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Single Reagent for Graphite Flotation

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Abstract

Generally, diesel and frother are used as reagents in graphite flotation. With the escalating cost of petroleum products and their negative impact on environment, attempts are made to formulate an eco-friendly single reagent to replace the diesel-frother system without affecting the flotation performance. CSIR-NML Madras Centre in collaboration with M/s Somu Organo Chem Pvt Ltd., India, has worked out the formulation and evaluation of single reagent on a low grade graphite ore sourced from eastern India. The petrography studies indicate that the ore primarily consists of quartz and graphite with minor quantity of mica and analyzing 87.85% ash content. The ore is crushed in stages followed by primary coarse wet grinding to 242 µm (d_{80}). Rougher flotation is carried out in Denver flotation cell with a view to eliminate gangue as much as possible in the form of primary tailings with minimal loss of carbon. Regrinding of rougher concentrate to 216 µm (d_{80}) is opted to improve the liberation of graphite values. This approach involving a primary coarse grinding and regrinding of rougher float followed by multi-stage cleaning using this single reagent is found to yield better recovery and grade when compared with that of the dual reagent system. A final concentrate of 12.03% weight recovery with 3.22% ash could be achieved. Based on encouraging laboratory studies using the single reagent, plant trials were carried out. From the cost benefit analysis, this single reagent proves to be an economically viable in place of diesel-frother for processing low grade graphite.

Keywords: Low grade graphite; liberation; froth flotation; single reagent; diesel.

1. Introduction

The global graphite market consists of two main products namely amorphous graphite and flake graphite (Wakamatsu and Numata, 1991). Graphite generally occurs as a result of metamorphism of organic matter in sediments. Flake graphite is assumed to be derived from the fine-grained sediments rich in organic matter. As metamorphic grade increases, carbonaceous material converts to amorphous graphite (Dey and Pathak, 2005). Flake graphite

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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 r.o.m ore of low grade, containing about 10% fixed carbon (FC), has to be invariably beneficiated before marketing. Processes for graphite beneficiation depend upon the nature and association of gangue minerals present. Graphite can be enriched easily by flotation because of its natural hydrophobicity (Kaya et al, 2007 and Acharya et al, 1996). Froth flotation process is used widely as it helps in producing a high-grade graphite concentrate (Kim et al, 2003) which finds applications in refractories, batteries and high temperature lubricants (Mitchell, 1993). Flotation utilizes the differences in the surface properties mainly the hydrophobicity of graphite (Chau et al, 2009; Kim et al 2002 and Deryagin et al, 1982) which is one of the main factors in determining the separation efficiency by flotation. Beneficiation of low grade graphite ore using mechanical cell and column flotation cell were studied (Bhaskar Raju et al, 2000; El-Raheim, 2004 and Narasimhan et al, 1972) by using conventional hydrocarbon oils as collectors.

In froth flotation, graphite ores are often subjected to a suitable hydrocarbon oil treatment to alter their hydrophobicity, enhance recovery, and / or improve selectivity (Patil et al, 2000). Conventionally, the collectors used in most of the graphite flotation studies are diesel, kerosene, petrol in combination with pine oil as frother (Didolkar et al, 1997). Various combinations of these conventional reagents namely kerosene collector in combination with 90:10 ratio of methyl isobutyl carbinol (MIBC) and ethyl alcohol were studied in graphite flotation (Ravichandran et al, 2012). Also commercial grade frothers such as pine oil, MIBC, eucalyptus oil and ethyl alcohol with kerosene and diesel oil as collectors were used in graphite flotation (Ravichandran et al, 2013). Flotation studies of graphite in aqueous salt solution using sodium acetate were carried out (Grabowski and Drzymala 2008). The polyethylene oxide type of frothers like polyoxypropylene glycol butyl ether was also studied as a replacement for MIBC in graphite flotation (Pugh, 2000). Major studies on collectors and frothers other than diesel/kerosene and MIBC/pine oil respectively were found much in graphite flotation literatures. Hence in this present investigation, eco-friendly single reagent was developed as a replacement for diesel-frother system. This single reagent developed was tested for its efficiency at laboratory scale. After encouraging results are obtained at laboratory scale, trials were undertaken at industrial scale in a graphite beneficiation plant with 5.0 t/h flotation circuit. Also the cost benefit analysis was carried out to study the economic feasibility of the single reagent developed with that of diesel-frother system.

2. Materials and methods

2.1. Materialsa low grade run-of-mine graphite ore was received from Jharkhand state of India. The ore is crushed in stages followed by mixing thoroughly. A representative sample was drawn for size and chemical analysis (Reddy et al, 1997) and the results are shown in Table 1.

Table 1: Analysis of graphite ore.

Sample	Ash, %	Moisture, %	Volatile Matter, %	Fixed Carbon, %
Graphite ore	87.80	0.12	3.49	8.59

2.2. Size analysis

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

Table 2: Size & Ash Analysis of stage-crushed graphite.

Size, µm	Wt. retained, %	Ash, %	Ash Distribution, %
+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

It is evident from the above table that the ash content is above 88.83% in all size fractions. This implies that there are hardly any free and liberated graphite flakes in any size range and demands size reduction before attempting to recover the graphite values.

2.3. Mineralogy

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 feasibility studies. Petrographic characters (Figure 1a to 1d) under optical microscope indicated that the sample consists primarily of quartz and graphite in the form of both fine & thick flakes dispersed in the silica matrix, with minor quantity of mica (biotite).

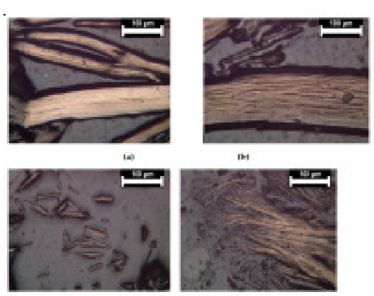

Figure 1. (a) Petrographic images of graphite ore with long graphite flakes dispersed within silica matrix; (b) Thick fibrous graphite surrounded by silica; (c) Fine flakes of graphite dispersed in silica matrix and (d) Thin fibrous graphite.

2.4. X-ray diffraction studies

The graphite ore was subjected to x-ray diffraction studies for mineralogical phase analysis especially the identification of non-graphite minerals (Delviller et al, 1992) and the diffractogram is shown in Figure 2. The characteristic x-ray of copper-Ká radiation with 1.54 Å wavelength was used in this diffraction study. The sample was found to consist predominantly of quartz and with minor fractions of graphite and traces of mica. This high intensity of quartz presence contributes to the high ash content of the ore.

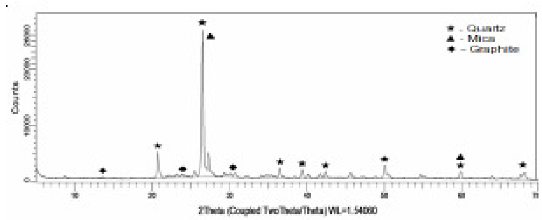

Figure 2. X-ray diffractogram of graphite ore

2.5. Flotation methodology

The flotation experiments were conducted in Denver laboratory flotation machine, D-12 with a cell volume of about 3000 ml. The flotation studies were carried out on graphite feed with eco-friendly single reagent Sokem 705C. Also, flotation tests using conventional reagents namely diesel as collector for graphite and pine oil as frother and sodium silicate as depressant for silica / silicate bearing minerals were carried out for performance comparison and evaluation of the single reagent with the conventional reagents. Flotation experiments were conducted at 15% solids at natural pH and impeller speed of 1200 rpm. The graphite slurry was conditioned with the reagents for 3 minutes each and the froth (float) was collected till froth formation ceased. The collected float and tailings were dewatered, dried, weighed and subjected to ash analysis. The combustible recovery (CR), ash rejection (AR) and efficiency index (EI) were calculated from the weight recovery / yield and ash values for each test using the following equations respectively.

$$\%CR = \frac{M_C(100 - A_C)}{M_f(100 - A_f)} x100$$

$$\%AR = 1 - \left(\frac{M_C A_C}{M_f A_f}\right) x100$$

$$EI = (CR + AR) - 100)$$

where,

A_c = ash content in concentrate, %

A_f = ash content in feed, %

M_c = Mass of concentrate, g

M_f = Mass of feed, g

Also plant trials were conducted at a graphite flotation plant circuit of 5.0 t/hr capacity using Sokem 705C for validating the findings obtained from the laboratory flotation studies.

2.6. Plant trails

Plant trials were conducted at a graphite flotation plant with a treatment capacity of 5.0 t/hr. For this purpose 0.05tonnes of the reagent Sokem 705C was used for 7 shifts continuously to validate the findings obtained in the laboratory studies.

2.7. Flotation reagents

The new eco-friendly single reagent Sokem 705C, a proprietary chemical of ether-alcohol based was used in this graphite flotation studies. This single reagent was manufactures by M/s Somu Organo Chem. Pvt. Ltd., Bangalore and evaluated by CSIR-NML Madras Centre. Commercial grade diesel and pine oil used for the flotation experiments were purchased from the local market.

3. Results and Discussion

3.1. Effect of primary grind time

Flotation tests for primary grind optimization studies were carried out using single reagent, Sokem 705C. Sodium silicate (3.0 kg/t) was used as depressant for silica bearing minerals during grinding. The graphite ore was subjected to primary wet grinding in a ball mill for different periods of grinding time. Each time the ground sample was subjected for size analyses to find out d_{80} of it. The grinding media used was 3.5 kg of steel balls and grinding was carried out at 65% solids by weight. After primary grinding, 0.137 kg/t Sokem 705C was added as collector for graphite flotation and this same dosage was maintained for all the grind variation studies. The concentrate and tailing obtained were dried and subjected to ash analysis and tabulated as shown in Table 3.

Table 3. Effect of primary grind variation on flotation Sodium silicate: 3.0 kg/t; Sokem 705C: 0.137 kg/t; Ash of Head sample: 87.85%

Primary grind time, minutes	d_{80} size, µm	Products	Yield, %	Ash, %	CR, %	AR, %	EI
0	605	Concentrate	14.11	49.15	60.77	92.13	52.90
		Tailing	85.89	94.60			
5	349	Concentrate	19.12	44.11	84.62	90.35	74.97
		Tailing	80.88	97.60			
7	292	Concentrate	19.20	48.42	86.42	89.50	75.92
		Tailing	80.80	98.07			
9	242	Concentrate	17.03	35.93	86.96	93.00	79.96
		Tailing	82.97	98.03			
11	206	Concentrate	17.57	38.29	88.12	92.33	80.45
		Tailing	82.43	98.23			
13	180	Concentrate	16.63	34.18	89.45	93.52	82.97
		Tailing	83.37	98.45			
15	164	Concentrate	16.14	32.93	88.98	93.95	82.93
		Tailing	83.86	98.41			

From the above results, the primary grind at 9 minutes was found to be optimum as the ash content of the rougher concentrate was relatively lower at 35.93% with respectable weight recovery of 17.03%. Also the ash rejection in tailings was high at 93.00% at the coarsest possible primary grind. It is essential to keep the graphite flake size as big as possible which is the criteria for its value/price. Hence, the d_{80} of 242 µm obtained at 9 minutes primary grind was found to be optimum and all subsequent flotation tests were conducted at this particle size.

3.2. Effect of Sodium silicate

The d_{80} size of primary grind was fixed at 242 µm. After primary grind variation studies, sodium silicate dosage optimization was carried out. A fixed dosage of 0.137 kg/t for Sokem 705C was maintained for flotation for all the tests in this series and the flotation test results are given in Table 4.

Table 4. Effect of sodium silicate variation on flotation
Primary grind d_{80}: 242 µm; Sokem 705C: 0.137 kg/t; Ash of Head sample: 87.85%

Sodium silicate dosage, kg/t	Products	Wt, %	Ash, %	Ash Distribution, %	CR, %	AR, %	EI
1.0	Concentrate	19.48	49.22	11.05	74.76	88.95	63.71
	Tailing	80.52	95.85	88.95			
2.0	Concentrate	16.87	43.16	8.39	72.37	91.61	63.98
	Tailing	83.13	95.60	91.61			
3.0	Concentrate	17.48	43.40	8.77	73.18	91.23	64.41
	Tailing	82.52	95.60	91.23			

The sodium silicate dosages were varied from 1 to 3 kg/t. From the results obtained, the sodium silicate dosage was optimized at 3 kg/t as the efficiency index is high at 64.41%. The dosage of sodium silicate was maintained at 3.0 kg/t for further optimization studies.

3.3 Effect of single reagent Sokem 705C

The primary grind (d_{80}: 242 µm) and sodium silicate dosage (3 kg/t) were optimized. Further flotation tests on single reagent Sokem 705C dosage variation were carried out to study its effect and the results are given in Table 5.

Table 5: Effect of Sokem 705C at primary grind on graphite flotation Primary grind d_{80}: 242 µm; Sodium silicate: 3.0 kg/t; Ash of Head sample: 87.85%

Sokem 705C dosage, kg/t	Products	Wt, %	Ash, %	Ash Distribution, %	CR, %	AR, %	EI
0.110	Concentrate	14.92	25.02	4.29	85.90	95.71	81.61
	Tailing	85.08	97.85	95.71			
0.137	Concentrate	15.12	26.69	4.63	86.67	95.37	82.04
	Tailing	84.88	97.99	95.37			
0.160	Concentrate	16.36	30.44	5.73	86.45	94.27	80.72
	Tailing	83.64	97.88	94.27			
0.181	Concentrate	17.65	34.08	6.93	88.26	93.07	81.33
	Tailing	82.35	98.12	93.07			
0.195	Concentrate	18.56	38.49	8.20	88.58	91.80	80.38
	Tailing	81.44	98.19	91.80			

From the above flotation results, the optimized Sokem 705C dosage was found to be at 0.137 kg/t. At this dosage, the rougher concentrate assaying 26.69% ash with weight recovery of 15.12% and ash rejection of 95.37% in tailings could be achieved. The efficiency index at this dosage was found to be the highest at 82.04.

In order to compare the performance of single reagent Sokem 705C with diesel-pine oil system, further graphite flotation studies were carried out using diesel and pine oil.

3.4 Effect of diesel

The primary grind (d_{80}: 242 µm) and sodium silicate dosage (3.0 kg/t) were maintained the same. Flotation tests on diesel dosage variation at fixed dosage of pine oil (0.154 kg/t) were carried out to study its effect and the results are given in Table 6.

Table 6. Effect of diesel at primary grind on graphite flotation , Primary grind d_{80}: 242 µm; Sodium silicate: 3.0 kg/t; Pine oil: 0.154 kg/t, Ash of Head sample: 87.85%

Diesel dosage, kg/t	Products	Wt, %	Ash, %	Ash Distribution, %	CR, %	AR, %	EI
0.198	Concentrate	13.88	34.42	5.45	73.81	94.55	68.36
	Tailing	86.12	96.25	94.55			
0.231	Concentrate	14.52	38.45	6.33	75.33	93.67	69.00
	Tailing	85.48	96.58	93.67			
0.265	Concentrate	15.43	38.74	6.85	74.32	93.15	67.47
	Tailing	84.57	96.13	93.15			
0.330	Concentrate	15.00	38.00	6.51	75.12	93.49	68.61
	Tailing	85.00	96.37	93.49			

From the results, the diesel dosage is optimized at 0.198 kg/t as the ash rejection is maximum in the tailings at 94.55% with relatively good efficiency index of 68.36 at relatively lower diesel consumption. Optimization of pine oil dosage was carried out at this diesel dosage.

3.5. Effect of pine oil

The primary grind (d_{80}: 242 μm), sodium silicate dosage (3.0 kg/t) and diesel (0.198 kg/t) were maintained the same. Flotation experiments on variation of pine oil dosage to study its effect were carried out and the results are given in Table 7.

Table 7. Effect of pine oil at primary grind on graphite flotation, Primary grind d_{80}: 242 μm; Sodium silicate: 3.0 kg/t; Diesel: 0.198 kg/t, Ash of Head sample: 87.85%

Pine oil dosage, kg/t	Products	Wt., %	Ash, %	Ash Distribution, %	CR, %	AR, %	EI
0.154	Concentrate	13.88	34.42	5.45	73.81	94.55	68.36
	Tailing	86.12	96.25	94.55			
0.185	Concentrate	14.40	37.63	6.17	73.87	93.83	67.70
	Tailing	85.60	96.29	93.83			
0.246	Concentrate	14.93	37.68	6.44	73.60	93.56	67.16
	Tailing	85.07	96.08	93.56			
0.277	Concentrate	15.51	40.43	7.17	73.97	92.83	66.80
	Tailing	84.49	96.15	92.83			

From the pine oil optimization results, it is observed that 0.154 kg/t dosage is sufficient for providing good separation (EI: 68.36). As the pine oil dosage is increased further, the concentrate grade is found to be diluted.

3.6. Comparison of single reagent Sokem 705C and diesel-pine oil system

The flotation tests were also carried on graphite using diesel as collector and pine oil as frother at a primary grind of d_{80}: 242 μm and sodium silicate dosage at 3.0 kg/t. The consumption of reagent dosages at equivalent metallurgical grade of the rougher concentrate was compared as shown in Table 8.

Table 8: Comparison of reagents consumption at rougher flotation of graphite

Reagent dosage, kg/t	Products	Wt., %	Ash, %	Ash Distribution, %	CR, %	AR, %	EI
Diesel: 0.198 Pine oil: 0.154	Concentrate	13.88	34.42	5.45	73.81	94.55	68.36
	Tailing	86.12	96.25	94.55			
Sokem 705C: 0.137	Concentrate	15.12	26.69	4.63	86.67	95.37	82.04
	Tailings	84.88	97.99	95.37			

From the above table, it can be observed that the consumption of single reagent Sokem 705C is relatively less as compared to diesel-pine oil system. The cost benefit analysis is given in the following discussion. Also, the performance of Sokem 705C is superior to that of diesel-pine oil system as evident from the efficiency index values.

3.7 Regrinding of rougher concentrate followed by 5-stage cleaner flotation

In the rougher flotation, it was seen that much of the gangue is rejected at the coarsest possible size in the form of primary tailings. In order to further liberate the graphite values in the rougher concentrate, regrinding was carried out. Hence, the rougher concentrate was subjected to regrinding for 40 minutes (d_{80}:144 μm) in ball mill with 1.5 kg/t sodium silicate followed by 5-stage cleaner flotation. During cleaner flotation, no reagent was added and the result of cleaner flotation is given in Table 8.

Conditions during rougher flotation:

Primary grind d_{80}: 242 μm; Sodium silicate: 3.0 kg/t

Sokem 705C: 0.137 kg/t

Ash of Head sample: 87.85%

Conditions during cleaner flotation:

Regrind (rougher concentrate) d_{80}: 144 μm; sodium silicate: 1.5 kg/t

Table 9: Cleaner flotation of reground rougher concentrate

Products	Yield, %	Ash, %
Final. Conc.	12.03	3.22
Cleaner-V Tails	0.23	26.51
Cleaner-IV Tails	0.20	58.38
Cleaner-III Tails	0.34	80.79
Cleaner-II Tails	1.00	92.76
Cleaner-I Tails	3.88	97.28
Primary Tails	82.32	97.48

From the above results, final concentrate of 12.03% yield with 3.22% ash content was obtained after 5-stage cleaning of reground rougher concentrate. The combustibles recovery was found to be 82.03% and the separation efficiency index of 81.58.

After the encouraging results on using single reagent Sokem 705C in place of the dual reagent system at laboratory scale studies, it was thought prudent to test the same at commercial plant level.

4. Plant trials using Sokem 705C

After ensuring that the efficacy of single reagent Sokem 705C is not inferior to that of the diesel-pine oil system in graphite flotation, trials were undertaken in an operating graphite flotation plant in eastern India. The plant of 5.0 t/h capacity involves multistage grinding-cum-flotation. In this plant, diesel and pine oil are being used as collector and frother respectively. Plant trials were conducted using Sokem 705C in this plant flotation circuit. The results of conventional plant practice with diesel and pine oil system and single reagent Sokem 705C are comparable as shown in Table 10.

Table 10. Results of plant trials conducted using single reagent Sokem 705C

Reagent	Dosage, cc/t	Feed Ash, %	Final Conc. Ash, %	Tailings Ash, %	Yield, % (graphite recovery)
Sokem 705C	266	87.50	3.95	94.40	10.90
Diesel & pine oil	225 & 758	87.50	3.90	94.00	11.08

The results of the plant trails indicates that the efficiency of separation using single reagent Sokem 705C is comparable in terms of yield and ash content in the final concentrate. The consumption of Sokem 705C is 266 cc/t of feed which is much less compared to that when diesel and pine oil are being used in the plant. This single reagent doesn't have adverse effect on the downstream operations in the plant such as thickener water quality. Also, the addition of this reagent also makes the operation easier.

4.1 Cost benefit analysis

The cost and benefits analysis was carried out to compare the economic viability in using single reagent in place of diesel-frother (pine oil) system. The throughput capacity of the flotation circuit in the plant where trials were carried out is 5.0 t/h. The reagents cost estimation comparison for treating one tonne of graphite is given in Table 11.

Table 11: Cost benefit analysis for single reagent, Sokem 705C and diesel-frother system

Parameters	Values	Cost (INR)
Feed rate (tonne/hour)	5.0	–
Diesel consumption (cc/t)	225	12.60
Frother (pine oil) consumption (cc/t)	758	113.70
Sokem 705C consumption (cc/t)	266	66.50

It is evident that the cost of single reagent is cheaper by half of the dual reagent cost per tonne of feed. The reagent cost per day for Sokem 705C would be 7980 INR while that of diesel-pine oil system would be 15156 INR. This is over and above the reduced capital expenditure and operational simplicity.

5. Conclusion

The single reagent Sokem 705C was evaluated for its efficiency on graphite flotation both in laboratory bench scale studies and in an operating graphite beneficiation plant. It was found that single reagent was superior to the conventional diesel-frother system, in terms of cost benefit analysis. Flotation results also indicate that the single reagent Sokem 705C is superior to diesel-pine oil system. Considering the plant operation point of view, handling single reagent system is much easier than compared to dual reagent system. This single reagent Sokem 705C proves to be economical compared to diesel-frother system. Moreover, it is biodegradable and environmental friendly.

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