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Beneficiation of low grade graphite ore from Multai area, Betul district, M.P.

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ABSTRACT

The low grade graphite ore sample from Multai area, Betul District, M.P. analysed 10% graphite and was found to be cryptocrystalline in nature and intimately associated with silicate gangue. Conventional mechanical cell flotation trials were conducted and various process variables such as frother/collector/depressant concentrations, pH of the pulp etc., were optimised. The best grade concentrate containing 60.1% F.C. was obtained after cleaning the rougher concentrate twice, with a recovery of 72.12%. Further improvement in the grade was possible by only resorting to the bulk oil flotation of the finely ground sample (less than 37 microns). The best grade containing 72.5% F.C. was obtained by bulk oil flotation of the rougher concentrate containing 53.7% F.C., with an overall graphite recovery of 74.6%. The column flotation trials studying process variables such as pulp density, depressant concentration and air flow rates, yielded the best grade of column concentrate containing 64% F.C. with a recovery of 90.88%. After analysing the results obtained by various techniques critically, the bulk oil flotation of the rougher cell concentrate, was found to be the most suitable technique.

INTRODUCTION

Because of the specific properties like refractoriness, good conductor of heat and electricity, greasiness etc., graphite finds important industrial applications. It is used in foundries, refractory and in the manufacture of lubricants, paints, crucibles, electrodes, pencils etc. Generally the naturally occurring graphite is of low grade and as such it is not suitable for industrial applications. Froth flotation is commonly used for the concentration of low grade graphite ore. This paper presents the results on the beneficiation of a low grade graphite ore sample from Multai, M.P., by using mechanical flotation cell, column cell and bulk oil flotation.

MINERALOGICAL AND CHEMICAL CHARACTERISTICS OF ORE SAMPLE

The sample of low grade graphite ore was procured from Multai town, M.P. The sample was available in small and large lumps and also in powder form. The sample was hard, compact and dark steel gray in colour. The microscopic examination carried out at IBM, Nagpur indicated quartz as predominant mineral (about 85%) with minor amounts of graphite (9 to 10%) and muscovite (2 to 3%). Tourmaline, hematite, talc and chlorite were also present in trace amounts (less than 1% each). Most of the graphite was cryptocrystalline in nature, usually present in close association with the silicate gangue. The microscopic examination of the feed sample to minus 300 mesh B.S.S., revealed the presence of some graphite inclusions in silicate gangue. Thus, liberation of graphite was not satisfactory even at a fine size of 300 mesh B.S.S.

The ore sample assayed 10.0% fixed carbon with 84.5% ash, 5.5% volatile matter and 1.36% moisture.

STUDIES WITH MECHANICAL FLOTATION CELL

The mechanical cell flotation tests were conducted in a laboratory Denver type cell. About 500 gms. of representative sample (95% passing through 100 mesh B.S.S.) was fed to the flotation cell, maintaining a pulp density of 14.28% solids. After adding all the flotation reagents and maintaining suitable flotation conditions, the flotation pulp was aerated and the rougher concentrate was collected. This rougher concentrate was then subjected to cleaning. The final concentrate obtained was then suitably collected, filtered in a vacuum filter, dried in an air oven and then weighed accurately. This dried concentrate was thoroughly mixed and a small representative sample (about 5 gms.) was collected using standard sampling procedures, and was subjected to proximate analysis. The tailings were also subjected to proximate analysis after suitable processing.

Effect of frother concentration on graphite flotation

Initially, a few flotation trials were conducted to study the effect of frother (pine oil) concentration on the grade and recovery of graphite. The results of these trials are indicated in the Table 1. It is clearly seen from the Table 1 that, the grade of concentrate increase continuously upto a frother concentration of 0.15 kg/ton beyond which the increase is very marginal. Hence, for further flotation trials, the frother concentration was kept fixed at 0.15 kg/ton.

Table 1 : Effect of frother concentration

Frother kg/t	Products	Weight (%)	Assay F.C. (%)	Distribution F.C. (%)
0.100	Conc.	18.63	16.40	31.02
	Tails	81.37	8.35	68.98
	Head (Cal).	100.00	9.85	100.00
0.125	Conc.	17.96	18.90	34.46
	Tails	82.04	7.87	65.54
	Head (Cal).	100.00	9.87	100.00
0.150	Conc.	17.06	21.80	37.95
	Tails	82.94	7.33	62.05
	Head (Cal).	100.00	9.80	100.00
0.175	Conc.	17.06	22.00	38.10
	Tails	82.94	7.35	61.90
	Head (Cal).	100.00	9.85	100.00
0.200	Conc.	16.97	22.10	38.27
	Tails	83.03	7.29	61.73
	Head (Cal).	100.00	9.80	100.00

Effect of pH on graphite flotation

The flotation trials were conducted at acidic, neutral and alkaline pH. The results are shown in the Table 2. It is clear from the Table 2 that, the grade of concentrate is showing increasing trend upto pH value of 10, beyond which there is no significant improvement. Hence, for further trials, the pH value of 10 was kept fixed.

Effect of depressant on graphite flotation

Various types of depressants such as gum arabic, quebracho, gelatin, tannic acid, sodium silicate were used in a few preliminary flotation tests. It is observed that Sodium Silicate was the most effective depressant. Hence, next set of trials were performed using various concentrations of sodium silicate. The results obtained are tabulated in the Table 3. It is clear from the Table 3 that the best grade concentrate is obtained at a depressant concentration of 2.5 kg/ton. Hence, for further trials the depressant concentration of 2.5 kg/ton was kept fixed.

Effect of collector on graphite flotation

Various types of non-ionising collectors such as kerosene, diesel, petrol etc.,

Table 2 : Flotation results at different pH values

pH	Products	Weight (%)	Assay F. C. (%)	Distribution F. C. (%)
7	Conc.	16.83	22.00	37.78
	Tails	83.17	7.33	62.22
	Head (Cal).	100.00	9.85	100.00
8	Conc.	16.63	24.80	41.87
	Tails	83.37	6.87	58.13
	Head (Cal).	100.00	9.85	100.00
9	Conc.	16.92	27.10	48.52
	Tails	83.08	5.86	51.48
	Head (Cal).	100.00	9.45	100.00
10	Conc.	17.11	28.02	50.47
	Tails	82.89	5.68	49.53
	Head (Cal).	100.00	9.85	100.00
11	Conc.	17.06	28.14	50.53
	Tails	82.94	5.67	49.47
	Head (Cal).	100.00	9.85	100.00

Table 3 : Flotation results at varying dosage of sodium silicate

Sodium Silicate (kg/t)	Products	Weight (%)	Assay F. C. (%)	Distribution F. C. (%)
1.0	Conc.	17.05	29.80	51.85
	Tails	82.95	5.69	48.15
	Head (Cal).	100.00	9.80	100.00
1.5	Conc.	17.31	32.10	56.41
	Tails	82.69	5.19	43.59
	Head (Cal).	100.00	9.85	100.00
2.0	Conc.	16.65	34.93	59.35
	Tailings	83.35	4.78	40.65
	Head (Cal).	100.00	9.80	100.00
2.5	Conc.	17.04	35.50	61.10
	Tails	82.96	4.64	38.90
	Head (Cal).	100.00	9.90	100.00
3.0	Conc.	17.16	35.70	61.57
	Tails	82.84	4.62	38.43
	Head (Cal).	100.00	9.95	100.00
3.5	Conc.	17.14	35.80	61.67
	Tails	82.86	4.60	38.33
	Head (Cal).	100.00	9.95	100.00

were used in the preliminary trials. The kerosene was observed to be the best collector. Hence, few flotation trials were conducted using varying concentrations of kerosene. The results of these trials are indicated in the Table 4. It is obvious from Table 4 that, the concentrate grade is increasing marginally beyond a kerosene oil concentration of 0.144 kg/t. And hence, for further trials, this was kept fixed.

Table 4 : Effect of kerosene oil addition

Kerosene oil (kg/t)	Products	Weight (%)	Assay F. C. (%)	Distribution F. C. (%)
0.048	Conc.	16.99	36.40	62.47
	Tails	83.01	4.48	37.53
	Head (Cal).	100.00	9.90	100.00
0.096	Conc.	16.65	37.90	64.06
	Tails	83.35	4.25	35.94
	Head (Cal).	100.00	9.85	100.00
0.144	Conc.	16.20	40.10	65.29
	Tails	83.80	4.12	34.71
	Head (Cal).	100.00	9.95	100.00
0.192	Conc.	16.11	40.50	66.24
	Tails	83.89	3.96	33.76
	Head (Cal).	100.00	9.85	100.00
0.240	Conc.	16.11	40.70	66.91
	Tails	83.89	3.87	33.09
	Head (Cal).	100.00	9.80	100.00

Effect of granulometry on graphite flotation

Upto this stage of experimentation, the particle size for flotation was kept constant i.e., 95% passing through 100 mesh B.S.S. A few flotation trials were conducted at finer sizes of the feed. The results are given in the Table 5. It is seen from Table 5 that, the concentrate grade sharply increases with finer sizes, due to the greater amount of liberation of graphite. The best grade concentrate can be obtained using a particle size of 95% passing through 300 mesh B.S.S. This was kept fixed for further trials.

Improvement in concentrate grade by clearing flotation

The flotation tests were carried out using optimum flotation reagents and conditions for rougher flotation followed by several number of cleanings. 0.025, 0.25 and 0.024 kg/t of frother, depressant and kerosene oil were added as additional

Table 5 : Effect of granulometry on flotation

Particle size	Products	Weight (%)	Assay F. C. (%)	Distribution F. C. (%)
95% - 100	Conc.	16.20	40.10	65.29
	Tails	83.80	4.12	34.71
	Head (Cal).	100.00	9.95	100.00
95% - 150	Conc.	15.22	48.80	72.31
	Tails	84.78	3.22	27.69
	Head (Cal).	100.00	9.85	100.00
95% - 200	Conc.	15.20	49.90	76.61
	Tails	84.80	2.73	23.39
	Head (Cal).	100.00	9.90	100.0
95% - 300	Conc.	14.93	53.70	81.40
	Tails	85.07	2.15	18.60
	Head (Cal).	100.00	9.85	100.00

dosage during each cleaning flotation. The results obtained are shown in the Table 6. It is clear from the Table 6 that, the best grade of graphite concentrate containing 60.1% F.C. can be obtained with a recovery of 72.12%.

Table 6 : Results of multi-stage cleaning flotation

Products		Weight (%)	Assay F. C. (%)	Distribution F. C. (%)
Rougher flotation				
	R. Conc.	14.93	53.70	80.18
	Tails	85.07	2.33	19.82
	Head (Cal.)	100.00	10.00	100.00
One stage cleaning flotation				
	Cl. Conc. I	12.97	57.70	74.84
	Cl. Tails I	2.16	27.96	6.04
	Pr. Tails	84.87	2.25	19.12
	Head (Cal.)	100.00	10.00	100.00
Two stage cleaning flotation				
	Cl. Conc. II	12.00	60.10	72.12
	Cl. Tails II	1.20	30.92	3.71
	Cl. Tails I	2.05	26.90	5.52
	Pr. Tails.	84.75	2.20	18.65
	Head (Cal.)	100.00	10.00	100.00

BULK-OIL FLOTATION

A representative feed sample, weighing 100 gms. (ground to –37 microns) was mixed with 100 ml. of kerosene oil in a beaker and thoroughly stirred for five minutes, with a view to wetting all the graphite particles with kerosene oil. The mixture (suspension) was then transferred to a laboratory flotation cell (three litres capacity) along with two litres of distilled water and stirred for five minutes. The rotor of the flotation cell was then removed, cleaned with an additional distilled water to remove adhering graphite particles and the volume was made up to then top of the overflow lip of the cell and the contents were allowed to settle for various intervals of time. After suitable settling time, all the graphite floating at the top was skimmed off completely along with some suspended water. This suspension was then transferred into one litre capacity separating funnel and cleaned with distilled water to remove any adhering mineral particles. The graphite floating in kerosene layer was removed, filtered, dried and weighed accurately and analysed for its fixed carbon content. The loss of kerosene in the process was also determined and was observed to be 25 ml. per batch of 100 gms. sample. The results obtained are shown in the Table 7.

Table 7 : Bulk-oil-flotation trials at different pH

pH	Settling Time, hrs.	Products	Weight (%)	Assay F. C. (%)	Distribution F. C. (%)
9.0	0.5	Conc.	13.90	63.20	87.85
		Tails	86.10	1.41	12.15
		Head (Cal.)	100.00	10.00	100.00
9.0	45	Conc.	14.00	64.60	90.44
		Tails	86.00	1.11	9.56
		Head (Cal.)	100.00	10.00	100.00
9.0	1	Conc.	14.20	65.80	93.44
		Tails	8.80	0.77	6.56
		Head (Cal.)	100.00	10.00	100.00
9.0	18	Conc.	14.00	66.90	93.66
		Tails	86.00	0.74	6.34
		Head (Cal.)	100.00	10.00	100.00
10.0	0.5	Conc.	14.00	64.80	90.72
		Tails	86.00	1.08	9.28
		Head (Cal.)	100.00	10.00	100.00
10.0	45	Conc.	14.19	65.70	93.23
		Tails	85.81	0.79	6.77
		Head (Cal.)	100.00	10.00	100.00
10.0	1.0	Conc.	14.20	66.80	94.86
		Tails	85.80	0.60	5.14
		Head (Cal.)	100.00	10.00	100.00
10.0	18	Conc.	14.00	68.00	95.20
		Tails	86.00	0.56	4.80
		Head (Cal.)	100.00	10.00	100.00

It is obvious from the Table 7 that, the best grade of concentrates containing 66.9% and 68% F.C. can be obtained from the ore sample by using bulk oil flotation technique at pH 9 and 10 with a very high recovery of 93.66% and 95.2% respectively by providing a settling time of 18 hours (overnight) to the suspension.

COLUMN FLOTATION

For the present investigation, the glass flotation column of inner diameter of 6 cms., was specially fabricated as indicated in the line diagram Fig. 1.

The compressed air was blown into the column through the bottom inlet F and a preconditioned feed-slurry was added from the feed-inlet C. After few seconds, the froth column raised and reached to the froth outlet B. Wash-water was

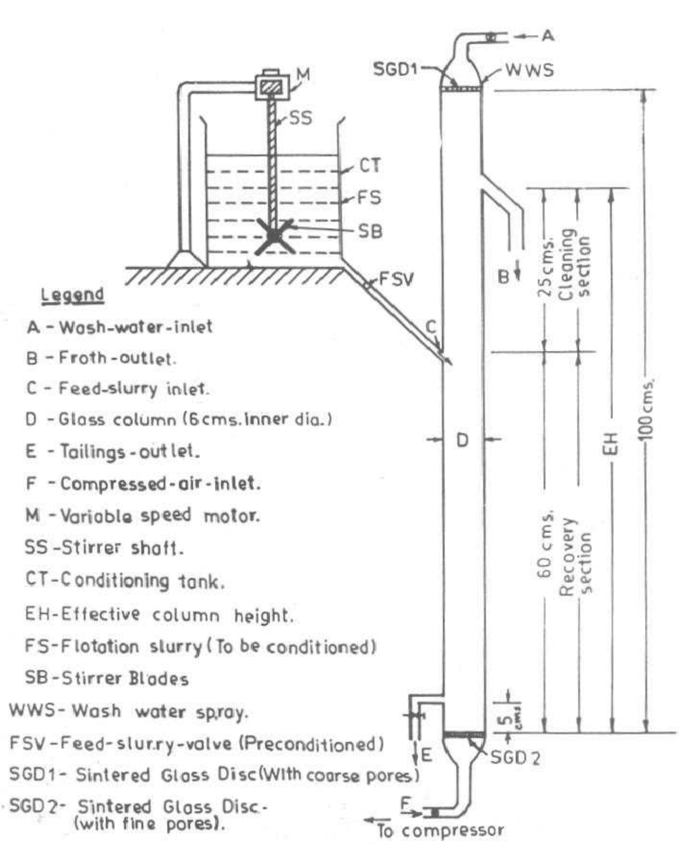

Fig. 2 : Experimental set-up for column flotation trials.

continuously sprayed from the inlet A and the cleaned froth was collected in a suitable container from the froth outlet B. After collecting the mineralised froth completely the waste slurry (tailings) containing impurities, could be withdrawn from the tailings outlet E. Again a fresh batch of feed-slurry was introduced and the whole procedure was repeated. The collected froth was then filtered, dried in an air-oven at 105°C and was weighed accurately and was subjected to proximate analysis. Similar procedure was adopted for the column tailings.

Effect of pulp density on column flotation

The column flotation trials (batch type) were carried out at different values of pulp density (% solids) levels using flotation reagents and conditions optimised by mechanical cell flotation trials. The results are indicated in the Table 8.

Table 8 : Effect of pulp density on column flotation of graphite

Pulp density, (%)	Products	Weight (%)	Assay F. C. (%)	Distribution F. C. (%)
14.28	Conc.	15.28	54.24	83.30
	Tails	84.72	1.96	16.70
	Head (Cal).	100.00	9.95	100.00
15.39	Conc.	14.95	57.76	86.79
	Tails	85.05	1.545	13.21
	Head (Cal).	100.00	9.95	100.00
16.67	Conc.	14.65	59.12	87.05
	Tails	85.35	1.51	12.95
	Head (Cal).	100.00	9.95	100.00
20.00	Conc.	14.47	59.89	87.10
	Tails	85.53	1.50	12.90
	Head (Cal).	100.00	9.95	100.00
25.00	Conc.	14.45	59.98	87.11
	Tails	85.55	1.499	12.89
	Head (Cal).	100.00	9.95	100.00

It is clear that grade as well as recovery values for column concentrates are found to be higher than the corresponding values obtained in cell flotation trials. However, the column flotation results fail to compete with those obtained Bulk-oil flotation trials, which is mainly due to better liberation of graphite from the gangue for the feed sample sizes (100% – 37 microns) used in Bulk-oil-flotation tests.

Effect of depressant (lactic acid) concentration

To prevent the flotation of mica, trials were conducted using varying amounts of lactic acid, improving the grade and recovery values as shown in Table 9.

Table 9: Eeffect of Lactic acid on column flotation of graphite

Lactic acid dosage, (Kg/t)	Products	Weight (%)	Fixed F.C. (%)	Distribution F. C. (%)
0.50	Conc.	14.21	61.58	87.51
	Tails	85.79	1.455	12.49
	Head (Cal).	100.00	10.00	100.00
0.75	Conc.	14.09	62.20	87.64
	Tails	85.91	1.44	12.36
	Head (Cal).	100.00	10.00	100.00
1.00	Conc.	14.05	62.50	87.81
	Tails	85.95	1.42	12.19
	Head (Cal).	100.00	10.00	100.00
1.25	Conc.	14.05	62.55	87.88
	Tails	85.95	1.41	12.12
	Head (Cal).	100.00	10.00	100.00

It is obvious from Table 9 that, an optimum value of 1 kg/ton of lactic acid yields the best concentrate grade of 62.5% F.C., with a recovery of 87.81% which is kept constant for further trials.

Table 10: Eeffect of air flow rate on column flotation of graphite

Air flow rate, lmp	Products	Weight (%)	Assay F. C. (%)	Distribution F. C. (%)
1.75	Conc.	14.00	62.55	87.57
	Tails	86.00	1.445	12.43
	Head (Cal).	100.00	10.00	100.00
2.00	Conc.	14.12	62.95	88.89
	Tails	85.88	1.294	11.11
	Head (Cal).	100.00	10.00	100.00
2.25	Conc.	14.15	63.15	89.36
	Tails	85.85	1.24	10.64
	Head (Cal).	100.00	10.00	100.00
2.50	Conc.	14.18	63.29	89.75
	Tails	85.82	1.194	10.25
	Head (Cal).	100.00	10.00	100.00
2.50	Conc.	14.20	63.85	90.67
	Tails	85.80	1.088	9.33
	Head (Cal).	100.00	10.00	100.00
2.50	Conc.	14.20	64.00	90.88
	Tails	85.80	1.06	9.12
	Head (Cal).	100.00	10.00	100.00

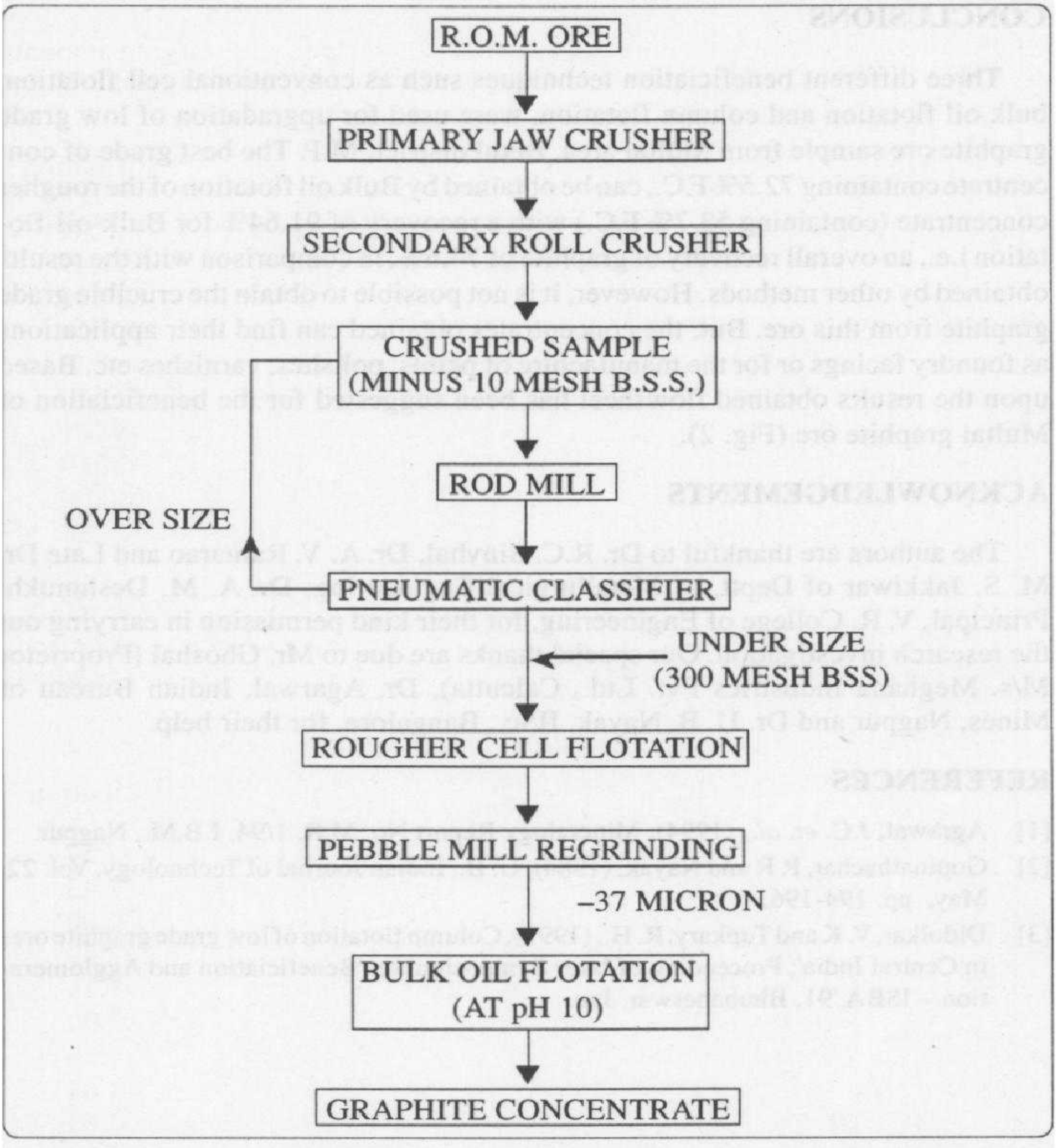

Fig. 2 : Suggested flow sheet for the beneficiation of Multai graphite ore.

Effect of air-flow-rate

The grade of the concentrate could be improved further by carrying out trials with varying air flow rates, blown through bottom-inlet F. The results are shown in Table 10. It is clear from Table 10 that, the best grade of concentrate with 64% F.C. with a recovery of 90.88%. However, the results are inferior to those obtained in Bulk-oil-flotation trials, due to insufficient liberation.

CONCLUSIONS

Three different beneficiation techniques such as conventional cell flotation, bulk oil flotation and column flotation, were used for upgradation of low grade graphite ore sample from Multai area, Betul district, M.P. The best grade of concentrate containing 72.5% F.C., can be obtained by Bulk oil flotation of the rougher concentrate (containing 53.7% F.C.) with a recovery of 91.64% for Bulk-oil-flotation i.e., an overall recovery of graphite of 74.6%, in comparison with the results obtained by other methods. However, it is not possible to obtain the crucible grade graphite from this ore. But, the concentrates obtained can find their applications as foundry facings or for the manufacture of paints, polishes, varnishes etc. Based upon the results obtained flowsheet has been suggested for the beneficiation of Multai graphite ore (Fig. 2).

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