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Flotation studies on low grade graphite ore from eastern India

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1. Introduction

Graphite and diamond are naturally occurring allotropes of crystalline carbon. Graphite is lustrous black carbon mineral which is relatively soft and greasy with a hardness of 0.5–1.0 on Moh's scale [1]. The global graphite market consists of two main products namely amorphous graphite and flake graphite [2].

Graphite generally occurs as a result of metamorphism of organic matter in sediments. Flake graphite is assumed to be derived from fine-grained sediments rich in organic matter. As metamorphic grade increases, carbonaceous material converts to amorphous graphite [1,3]. Flake graphite is classified based on the size of the crystal flakes and graded according to their graphitic carbon content and particle size. Microcrystalline graphite is commercially called as amorphous graphite. The run-of-mine low grade ore containing about 10% fixed carbon (FC) has to be invariably beneficiated before marketing.

The graphite beneficiation processes depend upon the nature and association of gangue minerals present in ore body. It can be enriched easily by flotation because of its natural hydrophobicity [3–5]. Froth flotation process is widely adopted since high-grade graphite concentrates, which are suitable for refractory, battery and lubrication applications, can be produced [6,7]. Flotation utilizes the differences in the surface properties, mainly the hydrophobicity of graphite [8–12]. In froth flotation, graphite ore is treated with hydrocarbon oil to enhance the hydrophobicity

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ABSTRACT

A low grade graphite ore from eastern India was beneficiated by flotation to improve its quality. The ore was composed of 87.80% ash and 8.59% fixed carbon. Primary coarse wet grinding (d_{80} : 186 µm) followed by rougher flotation in Denver flotation cell using diesel as collector and pine oil as frother yielded a rougher concentrate. Regrinding (d_{80} : 144 µm) of this rougher concentrate was opted for further liberation of graphite. It was followed by cleaning in laboratory flotation column. This combined process of relatively coarse primary grinding followed by regrinding and cleaning in flotation column resulted in final concentrate of 7.44% yield with 89.65% fixed carbon and 6.00% ash. This approach of two-stage grinding to recover the flake graphite at the coarsest possible grind can help to minimize grinding energy costs. A conceptual flow sheet which is cost effective was developed based on this methodology.

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and in turn recovery [13]. The effect of diesel and pine oil on flotation performance was discussed earlier [14]. In the case of lean and finely disseminated ores, fine grinding is essential to liberate values from gangue minerals [15,16]. Several stages of cleaning are essential to obtain the required grade of the concentrate by conventional flotation. Under such circumstances, beneficiation by flotation column is advantageous and the overall process can be made cost effective. It is established that the quality of concentrate achieved by three-stage cleaning by conventional flotation cells can be achieved by single stage cleaning by flotation column [17,18]. The low maintenance cost and less circuit complexity are added advantages of flotation column. Hence column flotation is gaining importance in the mineral industry. Beneficiation of low grade graphite ore using mechanical cell and column flotation cell was studied [19–22]. The present investigation aims at enrichment of a low grade graphite ore from eastern India by a combination of conventional and column flotation techniques accompanied by two-stage grinding.

2. Experimental

2.1. Materials

A low grade graphite ore was received from Jharkhand state of India. The ore is crushed in stages followed by thorough mixing. A representative sample was drawn for chemical analysis and the results are as shown in Table 1.

It can be observed that the high ash content indicates that the graphite ore was of low grade. Traces of carbonate minerals like

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Table 1

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unemical	analysis	or graphite ore.	

Sample	Ash (%)	Moisture (%)	Volatile	Fixed
Graphite ore	87.80	0.12	matter (%) 3.49	carbon (%) 8.59

calcite and specks of sulfide minerals like pyrite were found associated with the sample which contributed to the volatile matter.

2.2. Size analysis

The particle size distribution of the crushed graphite ore was carried out on BSS sieves and the weight percentage retained on each screen along with their ash values were presented in Table 2. The calculated d_{80} of this graphite sample was found to be 605 µm.

From the results in Table 2, it is evident that the ash content is above 88.83% in all fractions. This implies that graphite is not liberated and requires further size reduction before attempting to recover the graphite values.

2.3. Mineralogy of the ore

The mineralogical characterization of graphite ore was carried out to determine the extent of graphitic carbon content and graphite flake size. These two properties determine the economic value of the graphite and also provide a basis for beneficiation studies. Petrographic characterization (Fig. 1) under optical microscope indicated that the sample consists primarily of quartz and graphite in the form of both fine and thick flakes dispersed in the silica matrix, with minor quantity of mica (biotite).

2.4. X-ray diffraction analysis

The graphite ore was subjected to X-ray diffraction studies for mineralogical phase analysis, especially the identification of nongraphite minerals [23]. The X-ray diffractogram is shown in Fig. 2.

The characteristic X-ray of copper-K α radiation with 0.154 nm wavelength was used in this diffraction study. The sample was found to consist predominantly of quartz with minor fractions of graphite and mica (biotite). The high intensity of quartz peak and also the presence of mica indicate the high ash content of the ore.

Table 2

Size (µm)	Weight retained (%)	Ash (%)	Ash distribution (%)
-1680 + 850	12.13	91.01	12.23
-850 + 500	14.91	91.69	15.14
-500 + 300	17.49	90.02	17.45
-300 + 212	15.31	89.24	15.14
-212 + 106	22.86	90.92	23.02
-106	17.30	88.83	17.02

2.5. Column flotation

The flotation tests in this study were carried out using a 74 mm diameter laboratory scale flotation column designed and fabricated by CSIR-NML Madras Centre, India. A Plexiglas flotation column 5000 mm in height and 74 mm in internal diameter was used. The design of the column allows various parameters, such as column height, feed injection point, air flow rate and feed flow rate to be altered. The feed injection point was located approximately two thirds of the way up the column. The bubble generator (sparger) is an internal type that has been designed at NML Madras Centre. Electronically controlled metering pumps were used to feed and discharge the slurry. The pumps were designed to deliver an accurately measured volume of slurry with an error of ±3%. The pumping rate can be adjusted either manually or automatically by a signal from a remote control unit. Digital display of the pumping rate was also incorporated. A differential pressure transmitter was used to maintain a constant level between the slurry and froth phase. The output signal generated by the transmitter was looped with the stroke controller of the tailings pump so that the pumping rate could be automatically varied to maintain the interface level at a fixed froth depth. The controller was fitted with an alarm that could be set to go off if the incoming signal fell below or exceeded any level between 0% and 100% of input. Under steady-state conditions, the interface level could be maintained at a constant height to within ±10 mm. Purge rotameters with a differential pressure regulator and a needle valve were used to control the flow of air and wash water. This type of rotameter was chosen to minimize the variation in flow with changes in inlet and outlet pressures. The overall automated column that was developed at the Centre is shown schematically in Fig. 3. For each run a sufficient quantity of material was taken in the conditioner and conditioned with reagents. The conditioned slurry was fed to the column through the feed pump. At steady state, that is after running the column for three times of the slurry residence time, samples were collected for analysis. The column test procedure suggested by Delviller et al. was followed [24]. Diesel and pine oil used in the tests were of commercial grade.

3. Results and discussion

The low grade graphite ore was crushed in stages followed by primary coarse wet grinding at 186 μ m (d_{80}) using a laboratory ball mill. Sodium silicate was added during grinding to act as depressant for quartz and mica in the subsequent flotation. Rougher flotation was carried out in D12 Denver flotation cell to eliminate as much gangue as possible in the form of primary tailings with minimal loss of graphite values. The rougher concentrate was subjected to regrinding (d_{80} : 144 μ m) to further liberate graphite values. This rougher concentrate was used as the feed to laboratory scale flotation column to optimize its operating parameters.

All the column flotation tests were conducted at natural pH and 12% solids by weight, unless otherwise mentioned. The tests were



Fig. 1. Petrographic images of graphite ore. (a) Long graphite flakes dispersed within silica matrix. (b) Thick fibrous graphite in silica matrix.

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