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BENEFICIATION OF TARLI-DOMEL (AZAD KASHMIR) GRAPHITE ORE

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A study was undertaken to beneficiate a low grade graphite ore from Tarli Domel area of Azad Kashmir. The ore, initially containing 8-9% graphitic carbon, was upgraded by flotation to a concentrate assaying 86-87% graphitic carbon with a recovery of 97%. This paper deals with the optimization of various parameters for achieving the said grade and recovery.

Key words: Graphite, Beneficiation, Flotation, Regrinding.

Introduction

Graphite is the first mineral that was beneficiated by flotation [1]. The mineral being naturally hydrophobic [2], its beneficiation by flotation is easy and does not require large quantities of reagents. Recent studies show the use of selective flocculation in conjunction with flotation for recovering graphite.

Rao [3] studied the selective flocculation of graphite ore. The flocculated ore was subjected to flotation using Na-silicate as the depressor and pine and kerosine oils as the frother. Where kerosine was used, it improved the recovery but the grade was lowered. Sodium silicate produced highly stable suspension of the selectively dispersed mixed flocs. This technique of flotation considerably improved the grade with slight decrease in the recovery. Petukhv and Musavirov [4] used diesel fuel as conditioning agent during flotation of coal and graphite. The recovery increased, the flotation selectivity improved and consumption of reagents for conditioning decreased by using octa alkyl-1-1,3-dio -2-silacylohexane as the frothing agent. Chivhevsku *et al.* [5] used a still residue (SR) resulting from the 2-ethyl hexanal manufacture as the frothing agent instead of kerosine or T-80 oil during the flotation of graphite. The optimum consumption of S.R. was 176 g/ton at 88.67% graphite recovery. The use of S.R. as frothing agent was recommended to improve the economic indices of graphite flotation. The effect of waste methanol liquor on flotation of some non-metallic minerals containing long-chain alcohols, cycloalkanes, phenols and aldehydes was used by Wang Jim [6]. The flotation efficiency with oil collecting agents was increased by mixing with the waste methanol liquor.

Graphite has been shown to occur in various areas of Pakistan [7]. The deposits in Azad Kashmir are located at Nauseri, Reshian, Mohri Kel, Patla Pani, Bagnoo and Tarli-Domel. The extent of deposits at these places has not yet been established firmly. However, according to published data about 1.5 and 3 million tons of graphite ore are reported to be present at Mohri Kel (Azad Kashmir) and Malakand regions (NWFP) respectively [8,9].

Mineralogy. The mineralogical studies on the Tarli Domel ore indicated the presence of distinct slaty cleavage and schistosity. The host rocks are generally foliated and bear gangue minerals such as quartz, mica, biotite, calcite and chlorite. The graphite was found to be generally disseminated as fine to medium sized grains (0.05-0.2 mm). The graphitic carbon was associated with minor quantities of amorphous carbon and was found to be finely disseminated in micacious matrix. The grain size in nine random pieces varied from 150-53 microns. Flakes larger than 0.1 mm were rare. Libration studies revealed that 80% of graphite particles were liberated at a grind size of 105-76 microns.

Experimental

Material. Graphite ore from Tarli Domel area of Azad Kashmir was received through the courtesy of Azad Kashmir Mineral and Industrial Development Corporation (AKMIDC). The run-of-mine ore consisted of pieces as large as 15-25 cm across and as fine as 150 microns. Chemical analysis of the ore sample, prepared by primary and secondary crushing and coning-quartering, is indicated in Table 1.

Equipment. Laboratory jaw crusher (10x20 cm) and roll (25x15 cm) crushers were used for primary and secondary

Table 1. Chemical Analysis of Ore Sample.

Constituents	Weight%
Moisture	0.18
Graphitic carbon	9.42
Amorphous carbon	0.57
Fe_2O_3	12.42
Al_2O_3	5.08
SiO_2	64.99
CaO	6.67
MgO	0.55

crushing. Larger pieces of ore were first reduced in size by hammer. Grinding of rolls product was carried out in a Denver Laboratory rod mill (17.8 x 35.5 cm).

Flotation tests were conducted in a Denver D-12 flotation cell. This machine has been provided with a tachometer and a speed control device. The impeller can be changed and cell tank may be replaced depending upon the pulp volume. Air control is through a completely sealed air tap.

Size analysis were carried out using B.S.S. 410 sieves and a Laser Particle Size Analyser. The later equipment works in the size range of 0.9-175 microns.

Sample preparation. The run-of-mine ore was fed to the jaw crusher manually, the product opening having been set at 1.25 cm. The jaw product was subjected to secondary crushing in a roll crusher. The rolls product, which constituted the rod mill feed, had the granulometry as shown in Table 2.

TABLE 2. SIZE DISTRIBUTION OF ROLLS PRODUCT (FEED TO THE GRINDING MILL).

Size (microns)	Cumulative weight % Passing	Retained
3340	81.35	18.65
2050	62.38	37.62
1000	46.15	53.85
700	39.61	60.39
500	34.34	65.66
353	29.49	70.51
252	23.83	76.17
177	22.88	77.12
152	19.50	80.50
105	15.92	84.08
76	14.66	85.34
65	13.40	86.60
53	7.08	92.92
35	6.54	93.46

The rolls product was fed to the rod mill where it was ground for different time intervals and analysed for size distribution as shown in Table 3. The mill product for 45 mins. grinding, which was later on found to be the optimum, was used for subsequent flotation studies.

Flotation test work. In order to achieve a reasonable grade at a favourable recovery, flotation tests were undertaken to optimise various variables, like the grind time, pulp density, reagents (conditioning, flotation), regrinding, etc. A brief description of each is given below.

Effect of grind time. A set of four tests was carried out to determine the optimum time of primary grinding. One kg of ore was ground for durations of 30, 45, 60 and 90 mins. Flotations were carried out using sodium silicate 0.14 kg/t and kerosine oil 3 ml as collector at a pulp density of 22% and regrinding time of 45 mins. The results of these tests are presented in Table 4.

Effect of secondry grinding. In view of the results of Table 4, a primary grind time of 45 mins. (100% passing 105 microns) was considered to be optimum. But in order to improve the grade of the concentrate a set of experiments was carried out to observe the effect of regrinding the rougher concentrate containing 37.31% graphitic carbon (secondary grinding). In these tests the sodium silicate dosage was increased to 0.45 kg/t while the quantity of the collector was kept the same (3 ml kerosine oil). The results of this set of experiments are presented in Table 5. The particle size distribution of 45 mins. regrinding (100% passing 88 microns) is presented in Table 6.

Effect of conditioning agent. Sodium silicate was used as conditioning agent. It was added to the flotation pulp in the form of a solution prepared from the solid substance of commercial purity. The grind size were kept at 100% passing 105 and 88 microns respectively for primary and secondary

TABLE 3. SIZE DISTRIBUTION OF GROUND MILL PRODUCT.

Size (microns)	15mins. grinding Passing	15mins. grinding Retained	30mins. grinding Passing	30mins. grinding Retained	45mins. grinding Passing	45mins. grinding Retained	60mins. grinding Passing	60mins. grinding Retained	90mins. grinding Passing	90mins. grinding Retained
125	100.00	–	100.00	–	–	–	–	–	–	–
105	98.46	1.54	100.00	–	100.00	–	100.00	–	100.00	–
76	90.00	10.00	96.00	4.00	98.45	1.55	98.65	1.35	99.79	0.21
65	84.00	6.00	93.50	6.50	94.52	5.48	95.45	4.55	97.48	2.52
53	72.45	27.55	87.20	2.80	88.20	11.80	92.85	7.15	94.89	5.11
35	55.07	44.93	74.51	25.49	75.00	25.00	81.76	18.24	85.50	14.50
21	35.35	64.65	52.25	47.75	53.53	46.47	61.22	38.78	62.27	32.73
15	26.54	73.46	40.35	59.65	41.82	59.18	48.53	51.47	54.39	45.61
9	17.40	82.60	27.37	72.63	28.78	71.22	33.73	66.27	38.06	61.94
5	10.39	89.61	16.99	83.01	18.08	82.92	21.40	78.60	24.08	75.92
3	6.68	93.32	11.20	88.80	11.97	88.03	14.26	85.74	16.06	83.94
2	4.74	95.26	8.06	91.94	8.66	91.34	10.31	89.69	11.55	88.45
1.5	3.03	96.97	5.22	94.78	5.64	94.36	6.69	93.31	7.42	92.58
0.9	1.32	98.68	2.30	97.70	2.50	97.50	2.95	97.05	3.23	96.77

TABLE 4. EFFECT OF PRIMARY GRINDING TIME.

Grind time (min.)	Grade % graphitic C: Rougher concentrate	First cleaner concentrate	Second cleaner concentrate	Recovery % graphitic carbon	Remarks
30	30.81	46.38	53.10	97.0	K. oil in mill
45	37.31	45.52	59.82	97.0	– do –
60	51.00	64.24	68.70	96.7	K. oil in mill
90	47.81	65.90	72.34	97.0	– do –

TABLE 5. EFFECT OF SECONDARY GRINDING TIME.

Regrinding time (min.)	Grade% graphitic C: First cleaner concentrate	Second cleaner concentrate	Recovery % graphitic C
15	69.08	76.10	97
30	70.50	83.26	97
45	79.60	86.58	97
60	79.87	86.90	97

TABLE 6. SIZE DISTRIBUTION OF REGROUND MATERIAL (45 MINUTES SECONDARY GRINDING).

Size microns	Cumulative weight percent: Passing	Retained
88	100.00	–
76	99.62	00.38
65	98.95	1.05
53	96.18	3.82
35	88.77	11.23
21	69.69	30.31
15	56.39	43.61
9	39.15	60.85
5	23.92	76.08
3	11.18	88.82
2	3.68	96.32
1.5	0.10	99.90
0.9	0.00	100.00

TABLE 7. EFFECT OF Na_2SiO_3.

Quantity of Na_2SiO_3 Lb/t	Grade % Graphitic C: Rougher concentrate	Ist cleaner concentrate	2nd cleaner concentrate	Recovery % graphitic C
0.5	34.68	68.10	81.13	97
1.0	37.31	79.60	86.58	97
1.5	34.58	79.17	83.07	97

TABLE 8. EFFECT OF PULP DENSITY.

Pulp density (% solids)	Grade % Graphitic C: Rougher concentrate	Second cleaner concentrate	Recovery % graphitic C
22	37.31	86.58	97.00
30	34.54	82.09	97.70
35	29.87	79.42	98.12
45	26.43	78.00	98.30

grinding. The collector was the same (3 ml kerosine oil). Other flotation conditions like pulp density, further dosage were kept constant. The results of these tests are given in Table 7.

Effect of pulp density. The pulp density of the flotation pulp was varied from 22-45% keeping the other variables at constant values of grind size at 100% passing 105 and 88 microns respectively for primary and secondary grindings, 0.45% kg/t Na_2SiO_3, 3 ml/kg kerosine oil, etc. The results are given in Table 8.

Conclusion

The results of the study have shown that it is possible to concentrate the low grade graphite ore from Tarli Domel (Azad Kashmir) by flotation technique.

The study has made it possible to upgrade the ore to a concentrate containing 86% of graphitic carbon with a recovery of 97% at a pulp density of 22% solids, grind size at 100% passing 105 and 88 microns respectively for primary and secondary grinding and 0.45 kg/t of sodium silicate as the conditioning agent at a natural pH.

It has been observed that significant improvement in grade from 59.82-86.50% graphite carbon is achieved by regrinding the rougher concentrate (secondary grinding) to a grind size of 100% passing 88 microns. Table 4 has clearly indicated that by increasing the primary grinding time from 45 mins. to even 90 mins. It is only possible to achieve enrichment in grade from 53.10-72.34% graphite carbon at a fairly high grinding cost.

A comparison of the particle size distribution of the ground mill product and the rougher concentrate (Table 3 and 4) has clearly shown that the locked gangue particles have been released as a result of the secondary grinding and subsequently removed during the processing by the flotation technique, thereby resulting in the significant increase in the grade of the concentrate.

It has also been observed that the quantity of sodium silicate used as conditioning agent is very critical in determining the grade of the final concentrate (Table 7). Similarly the effect of the pulp density on the grade and recovery is very pronounced. Thus it is clear from Table 8 that with an increase from 22-45% in pulp density, the grade of the final concentrate falls from 86.58-78% graphitic carbon while the recovery increases from 97%-98.30% respectively.

Product quality and its uses. The graphite concentrate produced as a result of this study may be used as foundry facing material. In addition, this material may be purified further to obtain products suitable for use in the manufacture of lead pencils, carbon brushes, graphite electrodes, porous bearings, lubricants, etc.

At present, all the graphite and graphite based products are being imported. The grade of the concentrate produced in this study is comparable to the imported material for foundry use and may be exported in international market.

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References

1. N. L. Weiss, (ed.) *Mineral Processing Handbook* (Soc. of Min. Eng., AIME, 1985), Vol. 2, Sec. 28-2.
2. N. L. Weiss, ed., *Mineral Processing Handbook* (Soc. of Min. Eng. AIME, 1985), Vol. 1, Sec. 5-48.
3. V. Malleswara Rao, Proc. Symp. India (1981), Edited by Kamrath, V.A. Dep. Energy, Bombay, India (1983), pp. 265-70.
4. V. M. Petukhv, R. S. Musavirov, D. L. Rakhmankulov and T. V. Mikhailova (Magnito-gorsk Mining-Metallurgical Institute, Ufa Petroleum Inst.), USSR, SUI, 138, 190 (CI.B03D1/02) (1985).
5. V. B. Chivhevsku, L. G. Sarvinchuk, L. I. Belykh, A. A. Evstignee va, G. M. Smirnov, V. A. Glazkov and V. K. Kalomin (Magnitogarsk Gorno-Metal Inst., Magnitogarsk, USSR), Povysh. Eff. Tekhnol. Proiz Vod. Kach Nerudn, Nemetullerudn. Mater, 93-6 (RUSS) (1983).
6. Jim Wang (Shamgdong Call. Archit Mater. Peap. Republic China) Feijinshukuang (3) 22-5 (Ch) (1986).
7. S. T. Ali, Mineral Deposits and Showings in the Northern Part of the West Pakistan, GSP Information Release No. 2, March (1959), pp. 23, 31.
8. AKMIDC Report, Muzaffarabad and Engineers Combine Ltd., Lahore.
9. S. H. Faruqi, Report on Graphite Deposits of Azad Kashmir and their Development, Karachi, Dec. (1969).