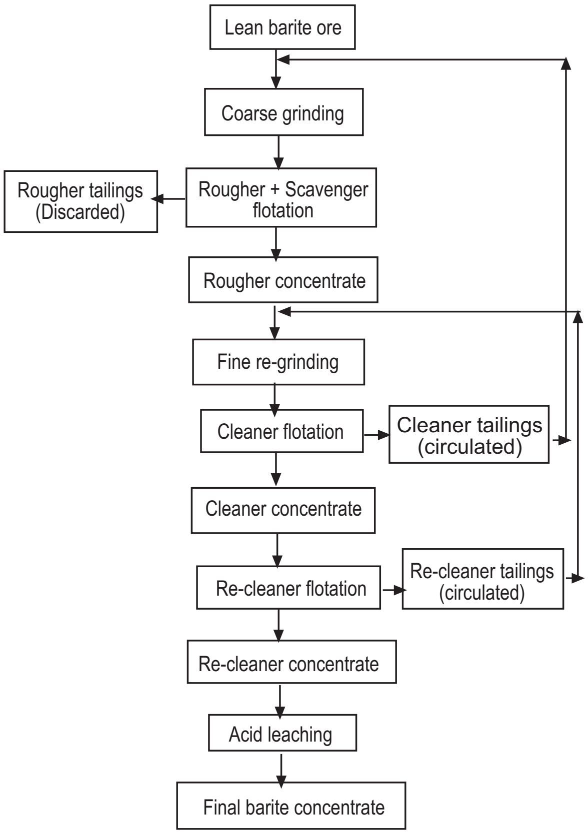


**Fig. 1.** Flow-sheet for the beneficiation medium grade barite ore of Duddar (Lasbela) Balochistan, Pakistan.

**Table 3.** Minerals identified in Duddar barite ore

| Seq. | 2 θ | d Value | Rel. I. | Minerals identified |
|---|---|---|---|---|
| 1 | 20.31 | 4.37 | 9.26 | gibbsite |
| 2 | 20.74 | 4.28 | 19.68 | quartz/gypsum |
| 3 | 21.07 | 4.21 | 6.93 | - |
| 4 | 23.11 | 3.85 | 29.67 | calcite |
| 5 | 25.19 | 3.50 | 86.15 | anhydrite |
| 6 | 26.17 | 3.45 | 100.00 | barite |
| 7 | 27.15 | 3.33 | 85.85 | quartz/barite |
| 8 | 29.05 | 3.09 | 55.81 | barite/galena/sphalerite |
| 9 | 29.32 | 3.03 | 10.42 | calcite |
| 10 | 31.86 | 2.80 | 35.27 | siderite/dolomite/anhydrite/gypsum |
| 11 | 33.322 | 2.68 | 25.99 | pyrrotite/sphalerite/galena/gypsum/pyrite |
| 12 | 36.48 | 2.46 | 9.67 | quartz/gibbsite/pyrite |
| 13 | 38.91 | 2.33 | 6.74 | anhydrite |
| 14 | 41.13 | 2.19 | 13.09 | dolomite |
| 15 | 42.94 | 2.11 | 60.54 | barite |
| 16 | 43.20 | 2.09 | 51.45 | barite |
| 17 | 43.46 | 2.08 | 18.52 | pyrrotite |
| 18 | 44.37 | 2.04 | 10.66 | - |
| 19 | 49.38 | 1.84 | 12.01 | calcite/quartz/sphalerite |
| 20 | 51.27 | 1.78 | 6.70 | dolomite |
| 21 | 52.33 | 1.74 | 9.87 | siderite/galena |
| 22 | 53.18 | 1.72 | 5.58 | pyrrotite |
| 23 | 55.19 | 1.66 | 11.77 | sphalerite |
| 24 | 56.33 | 1.63 | 3.88 | pyrite/sphalerite/galena |
| 25 | 60.85 | 1.52 | 9.72 | quartz/sphalerite |
| 26 | 66.86 | 1.38 | 4.80 | quartz |

forward and rapid return motion along with slurry control system for stratification of materials. A few preliminary experiments were performed at feed rate of 15 kg/h, wash water flow rate of 10 L/min, deck inclination angle of 7° and stroke length of 8 mm. The results of gravity separation tests are given in Table 4.

**Flotation tests.** The froth flotation tests were conducted using laboratory flotation machine (Model: D-12, Denver, USA). The feed for the flotation tests was prepared by wet grinding of barite ore in a laboratory rod mill (Denver, USA size: 7”×14”) with the addition of water in the mill to maintain the solid to liquid ratio of 1:1. The ground ore was transferred to stainless steel flotation cell of 4 L capacity. A number of batch type flotation tests were carried out at different sets of conditions so as to investigate the effects of important variables such as (a) grind size of the ore (b) pulp density (c) pH of the pulp (d) impeller speed (e) dosage of collector (f) amount of frother (g) quantity of regulator (h) conditioning time and (i) flotation time on the grade and recovery of the product. In all tests a scavenging flotation was conducted and scavenger concentrate was mixed along with rougher concentrate. After having optimizing rougher flotation recovery, the test work was done to produce required grade concentrate. The rougher concentrate was re-ground for further liberation and one cleaning flotation with addition of flotation reagents and another re-cleaning flotation without addition of flotation reagents was carried out. The concentrate produced was heated in oven at 250 °C to get rid of flotation reagents. The optimum parameters of flotation tests are reported in Table 5 while the metallurgical balance of typical test is given in Table 6. The complete chemical analysis of final barite concentrate produced by flotation is shown in Table 7.

**Table 4.** Metallurgical balance of a typical gravity concentration test of Duddar barite ore

| Tabling products | Weight (%) | Grade (%) ($BaSO_4$) | Recovery (%) ($BaSO_4$) |
|---|---|---|---|
| Concentrate | 54.26 | 84.07 | 60.08 |
| Middling | 17.50 | 78.60 | 18.10 |
| Tailing | 28.24 | 58.88 | 21.87 |
| Head | 100.00 | 76.06 | 100.00 |

**Table 5.** Flotation parameters and optimum conditions of Duddar barite ore processing

| Flotation parameters | Optimum conditions | | |
|---|---|---|---|
| | Roughing | Cleaning | Re-cleaning |
| Grind size (feed size) of ore | 100% -75 µm | 100% -65 µm | 100% -65 µm |
| Pulp density (% solids) | 25% Solids | 15% Solids | 10% Solids |
| pH of pulp (slurry) | ~10.0 | ~10.0 | ~10.0 |
| Agitation speed (aeration) | 1100 rpm | 1000 rpm | 900 rpm |
| Collector (sodium oleate) | 600 g/t | 300 g/t | Nil |
| Frother (polypropylene glycol) | 60 g/t | 30 g/t | Nil |
| Gangue depressant (sodium silicate) | 400 g/t | 200 g/t | Nil |
| Galena depressant (sodium chromate) | Nil | 20 g/t | Nil |
| Conditioning time | 12 min | 12 min | 12 min |
| Flotation time | 20 min | 20 min | 20 min |

**Table 6.** Metallurgical balance of a typical flotation test of Duddar barite ore

| Flotation products | Weight (%) | Grade (%) ($BaSO_4$) | Distribution (%) ($BaSO_4$) |
|---|---|---|---|
| Re-cleaner concentrate | 65.11 | 95.83 | 82.06 |
| Re-cleaner tailings | 5.10 | 58.89 | 3.95 |
| Cleaner concentrate | 70.21 | 93.15 | 86.01 |
| Cleaner tailings | 8.98 | 53.69 | 6.34 |
| Rougher concentrate | 79.19 | 88.68 | 92.35 |
| Rougher tailings | 20.81 | 27.95 | 7.65 |
| Head sample | 100.00 | 76.04 | 100.00 |

**Leaching tests.** Acid leaching tests were performed on final flotation concentrate using different concentrations of commercially available hydrochloric acid (37%). About 100 g portions of concentrate were taken in 4 different Pyrex glass beakers (500 mL). These were treated with 100 mL diluted solution of 5, 10, 15 and 20% hydrochloric acid, respectively, with constant stirring on hot plate at 95 °C to boiling temperature for 1 h. These were cooled, filtered and washed with distilled water until free of acid. The residues left on filter paper were dried for 1 h in electric oven at 110 °C and evaluated for comparison of $BaSO_4$ content. The results obtained are shown in Table 8. The flow sheet developed for the overall process is shown in Fig. 1.

## Results and Discussion

Chemical analysis of head sample (Table 2) indicates that $BaSO_4$ content in the ore is 76.04%. Main undesirable gangue impurity is silica which is 10.77%. The other relatively a lower impurity is iron oxide, whose concentration is 4.85% whereas, alumina and calcium oxide are found to be 2.31% and 1.65%, respectively. Other minor impurities include magnesium oxide which is less than 1%, heavy metal such as lead is 0.08% and zinc is 0.06%. It is obvious from the result that ore is medium grade and is not suitable for final application until beneficiated. In order to produce a commercial grade barite concentrate assaying more than 90% $BaSO_4$ with specific gravity around 4.2, some kind of beneficiation work is required for further up-gradation of this ore.

Mineralogy prior to the selection of beneficiation process is crucial parameter. It provides information about valuable and valueless minerals present in the ore deposit which in turn help to decide the process selection. Mineralogical characterization of the ore was carried out by x-ray diffraction technique. The x-ray diffraction (Fig. 2) of the head sample confirms that ore predominantly consisted of barite along with subordinate amount of quartz, anhydrite and gypsum minerals as major peaks of XRD correspond to standard values of these minerals. It was identified by JCP.CAT search and match programme of the x-ray diffractometer that the minor peaks corresponds to calcite, aragonite, dolomite, siderite, gibbsite, galena, sphalerite, pyrrotite and pyrite minerals (Table 3).

Many researchers attempted different mineral processing techniques and their combination in order to achieve the desired goal from wide variety of barite ore deposits.

**Table 7.** Chemical analysis of final Duddar barite concentrate and requirement of industries

| Constituents | Flotation concentrate | Leached concentrate | Requirements of different industries |
|---|---|---|---|
| Barite ($BaSO_4$) | 95.85% | 98.86% | Min. 92.0% for drilling mud, 95.0% for all other |
| Silica ($SiO_2$) | 1.30% | 1.02% | Max. 2.50% for drilling mud, 1.50% for glass |
| Iron oxide ($Fe_2O_3$) | 0.50% | 0.02% | Max. 0.15% for glass, 1.0% for chemicals, 0.05% for paint |
| Aluminum oxide ($Al_2O_3$) | 0.34% | 0.025% | Max. 0.03% for drilling mud, 0.15% for glass |
| Calcium oxide (CaO) | 0.94% | 0.006% | Max 0.01% for drilling mud |
| Magnesium oxide (MgO) | 0.36% | 0.001% | Max 0.01% for drilling mud |
| Total heavy metals (Pb) | 0.01% | Nil | Max 0.001% |
| Moisture content | Nil | Nil | Max 0.1% |
| Loss on ignition (LOI) | 0.11% | 0.07% | Max 0.5% |
| Colour | off white | Beige white | Beige white |
| Specific gravity | 4.28 g/cm$^3$ | 4.35 g/cm$^3$ | Min. 4.2 g/cm$^3$ for mud |
| Particle size | 100.0% < 63 µm | 100.0% < 63 µm | Max. 3.0% > 75 µm for drilling mud, Vary for others |
| Flotation chemicals | Nil | Nil | Nil for drilling mud |

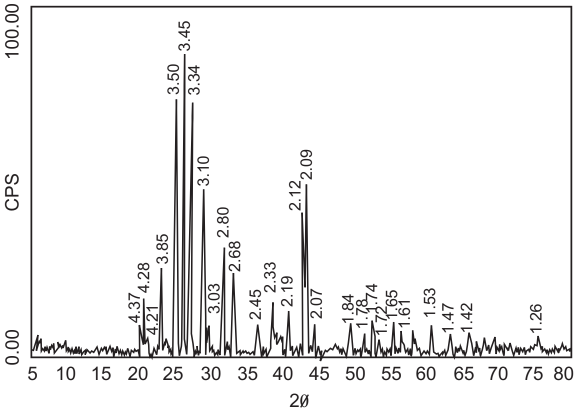


**Fig. 2.** XRD of Duddar barite ore representing the peaks of various minerals.

Ciccu *et al*. (1987) applied gravity concentration using jigs and flotation method with Na-Cetylsulphate to upgrade barite ore collected from Barega mine in Sardinia, Italy into a concentrate assaying 94-95% $BaSO_4$. Nwoko and Onyemaobi (1997) used gravity concentration using Wilfley Mineral Jig on Nigerian barite ore possessing 87% $BaSO_4$ to obtain a barite concentrate having 98% $BaSO_4$ for industrial use with 95% recovery. Hadjiev *et al*. (2000) applied magnetic separation to separate iron minerals and then froth flotation on tailings using OMC 199 and AERO 845 flotation reagents in ratio of 2:1 to obtain a barite concentrate ranging from 87% to 97% but with relatively low recovery of around 53% from Kremikovtzi ore-deposit, Bulgaria containing 32% $BaSO_4$. Khan *et al*. (2003) applied leaching process using 25% HCl to leach out the impurities from Gunga barite ore, District Khuzdar, Balochistan containing 92% $BaSO_4$ and produced a concentrate assaying 95% $BaSO_4$. Chemical processing could not minimize silica content and only minimized the iron, calcium and magnesium content. Moreover, it is more expensive as compared to physical processing when applied on low to medium grade ores due to more consumption of chemicals. Raju *et al*. (2004) applied direct flotation using oleic acid (anionic collector) as well as reverse flotation in the presence of Armoflote (cationic collector) and could produce barite concentrate of 96% purity from Mangampet barite deposit of Andhra Pradesh, the largest deposit in the world. Kumar *et al*. (2005) applied cationic flotation on Indian barite ore using Armoflote collector followed by dilute sulphuric acid leaching of 96% flotation concentrate and prepared a barite concentrate of 98% purity with 80% recovery. Udenko *et al*. (2011) upgraded a barite ore of Azara, Nasarawa State, Nigeria, from 75.40 to 91.90% content with recovery of 86% through flotation technique using palmitic acid as collector.

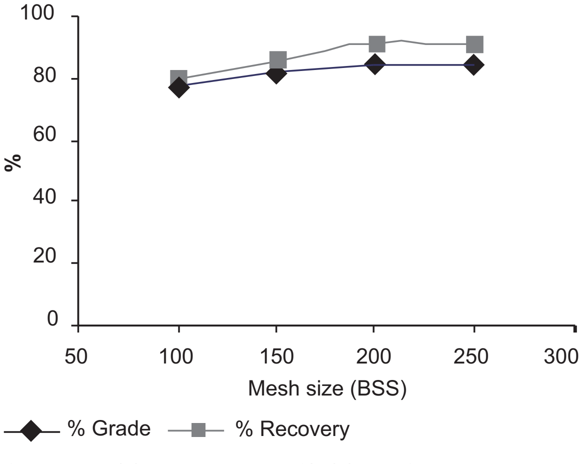
In order to minimize the impurities and as a consequence maximize the percentage of barium content in the ore, it was decided to use gravity concentration and froth flotation techniques for up gradation of this ore. Gravity concentration tests were performed employing laboratory sized Wilfley shaking table. It produced three products named as concentrate, middling and tailing. As can be seen from metallurgical balance of these results (Table 4) that barite was concentrated however, a significant fraction of the barite also reported to the middling and tailing products. The results of the gravity based test work revealed that the barite was not sufficiently liberated at coarser sizes so it could not produce desired grade concentrate with acceptable recovery. The most reasonable result obtained by gravity processing is a grade of 84.07% at a recovery of 60.08%. Obviously, gravity based separation could not be applied successfully to this ore due to fine-grained nature of this ore. Then froth flotation technique using Denver flotation machine was employed for up-gradation of $BaSO_4$ content. The froth flotation technique seemed to be a better option for attaining the desired grade and recovery.

**Effect of feed size of ore.** To study the influence of feed size, different feed sizes of the ore were prepared. The ore was wet ground in rod mill to achieve desired feed size with solid to liquid ratio of 1:1. The flotation tests were carried out on feed sizes of almost 100% passing 100, 150, 200 and 250 mesh sizes. The experiments were performed at arbitrarily selected values and reagents, i.e., at natural pH, 30% pulp density, 1000 rpm, 15 min conditioning period and 20 min of froth collecting time with sodium oleate as collector, polyglycol-400 as frother and sodium silicate as depres-sant. Figure 3 presents the results of the effect of particle size on the grade and recovery of $BaSO_4$ concentrate. This figure depicts a gradual increase in the grade of concentrate with an appreciable increase in the recovery of $BaSO_4$ with the decrease in particle size up to 200 mesh (75 μm). It was established that as the grind size became fine to finer i.e., from 200 to 250 mesh size, the grade improved a little with slight decrease of recovery. The best flotation recovery was obtained when the feed size was almost 100% passing 200 mesh size. This feed size was considered as optimum and selected for onward flotation tests. At still finer grinding i.e., after 200 mesh size, it was noticed that more slimes were generated which had actually lowered the recovery of concentrate. Moreover, lower recovery as well as grade at coarse particle size revealed that barite was not sufficiently liberated from associated gangue and thereby reported to the tailings.

**Effect of pulp density of slurry.** In order to study the effect of pulp density on the flotation behaviour of the composite ore, a series of four experiments was conducted using 20, 25, 30 and 35% solids in the slurry. The flotation feed was conditioned initially at 60-65% solids. The slurry having grind size of 100% passing 200 mesh was conditioned for 15 min at 1000 rpm using appropriate quantities of sodium oleate as collector, polyglycol-400 as frother and sodium silicate as depressant at natural pH. The feed was then diluted to 20-35% solids. The test results are shown graphically in Fig. 4. It may be observed that pulp density variation has a significant influence on grade and recovery of $BaSO_4$. As the pulp density increased from 20 to 25% solids, recovery is improved profoundly with no fall in grade. But when it is further increased to 30% and then 35% solids, a slight change takes place resulting in lower grade and recovery. This behaviour may be due to the fact that a pulp density higher than 25% solids produced thick froth layer which was difficult to wash. It can be explained that the draining out of entrapped gangue particles was not proper at higher pulp densities (Jain, 2001). Accordingly, in subsequent experiments, the pulp density was fixed at 25% solids.

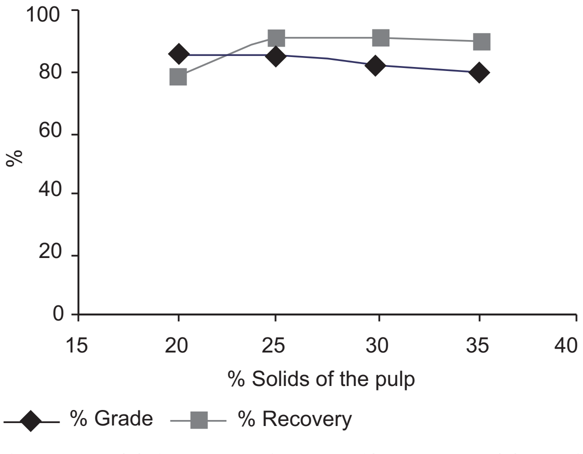
**Effect of pulp pH.** In a series of flotation experiments, pH of the pulp was varied using soda ash as pH modifier and keeping other variables constant. It was found that for different collectors, the froth formed in the pH range of 8-12. With sodium oleate collector it was found that better results were obtained near pH 10. The results attained at different pH values of pulp using sodium oleate as collector are shown in Fig. 5. The plot of barite recovery against pH showed that as the pH increased from 8 to 10, both grade and recovery were increased but after that grade and recovery were dropped slightly. This shows that at pH 10, the mineral-collector bond is more stable leading to maximum recovery. Moreover, at this point, the net adsorbed ion charges become zero so the surface of the barite particles for the adsorption of the collector becomes saturated with collector monolayer (Zhao *et al*., 2014). It was accordingly, decided to maintain the pH at 10 for the subsequent flotation trials for the composite ore.

**Effect of agitation speed.** Some flotation experiments were carried out in order to observe the effect of agitation speed on the grade and recovery of barite concentrate keeping other variables constant and varying the agitation



Conditions: (a) Pulp density: 30%; (b) pH of the pulp: Natural; (c) Impeller speed: 1000 rpm; (d) Collector (sodium oleate): 500 g/ton; (e) Frother (polyglycol-400): 50 g/ton; (f) Depressant (sodium silicate): 500 g/ton; (g) Conditioning time: 15 min and (h) Flotation time: 20 min.

**Fig. 3.** Effect of feed size on grade and recovery of barite rougher concentrate.



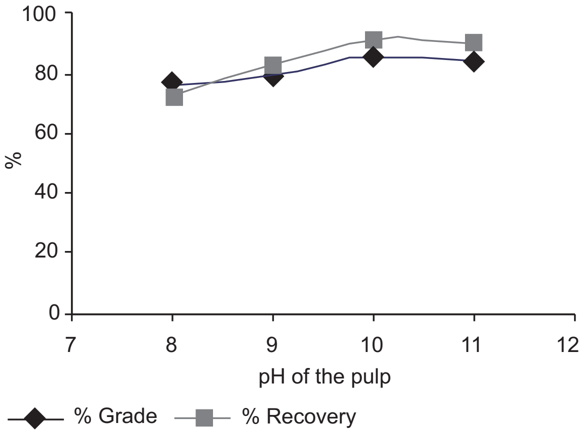
Conditions: (a) Grind size of ore: 100%-200 mesh; (b) pH of the pulp: Natural; (c) Impeller speed: 1000 rpm; (d) Collector (sodium oleate): 500 g/ton; (e) Frother (polyglycol-400): 50 g/ton; (f) Depressant (sodium silicate): 500 g/ton; (g) Conditioning time: 15 min and (h) Flotation time: 20 min.

**Fig. 4.** Effect of pulp density on grade and recovery of barite rougher concentrate.

speed. The flotation feed was conditioned at 25% solids at a pH of 10 which is achieved through the addition of sodium carbonate. The reagents were added and feed was conditioned for a minimum of 15 min prior to the flotation. The reagents used and their quantities are mentioned below the respective figure. The plot of percentage grade and recovery against agitation speed is shown in Fig. 6. It was found that impeller speed of 1100 rpm yielded the best grade concentrate along with high recovery indicating the adequate mixing and aeration in the pulp. Figure 6 elucidates that recovery increases with agitation speed up to 1100 rpm beyond which grade decreases. It was observed that at lower impeller speed (less than 1000 rpm) the surface is quite low so that froth height and its removal were improper leading to lower recovery. On the other hand, higher impeller speed (1200 rpm and above) spilled the pulp along with froth into the launder resulting in lower grade.

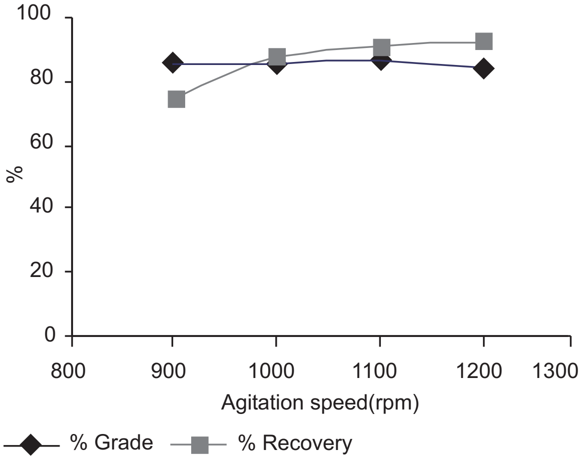
**Effect of collector dosage.** Various types of cationic and anionic collectors are used in flotation of barite ores. The cationic collectors, mostly primary fatty amines, may be used in the reverse flotation of silica and silicate impurities from barite ore when the ore is high grade. The anionic collectors, largely fatty acids and their salts are used in the flotation of barite mineral in case of low grade ores. Alkyl sulphonates or petroleum sulphonates are rarely used as collector for barite although they have greater selectivity but their collecting power is low. As flotation response of calcite and fluorite is similar with fatty acid collectors, the separation from these minerals present some problems. Many investigators have shown that sulphonate are more selective in these systems (Wills and Napier-Munn, 2006; Davis, 1985). The AERO 827 promoter has also been used for many years to float barite mineral. The AERO 845 promoter is particularly preferred where high selectivity against fluorite and calcite is required. The newest AERO 856 promoter has much greater selectivity and has exhibited a significant increase in recovery (Day, 2002).

For present medium grade barite ore, different anionic collectors such as tall oil, oleic acid, palmitic acid, Aero 845 (sulphosuccinamate), and sodium oleate were tried in separate batches in neutral and basic pH range, keeping other parameters constant. Sodium oleate yielded the best over all metallurgical performance in terms of grade and recovery. Although, AERO 845 collector gave a higher grade of concentrate than was obtained with sodium oleate collector but it gave substantially lower recoveries. It appears that sodium salt of fatty acid is more selective for barite than fatty acid collectors. For this test work, sodium oleate was used as the collector since it gave much higher recoveries



Conditions: (a) Grind size of ore: 100%-200 mesh; (b) Pulp density 25%; (c) Impeller speed: 1000 rpm; (d) Collector (sodium oleate): 500 g/ton; (e) Frother (polyglycol-400): 50 g/ton; (f) Depressant (sodium silicate): 500 g/ton; (g) Conditioning time: 15 min and (h) Flotation time: 20 min.

**Fig. 5.** Effect of pulp pH on grade and recovery of barite rougher concentrate.



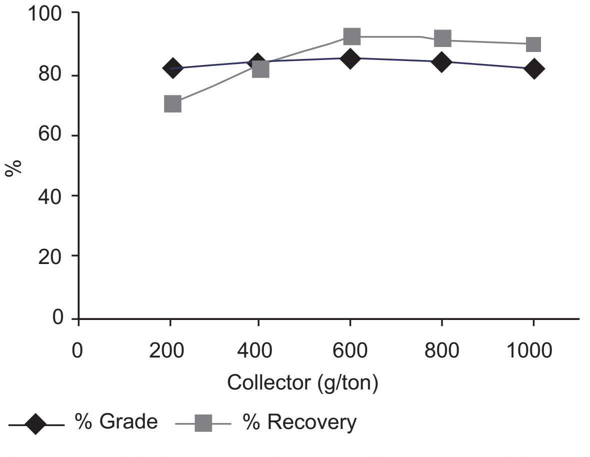
Conditions: (a) Grind size of ore: 100%-200 mesh; (b) Pulp density: 25%; (c) pH of the pulp: ~10; (d) Collector (sodium oleate): 500 g/ton; (e) Frother (polyglycol-400): 50 g/ton; (f) Depressant (sodium silicate): 500 g/ton; (g) Conditioning time: 15 min and (h) Flotation time: 20 min.

**Fig. 6.** Effect of agitation speed on grade and recovery of barite rougher concentrate.

in the rougher flotation. The results of flotation tests at different concentrations of collector are presented in Fig. 7. It can be seen from the obtained results that with increase in concentration of collector from 200 to 600 g/ton, higher recovery of barite results with a slight change in its grade. The collector sodium oleate yielded the best metallurgical results at a concentration of 600 g/ton and hence, it is the optimum value. This amount shows the starvation level i.e., the concentration required for making the monomolecular layer of collector on surface of particles and increased concentration tends to reduce the selectivity by collecting and floating other minerals (Wills and Napier-Munn, 2006).

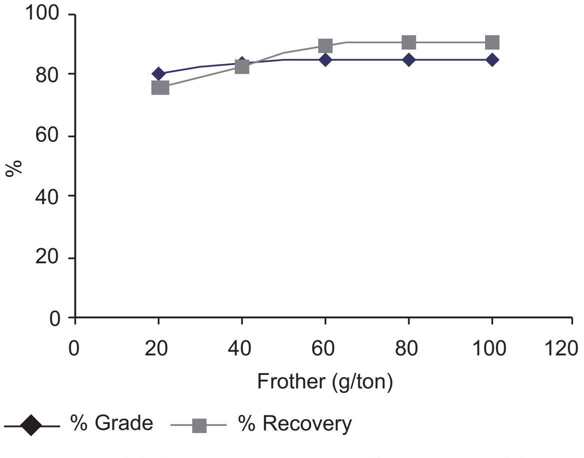
**Effect of frother dosage.** A few flotation experiments were conducted to study the effect of type and dosage of frother on flotation behaviour of barite ore. Various types of frother such as pine oil (mainly terpineol), Cresol (cresylic acid), Aerofroth 65 (polypropylene glycol) and methyl isobutyl carbinol (MIBC) were tried. In a few earlier experiments, pine oil was used as frother in combination with sodium oleate collector, but no considerable change in froth stability was observed. The bubbles produced were of larger size and broke immediately after formation. Then cresylic acid was employed as frother in combination with sodium oleate collector, but the results with respect to grade and recovery were also poor leading to premature breakdown of froth. Results using methyl isobutyl carbinol were also similar showing no encouraging results. Then in another experiment a combination of polyglycol-400 with sodium oleate collector was used. This combination gave very good results in terms of stable froth with dense small size bubbles. Many flotation tests were performed by varying its concentration. The effect of frother dosage on the flotation of composite ore is shown in Fig. 8. The results describe that the best grade and recovery was obtained when 60 g/ton of polyglycol-400 was used as frother and further dosage has no pronounced effect.

**Effect of depressant.** Different contaminants present in barite ore were quartz, various carbonates and sulphides. In order to depress these gangue minerals, three different types of depressants were tried. These were sodium polyphosphate, sodium polyacrylate and sodium silicate. It was observed from these preliminary experiments that sodium silicate was the most effective depressant employed. Hence, further trials were continued using sodium silicate as a regulator. The results obtained using different quantities of depressant are shown graphically in Fig. 9. It is obvious from this figure that with increase in amount of the depressant from 100 to 400 g/ton both the grade and recovery of the concentrate were improved and after that value the grade improved a little but the recovery was decreased.



Conditions: (a) Grind size of ore: 100%-200 mesh; (b) Pulp density: 25%; (c) pH of the pulp: ~10; (d) Impeller speed: 1100 rpm; (e) Frother (polyglycol-400): 50 g/ton; (f) Depressant (sodium silicate): 500 g/ton; (g) Conditioning time: 15 min and (h) Flotation time: 20 min.

**Fig. 7.** Effect of collector dosage on grade and recovery of barite rougher concentrate.



Conditions: (a) Grind size of ore: 100%-200 mesh; (b) Pulp density: 25%; (c) pH of the pulp: ~10; (d) Impeller speed: 1100 rpm; (e) Collector (sodium oleate): 600 g/ton; (f) Depressant (sodium silicate): 500 g/ton; (g) Conditioning time: 15 min and (h) Flotation time: 20 min.

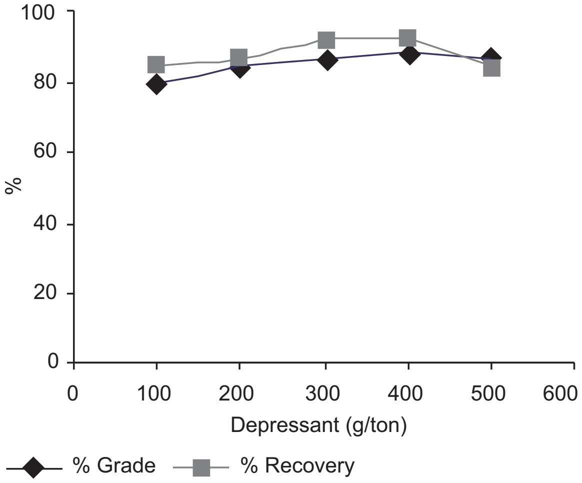
**Fig. 8.** Effect of frother dosage on grade and recovery of barite rougher concentrate.

Hence, for further trials the depressant amount was kept fixed at 400 g/ton. This effect can be explained that sodium silicate efficiently depresses the gangue minerals with proportionate amount but excessive amount decreases the recovery due to an over coating of some middling particles (Wills and Napier-Munn, 2006).

**Effect of conditioning period.** Preliminary flotation experiments were conducted at an arbitrary selected value of 15 min after the addition of reagents. While later on, in order to determine the optimum conditioning period, it was varied from 8 to 16 min keeping other conditions constant. The plot of percentage recovery versus conditioning time is shown in Fig. 10. It indicates that recovery increases with time up to 12 min beyond which recovery decreases. It can be seen from this figure that, conditioning time of over 10 min and less than 14 min produces higher grade and recovery. It was found that maximum recovery of 92% could be obtained in rougher flotation at a conditioning time of 12 min. Increasing the conditioning time beyond this period reduced the recovery as well as the grade to some extent. This may be due to shearing of collector layer initially deposited on the particle's surface due to reverse process of desorption of the collector molecules from the surface of the barite particles (Raju *et al*. 2004). In the later tests a conditioning time of 12 min was considered as optimum and selected.

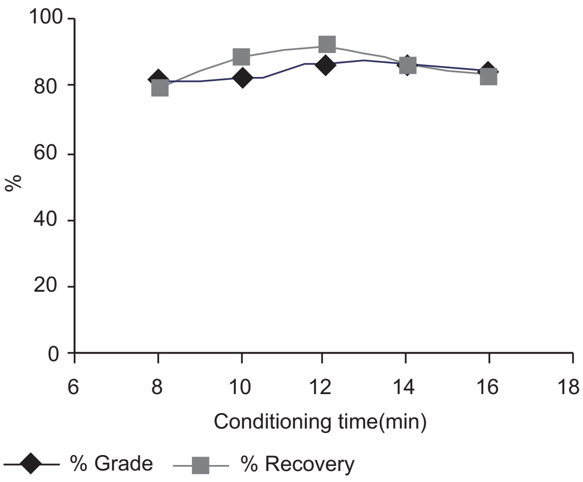
**Effect of flotation time.** After adjusting the other parameters constant i.e., feed size of 100% passing 75 μm, pulp density set at 25% solids and pulp pH of 10, conditioning time for 12 min, a series of four tests was performed to study the effect of flotation time, in which the froth collecting time was varied from 5 to 25 min. The froth was collected separately for these different time intervals. When froth became thinner, additional amount of collector was added and conditioned for another 5 min. This was collected as scavenger concentrate and mixed along with rougher concentrate. The results of this series of tests are recorded and presented in Fig. 11. It is evident from this figure, that most suitable condition was arrived in about 20 min when nearly all the froth was skimmed off and cell was completely barren.

**Effect of cleaning flotation.** After optimizing the rougher flotation parameters, it was decided to conduct one series of cleaning flotation tests to improve the grade of rougher barite concentrate. A few flotation tests were carried out at optimum conditions and a sufficient quantity of rougher concentrate was collected for this purpose. It was observed that about 90% of $BaSO_4$ was recovered in rougher concentrate whereas 10% was lost



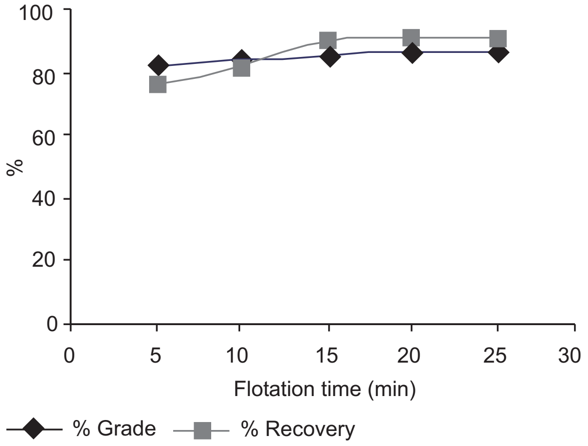
Conditions: (a) Grind size of ore: 100%-200 mesh; (b) Pulp density: 25%; (c) pH of the pulp: ~10; (d) Impeller speed: 1100 rpm; (e) Collector (sodium oleate): 600 g/ton; (f) Frother (polyglycol-400): 60 g/ton; (g) Conditioning time: 15 min and (h) Flotation time: 20 min.

**Fig. 9.** Effect of depressant quantity on grade and recovery of barite rougher concentrate.



Conditions: (a) Grind size of ore: 100%-200 mesh; (b) Pulp density: 25%; (c) pH of the pulp: ~10; (d) Impeller speed: 1100 rpm; (e) Collector (sodium oleate): 600 g/ton; (f) Frother (polyglycol-400): 60 g/ton; (g) Depressant (sodium silicate): 400 g/ton and (h) Flotation time: 20 min.

**Fig. 10.** Effect of conditioning time on grade and recovery of barite rougher concentrate.



Conditions: (a) Grind size of ore: 100%-200 mesh; (b) Pulp density: 25%; (c) pH of the pulp: ~10; (d) Impeller speed: 1100 rpm; (e) Collector (sodium oleate): 600 g/ton; (f) Frother (polyglycol-400): 60 g/ton; (g) Depressant (sodium silicate): 400 g/ton and (h) Conditioning time: 12 min.

**Fig. 11.** Effect of flotation time on grade and recovery of barite rougher concentrate.

in rougher tailing. This loss was reduced to 7.65% by scavenging of tailings with the use of additional reagents (Table 6). The scavenging concentrate was mixed along with rougher concentrate to maximize the recovery up to 92.35%. The rougher concentrate obtained in the series of experiments was ground in a rod mill to get a product of 100% passing 250 mesh size (63 µm). The solid to water ratio was kept at 1:1.

The rougher concentrate was then subjected to cleaner flotation. In the cleaner flotation, additional amount of reagents was added to condition the newly liberated particles. A small quantity of sodium chromate (20 g/ton) was also added to depress galena (Jain, 2001). The cleaner flotation tests were carried out at pH 10 with 15% pulp density. Conditioning was done for 12 min at agitation speed of 1000 rpm. Recovery of $BaSO_4$ in the cleaner concentrate was found to be 86.01% at a grade of 93.17% $BaSO_4$. It can be seen in this comparison that cleaning of the rougher concentrate produces substantially higher grade in the cleaner concentrate at the expense of lower recovery (Table 6). Based on these results it was anticipated that the flow sheet for treating this ore would consist of multiple stages of flotation to improve the grade of barite concentrate.

The cleaner concentrate obtained was subjected to re-cleaner flotation without using any flotation reagents and further re-grinding (Table 5). The pulp density and agitation speed were slightly lowered down to 10% and 900 rpm, respectively, to get higher grade without considering the recovery. The results of re-cleaner flotation tests summarized in Table 6 describe that the cleaner concentrate after further re-cleaning step yielded a product assaying 95.83% $BaSO_4$ at about 82.06% recovery. Higher grade flotation product was achieved with re-cleaning flotation which decreased the recovery in final concentrate. It was observed that sodium chromate has successfully depressed the galena in cleaning stages. Overall recovery has been improved by recycling the cleaning and re-cleaning tailings. The developed flow sheet (Fig. 12) shows the multiple recycling of intermediate tailings at appropriate points to earlier stages of flotation. This has ensured a higher grade and recovery of the final concentrate by limiting the loss of barite to the discardable rougher tailings.

Table 6 presents the chemical composition of the final flotation concentrate. The production of barite concentrate with quality according to the requirements (API, 2010) demands the removal contaminants in barite, such as silica, iron oxide, aluminum oxide, calcium oxide, magnesium oxide and heavy metal sulphide which can cause problems in certain mud systems. Drilling-grade barite is expected to have a grade of at least 92% barium sulphate, specific gravity of 4.20 g/cm$^3$, maximum 3% residue left on 200 mesh screen (75 µm) and should be free of flotation reagents to meet standard API specifications. The concentrate produced contained 95.83% barium sulphate content having specific gravity of 4.28 g/cm$^3$ and was found free of flotation reagents. The particle sizes also fall predominantly in the range finer than 63 µm i.e., well within the required 75 µm limit. However, the impurities of iron ($Fe_2O_3$) and alkaline earth metals (CaO and MgO) are slightly higher in flotation concentrate but content of $SiO_2$ is lower than admissible limit. This is due to the reason that carbonaceous minerals such as siderite, calcite and dolomite also float to some extent with same collector and silica is depressed effectively by sodium silicate. Hence, it was decided to leach out these impurities.

**Effect of acid leaching.** The requirements of barite concentrate for different industries along with standard specifications are also summarized in Table 7 for comparison (Mills, 2006; Khan *et al.*, 2003, Ahmad and Siddiqi, 1992). In the glass industry, the requirements are somewhat rigid but vary to some extent. The general requirements include $BaSO_4$ 96-98%, iron oxide 0.1-0.2% and no more than 1.5% silica and 0.15% alumina.

The barite used in paints should have minimum 95% $BaSO_4$ content and less than 0.05% iron oxide. The rubber industry requires product to be over 99.5% pure with traces of silica, iron oxide and alumina. For the manufacture of various barium chemicals, barite should have at least 95% $BaSO_4$ content, iron oxide less than 1.0% and only traces of fluorine (Mills, 2006; Ahmad and Siddiqi, 1992). Impurities are minimized because these affect the reduction reaction of barite by charcoal or natural gas at 850-950 °C and consequently lower the yield of barium sulphide which is the starting material of other barium chemicals (Khan *et al*., 2003). Silica and alumina when present in appreciable amounts form barium silicate and barium aluminate and keep barite in residue after leaching of barium sulphide with water. Iron is the main colour imparting component which may be present due to iron minerals as well as from abrasion of grinding mills. For paper, glass, ceramics and paint industry its removal is necessary.

Hydrochloric acid and sulphric acid are commonly used for leaching of barite (Kumar *et al*., 2005; Khan, 2003). Sulphuric acid leaching is preferred when ore contain witherite ($BaCO_3$) which is converted into barite on leaching. The main advantage of using hydrochloric acid is that it leaches out anhydrite mineral. Treatment of the final flotation concentrate with hydrochloric acid increases the grade from 95.83 to 98.86% and decreases the unwanted impurities of iron oxide, calcium oxide, magnesium oxide and aluminum oxide to permissible limits. Acid soluble salts of these metals go into the filtrate leaving behind comparatively pure barite. The concentration of 15% hydrochloric was found sufficient to leach out the objectionable impurities (Table 8).

This investigation highlights the applicability of various processes for the beneficiation of low to medium grade barite ore and gives conclusive result regarding the most suitable method for attaining the desired grade and recovery. The technology developed is economical and helpful for the beneficiation rejected and lean grade barite ore for commercial exploitation. The concentrate produced is useful for drilling mud, barite based chemical production and other industrial applications.

**Table 8.** Effect of acid concentration on the purity of final Duddar barite concentrate

| Test no. | Conc. of dilute HCl | Purity of Barite ($BaSO_4$) |
|---|---|---|
| 1 | 5% | 97.63% |
| 2 | 10% | 98.05% |
| 3 | 15% | 98.86% |
| 4 | 20% | 98.87% |

## Conclusions

Mineralogical evaluation of the ore shows that it contains mainly barite as an economical mineral whereas the others are associated gangue minerals. The gravity concentration tests on Duddar barite ore using Wilfley shaking table show that it is not helpful for this ore due to heavy losses of barite in tailings which results in poor recovery. Even the best test resulted in insignificant grade of 84.07% with 60.08% recovery. The direct flotation results show that a barite concentrate assaying 95.85% $BaSO_4$ can be obtained with the recovery of 82.06% from this ore. The optimum grade and recovery of rougher concentrate was attained when the slurry of 100% passing 200 mesh size was conditioned for 12 min with 25% solids at pH 10 maintained with soda ash. The best reagents combination used in the flotation was found to be sodium oleate as collector at dosage of 600 g/ton, sodium silicate as depressant at 400 g/ton, Aerofroth 65 as frother at 60 g/ton. In the closed circuit, one roughing along with scavenging followed by regrinding and two consecutive cleanings operations produced maximum grade and recovery. It is concluded that direct flotation is the most suitable method for the up-gradation of this fine-grained ore for commercial exploitation. The obtained leached concentrate is suitable for the barium chemicals industry.

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