Ultra Chemistry Vol. 8(2), 159-168 (2012).

Beneficiation of low grade graphite ore deposits of Tamilnadu (India)

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(Acceptance Date 23rd May, 2012)

Abstract

Low grade graphite ore containing 14-15% fixed carbon (F.C) from Tamilnadu Minerals Limited (TAMIN) have been investigated for beneficiation. It responds well to the flotation technique. The existing graphite beneficiation plant has the capacity to process 200 tons of ore per day to yield graphite concentrate with fixed carbon content of 84 to 96% purity and recovery of 80 to 88 %. The representative sample collected from the plant in 15 days duration is matching well with the feed quality of plant. The rod mill discharge sample proximate analysis of each size fraction was carried out on dry basis. Kinetic flotation study of Methyl Isobutyl Carbinol (MIBC) and ethyl alcohol 90:10 with kerosene 10:90 ratio reagent dosages gives better recovery and assay. Similarly the above reagent dosage gives rapid flotation rate. Beneficiation studies indicated that the percent recovery of final lock cycle test is 85% and fixed carbon content of floatation concentrate is 96.24%. The +72 mesh size of float concentrate is 25.2% which is meeting as per the requirement. Bench-scale flotation test shows that it is possible to obtain a maximum grade of 97% F.C, with a recovery of 89%.

Key words: Industrial minerals; beneficiation; froth flotation; reagents; lock cycle test; kinetic study.

Nomenclature

MIBC	Methyl Isobutyl Carbinol
RMD	Rod mill discharge
MOG	Mesh of grind
F.C	Fixed carbon
V.M	Volatile matter
BSS	British s*tandard sieve*

1. Introduction

Graphite constitutes one of the most important minerals for the manufacture of special types of refractory and other materials. In nature, it occurs both in flaky crystalline as well as amorphous forms. Graphite has a unique role in different industries due to its various

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physical and chemical characteristics. India has a recoverable graphite reserve of 3.1 million tons. Low grade graphite ore forms the bulk of these deposits, which needs to be beneficiated[1-7]. Crystalline flaky size and the relationship of graphite with the associated gangue minerals influence the beneficiation process. Commercial graphite is a relatively expensive industrial mineral. To obtain good quality of graphite concentrate, beneficiation is essential in order to obtain optimal prices for the finished product. A survey of literature indicates that there are few references concerning fine graphite concentration. Instead, many efforts have been made on the concentration of flaky graphite[8-9] and graphite flotation reagents[10]. Froth flotation is commonly used for the concentration of low grade graphite ore. This paper present the results on the beneficiation of low grade graphite ore from TAMIN, India, by using mechanical cell and column flotation.

There are several papers in the literature reporting the flotation of graphite. Parks and Dodd[10] described the flotation of Northern Tanzania graphite to give a high grade, large flaky (>300 micron) product using MIBC frother with sodium silicate gangue depressant. Wakamasu and Numat[11,12] reviewed the flotation of graphite from a surface chemistry viewpoint, and discussed the flotation processes in Sri Lanka, Korea and Japan. Patil *et al.*,[13], discussed the flotation of an Indian ore using sodium silicate and a guar gum depressant. In all reported cases to date, MIBC or kerosene was used as a frother. A more fundamental paper published and illustrates the effect of inorganic electrolytes on the flotation of graphite[14].

Most of the mineral resources in India are low to medium grade which is need beneficiation in some form or other to make them suitable for use in mineral based industries. As such, these need to be upgraded to the desired specification (within specified tolerance limits). Beneficiation of graphite includes gravity concentration and froth flotation. Sometimes chemical treatment like acid leaching and chloridisation are also applied for production of high purity graphite over 99% F.C. Comprehensive beneficiation studies have been carried out on Sivaganga graphite ore samples and a brief account is being presented in this paper.

2. Materials and Methods

2.1. Materials

The representative sample collected from the rod mill discharge in 15 days duration and contains an average of 14.83% F.C. Sodium silicate was used as dispersing reagent for silicate phase minerals. The sodium carbonate was used as pH regulator. The commercial grade kerosene was used as collector whereas commercial grade MIBC and ethyl alcohol in the ratio of 90:10. The above frother is mixed with the kerosene collector in the ratio of 10:90.

2.2. Methods :

The mill discharge (RMD) of size -600 µm sample is taken in a mechanical flotation cell and it is conditioned with Na_2CO_3 to adjust the p^H to 8.5. Sodium silicate solution was added to the pulp to depress the gangue minerals and conditioned. Methyl Isobutyl Carbinol *(MIBC)* and ethyl alcohol frothers are mixed with the kerosene collector is added and the float concentrate is collected. Subsequently, bench scale conventional flotation tests were conducted after grinding the float sample to a suitable fineness.

3. Experimental set-up and procedure

3.1 Laboratory Denver D12 flotation cell:

Kinetic and bench scale conventional flotation tests were performed in a flotation machine is shown in Fig.1 supplied by Denver. The apparatus provides recirculation and agitation to maintain thorough mixing and solid-liquid suspension. The vertical position of the agitator can be adjusted by pulling out the stop knob on the left side of the unit and turning the lever on the right-hand side. The rotation speed (rpm) of the agitator is varied by turning the knob at the top of the motor and at the back of the unit. An rpm tachometer is located at the top of the unit. Aeration is controlled using a needle valve. All the tests pertaining to selection and optimization of reagent's dosages were performed in this flotation cell.

The representative ground ore pulp from the rod mill discharge of size -600 µm sample of 1 kg was taken in 5 lit capacity flotation cell and it is conditioned with Na_2CO_3 (1.4 kg/tons) to adjust the p^H to 8.5. Subsequently, sodium silicate was added to the pulp (0.5kg/ton) to depress the gangue minerals and conditioned at RPM of 1500 for 5 minutes. Further, the pulp was also conditioned with (0.2 kg/ton) of MIBC and ethyl alcohol in the ratio of 90:10. The above frother was mixed with the kerosene collector in the ratio of 10:90. The pulp was conditioned for 5 minutes rigorously. Initial float concentrate was collected every 10 seconds interval and the final float was collected for additional 2 and 4 minutes respectively. The total collection time up to 8 minutes and sample was subjected to chemical analysis. The experiments were repeated twice to verify the repeatability of the kinetics and to make out the reagents efficiency.

Bench scale flotation tests were conducted after grinding the sample in rod mill to a suitable fineness. Further the RMD sample floats were ground in a ball mill. The grinding time is varied to obtain the desired mesh of grind (MOG). The above recommended optimized reagents from the gas hold-up studies were used.

4. Results and Discussion

The typical results of the flotation kinetic studies are given in Table.1 and shown in Fig. 2. From the results, it is observed that the cumulative percent recovery of 91.3% is optimum at 120 seconds. However, 8 minutes of flotation residence time is required to improve the overall cumulative percent recovery to 93%. The particle size distributions of float concentrate and tailings results are given in Table 2. Hence, it is confirmed that the reagents used in the present study are working effectively.

4.1 Final open cycle test :

The bench scale tests were carried out using mechanical flotation cell having 5 liter capacity. The rod mill discharge (RMD) sample floats were ground in (26 x 32 cm, 66 RPM and 40% solids with 12 kg steel balls of 15-18 mm diameter) a ball mill. The grinding time was varied to obtain the desired mesh of grind.

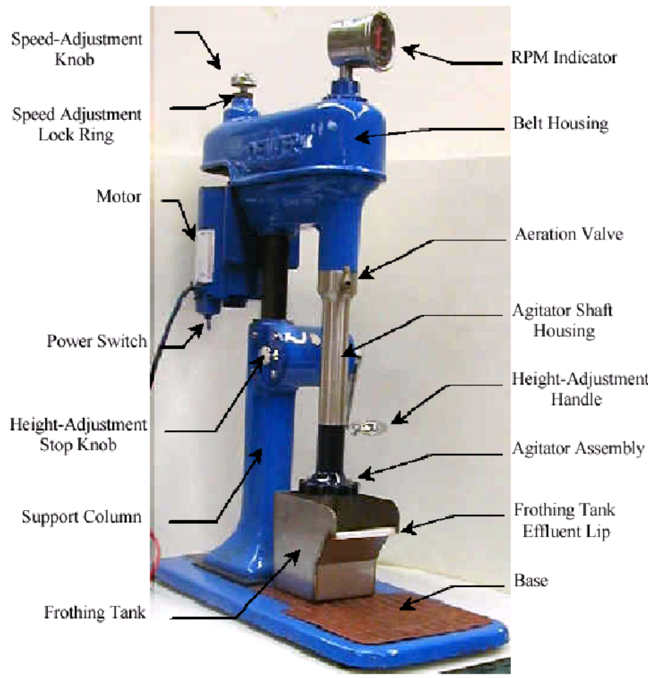

Fig. 1. Denver D12 Laboratory flotation machine

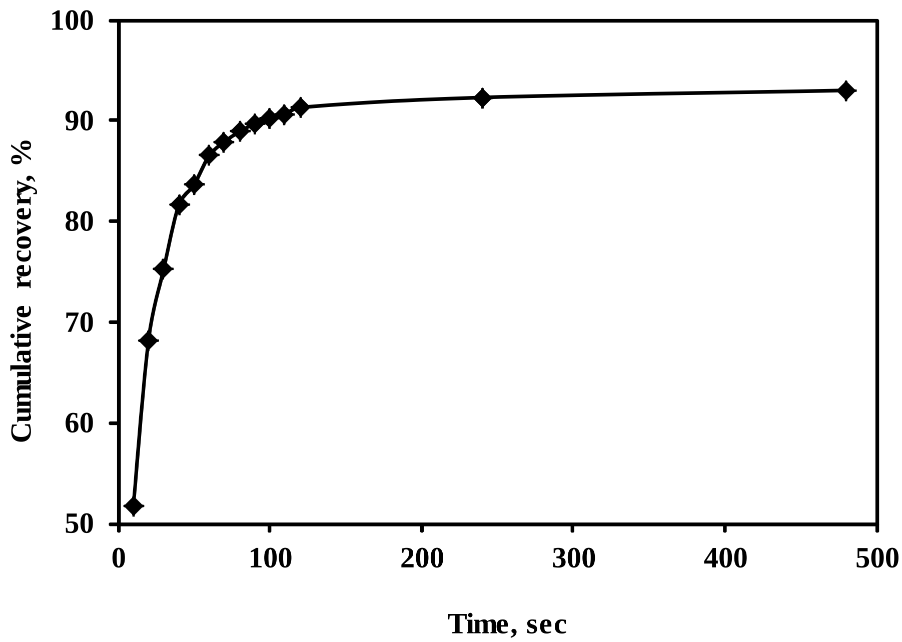

Fig. 2. Time verses cumulative percent recovery

Table 1. Kinetic Floatation Study

Sl. No.	Product	Floatation time, sec.	Wt, % of float concentrate	Assay, %	Distri-bution, %	Cumulative weight, %	Cumu-lative assay, %	Cumu-lative recovery (R), %	Cumulative loss in recovery (100-R)
1	Float – 1	0-10	11.82	65.46	51.7	11.82	65.46	51.7	48.3
2	Float – 2	10-20	3.98	61.63	16.4	15.80	64.50	68.1	31.9
3	Float – 3	20-30	1.96	54.46	7.1	17.76	63.38	75.2	24.8
4	Float – 4	30-40	1.81	53.14	6.4	19.57	62.44	81.6	18.4
5	Float – 5	40-50	0.77	39.11	2.0	20.34	61.55	83.6	16.4
6	Float – 6	50-60	1.10	38.99	2.9	21.44	60.40	86.5	13.5
7	Float – 7	60-70	0.69	30.08	1.4	22.13	59.45	87.9	12.1
8	Float – 8	70-80	0.60	25.96	1.0	22.73	58.57	88.9	11.1
9	Float – 9	80-90	0.59	20.06	0.8	23.32	57.59	89.7	10.3
10	Float –10	90-100	0.35	20.24	0.5	23.67	57.04	90.2	9.8
11	Float –11	100-110	0.42	14.00	0.4	24.09	56.29	90.6	9.4
12	Float –12	110-120	0.73	13.35	0.7	24.82	55.03	91.3	8.7
13	Float –13	120-240	5.10	3.01	1.0	29.92	46.16	92.3	7.7
14	Float –14	240-480	4.90	2.17	0.7	34.82	39.97	93.0	7.0
15	Tailing	-	65.18 100.0	1.60	7.0 100.0	100.0	14.96	100.0	00

Table 2. Particle size distributions of float concentrate and tailings

Mesh size (BSS)	Float concentrate Wt, %	Float concentrate Cumulative Wt, %	Tailings Wt, %	Tailings Cumulative Wt, %
+44	14.24	14.24	15.89	15.89
-44+60	12.27	26.51	09.58	25.47
-60+72	16.09	42.60	15.75	41.22
-72+100	15.07	57.67	14.45	55.67
-100+150	12.55	70.22	13.19	68.86
-150+200	08.45	78.67	08.01	76.87
-200	21.33	100.00	23.13	100.00

Table 3. Conditions of open cycle test

Stages	Cell volume, in litre	RPM	% solids	pH	Reagents	Dosage kg/t	Conditioning time	Float time
	5	1500	20	7.0	Na_2CO_3	1.4	1	-
Rougher x	5	1500	20	8.5	Na_2SiO_3	0.5	5	-
5 times	5	1500	20	8.5	F/C mix	0.1	5	1
	5	1500	15	8.5	-	-	-	-
Scavenger	5	1500	15	8.5	Na_2SiO_3	0.5	1	-
x 5times	5	1500	15	8.5	F/C mix	0.1	5	7
The composite float concentrate ground in 26 x 32 cm ball mill with 12 kg of balls for 20 min grinding.								
Ist cleaner	5	1500	16	8	Na_2CO_3	1.0	1	-
mechanical	5	1500	16	9	Na_2SiO_3	0.5	5	-
cell	5	1500	16	9	F/C mix	0.04	2	2
IInd cleaning in column	2.5	AFR 5 lpm	10	8.5	F/C mix	0.04	5	15

Table 4. Test result of open cycle test on rod mill discharge sample

Product	Wt,%	Assay,%	Distribution,%
Scavenger tails (5 times)	70.0	1.60	7.5
Scavenger float (5 times)	14.0	29.90	28.0
Ist cleaner tails	7.0	31.30	14.6
IInd cleaner tails	1.9	32.25	4.10
IInd cleaner column float	7.1	96.30	45.8

Table 5. Conditions of lock cycle test

Stages	Cell vol. litre	RPM	% solids	pH	Reagents	Dosage kg/t	Condi- tion time min	Float time min
	5	1500	20	7.0	Na_2CO_3	1.4	1	-
Rougher x	5	1500	20	8.5	Na_2SiO_3	0.5	5	-
5 times	5	1500	20	8.5	F/C mix	0.1	5	1
Scavenger	5	1500	15	8.5	-	-	-	-
x5 times	5	1500	15	8.5	Na_2SiO_3	0.5	1	-
	5	1500	15	8.5	F/C mix	0.1	5	7
The composite float concentrate ground in 26 x 32 cm ball mill with 12 kg of balls for 20 min grinding.								
I^st cleaner	5	1500	16	8	Na_2CO_3	1.0	1	-
mechanical	5	1500	16	9	Na_2SiO_3	0.5	5	-
cell	5	1500	16	9	F/C mix	0.04	2	2
II^nd cleaning in column	2.5	AFR 5 lpm	10	8.5	F/C mix	0.04	5	15

Table 6. Final lock cycle test results

Description	% Distribution	% F.C	% Recovery
Float concentrate	13.2	96.24	85.0
Tails	86.8	2.17	15.0
Radicals	% F.C	%V.M	% Ash
	96.24	1.32	2.44

Table 7. Sieve analysis of column float concentrate

Mesh size (BSS)	Weight %	Cumulative weight %
+ 52	6.20	6.20
- 52 + 72	18.98	25.18
-72 + 100	15.44	40.62
- 100 + 200	30.66	71.28
- 200	28.72	100

Table 8. Chemical analysis of ash

Radicals	Percentage
SiO_2	77.15
Al_2O_3	8.86
Fe_2O_3	11.63
CaO	0.82
MgO	0.41
Na_2O	0.26
K_2O	0.15
P_2O_5	0.04
S	0.22

The ground ore pulp from the rod mill discharge of the plant was taken into consideration to simulate the plant conditions. After optimizing the flotation conditions, the floats were ground in a ball mill to achieve the desired MOG. The ground rougher float was subjected to cleaning in the flotation column after conditioning with the reagents. The reagent dosages, column parameters were fixed on no load gas hold-up conditions.

Based on the kinetic studies, an open cycle flotation test was carried out on RMD sample comprising of rougher, scavenger, regrinding of rougher float, first cleaner flotation tests in mechanical cell followed by the IInd cleaning in a laboratory column. The test conditions are given in Table 3. The open cycle test results are given in Table 4. From the table, it can be seen that the open cycle test could yield a graphite concentrate of 96.30% F.C with 45.8% recovery.

4.2. Final lock cycle test :

The previous open cycle test could yield a graphite concentrate of 96.30% F.C with 45.8% recovery. The middling accounted for 46.7% F.C distribution with 31.15% F.C grade necessitating the liberation by grinding followed by recirculation. The above lock cycle test was carried out in 5 cycles, recalculated the ground middling along with the RMD feed.

The ground middlings (scavenger float of 5 times, Ist cleaner tails and IInd cleaner column tails) were filtered 1/5 of the middlings were added along with 1 kg of RMD fresh feed. The 5 times of rougher and scavenger flotation was carried out to get the feed for grinding and subsequent cleaning. The final lock cycle test conditions and test results are given in Tables 5- 8. From the Table 6, it is evident that the percent recovery of final lock cycle test is 85% and the fixed carbon content of float concentrate is 96.24%. The +72 mesh of float concentrate is 25.2% which is meeting the requirement. The complete size and chemical analysis of ash of column flotation concentrate are given in Tables 7-8.

Conclusions

The representative sample collected from the plant in 15 days duration is matching with feed quality of plant. The fixed carbon content of different size fractions indicates that the coarse size contains more value in comparison with fine size. Hence, the liberation of graphite mineral is better and it contains good amount of flaky. The rod mill discharge sample proximate analysis of each size fraction was carried out on dry basis. Kinetic floatation study of MIBC and ethyl alcohol of ratio 90:10 with kerosene 10:90 ratio reagent dosages gives better recovery, assay and gives fast floatation rate. The result indicated that the final concentrate assaying 96.24% F.C, 1.32% V.M, and 2.44% ash. It is observed that the cumulative percent recovery of 91.3% is optimum at 120 seconds. However, 8 minutes of floatation residence time is required to improve the overall cumulative percent recovery to 93%. Hence, it is confirmed that the reagents used in the present study is working effectively. The percent recovery of final lock cycle test is 85% and fixed carbon content of float concentrate is 96.24%. The +72 mesh of float concentrate is 25.2% which is meeting as per the requirement. The results of the laboratory experiments are approaching to the rated capacity of plant.

Acknowledgement

The authors wish to thank to the Chairman and Managing Director, TAMIN, Chennai and Scientists, IMMT, Bhubaneswar and IBM, Bangalore for providing relevant support while preparing the manuscript.

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