Technology Section

Pakistan J. Sci. Ind. Res., Vol. 17, No. 6, December 1974

BENEFICIATION OF TIRAH GRAPHITE

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(Received October 13, 1973; revised October 14, 1974)

Abstract. Screening and flotation tests on the beneficiation of the Tirah graphite have been conducted for its commercial utilization. The graphite contains 28–30% fixed carbon and can be upgraded by flotation up to 60% with a recovery of 88%. The conditions of the beneficiation of Tirah graphite by screening and flotation techniques have been set. The product obtained can be utilized in foundry, paints and in the manufacture of refractory articles.

Very little has been done in Pakistan on the beneficiation and utilization of the indigenous graphite. The study is conducted on the upgrading of graphite for its possible commercial utilization.

Occurrence of numerous graphite deposits in NWFP region of Pakistan dates back to the post-independence period.[1,2] Although some of these deposits are individually very large, but their economic feasibility is handicapped by their low-contents of graphitic carbon which usually ranges from 5 to 15% in the deposit. Recently some samples of graphite, brought from Tirah (Khyber Agency) have created special interest, due to their high fixed carbon-contents as compared to the samples received from other areas. A detailed investigation for their beneficiation was, therefore, undertaken.

The Tirah graphite deposit, located in Spinkai area, is present in igneous rocks which are composed of medium grained-granite and pegmatite intruded by quartz-veins. Its mineralization was regarded by some geologists as the result of hydrothermal or gaseous emanations of the granite. The graphite on the whole roughly constitutes 10–50% of the total ore body. It is usually concentrated in joints and fractures of the igneous rocks where it shows as high as 48% of fixed carbon.*

The content of the fixed carbon in the graphite varies from 28 to 50%. The nature of the ore and the amount of fixed carbon has potential for commercial exploitation and utilization in the various indigenous industries in the country. At present Pakistan depends largely on the imported graphite for its consumption mainly in foundry, dry cell, pencil and crucible manufacturing industries. Some of the local graphite is, however, being utilized by paint industries and foundries.

Concentration techniques for graphite depend upon the nature and the texture of the ore such as the locking trend of minerals present in it. The flotation technique, has proved to be the most promising.

* Fixed carbon may be regarded as graphitic carbon for all practical purposes.

Physical Characteristics. The lustrous graphite is steel grey in colour and granular nature, the grains are subdivided into more or less triangular prisms. Grains are generally thick and tough and hence undergo little attrition during handling and normal physical testing. Most of the visible grains pass through 22 mesh screen.

The graphite can be divided into two main grades: grade (1) Flakes that range between 5–40 mesh; and grade (2) Dust which is less than 40 mesh.

The composition of both these grades are shown in Table 1.

Table 1. Composition of Grades 1 and 2 Ore.

Sample	Fixed carbon (%)	Volatile matter (%)	Moisture (%)	Ash (%)
Flakes grade (1)	48.18	7.13	1.75	42.94
Dust grade (2)	29.74	8.43	2.25	59.58

Experimental

Apparatus. (i) Sieve set, British Standard Sieve (B.S.S.); (ii) grinding mills: (a) Roller grinder Wedag floor type No. 572,620, and (b) Batch ball mill type LE 101, No. 275/1960, Budapest; (iii) flotation machine Wedag, Karl Kolb Laboratory type No. 573,500, and (iv) tube furnace, for fixed carbon estimation, Gallenkamp, Laboratory type (1 in dia).

Chemicals. All the chemicals employed in this investigation were laboratory reagent grade chemicals of E. Merck or B.D.H.

Procedure. Different sets of experiments were carried out on both these grades to find out the best conditions for beneficiation. Due to the flaky nature of grade (1) ore, it was subjected to sieve analysis[3] in order to separate the flakes containing high graphitic carbon from the gangue. The ore was first crushed and ground in a laboratory roller grinder for different time periods prior to sieve analysis. The ground ore was sieved using different mesh-size screens. The sieved portions were weighed and analysed for their fixed carbon contents. The data obtained from experiments on the grindability and sieve analysis of first grade graphite ore for different time intervals are summarized in Tables 2 and 3.

For grade (2) ore with graphitic carbon content of 29.79% flotation method was tried for its beneficiation. The ore was first ground in a laboratory batch ball mill, 150g ore are transferred to the flotation cell of flotation machine. Conditioning period prior to flotation was 5 min for each sample. The slurry was made with distilled water and terpineol was used both as collector and frother. The speed of the machine was set at 1500 rev/min and the frothing time was kept at 5 min. Two stage run with and without intermediate grinding had been conducted on primary concentrates. Intermediate grinding was carried out in water in the laboratory porcelain ball mill with porcelain balls, keeping the pH of the solution constant. The pH of the pulp was adjusted with $Ca(OH)_2$ and H_2SO_4.

In a series of single stage flotation experiments[4] the effect of varying particle-size of ore, concentration of the collector, pulp density and pH values[5] were studied.

Results

The results of the grindability and sieve analysis are given in Table 2 and 3.

The sieve analyses of grade 1 ore show that after 40 min of grinding 46.77% particles of +42 mesh size were obtained with the fixed carbon contents ranging from 44.5 to 55%. 21.88% particles were of —42+120 mesh size with the maximum graphitic carbon content of 57–60%, 31.35% particles passing through 120 mesh sieve had 54.58% fixed carbon. Thus the original ore showing a composition of 48.18% graphitic carbon can be upgraded up to 57-60% with recovery percentage of 21.88 (—42+120 fraction) and up to 54–60% with a recovery of 53.2% (—42 fraction).

Results from single stage flotation tests conducted on grade 2 ore using terpineol as collector at different pH values are given in Table 4.

It is evident from Table 4 that there is a gradual improvement in fixed carbon of the concentrate from acidic medium to neutral medium (pH 7). Further increase in pH does not affect fixed carbon in the concentrate. Therefore, in the subsequent experiments the pH was maintained at 7. Also impeller speed of 1500 rev/min with a flotation period of 5 min was kept fixed in each test.

Results presented in Table 5 show the manner in which the fixed carbon content varies in the concentrate with the increase in pulp density.

A concentrate, obtained by employing a pulp density of 15% shows the highest recovery of 87.50% with the maximum fixed carbon content of 60.57%

Second stage flotation with intermediate grinding for varying periods was made on the primary con-

Table 2. Sieve Analysis of Graphite Ore Grade 1 after Grinding for Different Time.

Size mesh B.S.S.	Time (min) 10	Time (min) 20	Time (min) 30	Time (min) 40
+ 10	53.09	48.08	44.46	30.15
— 10+ 22	13.92	10.65	10.98	6.56
— 22+ 42	9.40	6.50	7.49	10.06
— 42+ 60	4.03	4.36	5.16	8.55
— 60+ 85	3.82	5.43	5.24	5.04
— 85+120	2.37	4.35	4.25	8.29
—120+150	2.27	2.45	2.25	4.18
—150+170	1.79	2.44	2.50	5.04
—170+200	1.58	2.16	2.34	5.04
—200	7.73	13.58	15.33	17.09
	100.00	100.00	100.00	100.00

Table 3. Effect of the Particle Size of Ground Grade 1 Ore (Percentages Assays of Fractions).

Mesh size	Weight (%)	Fixed carbon (%)
+ 10	30.15	44.50
— 10+ 22	6.56	48.00
— 22+ 42	10.06	55.00
— 42+ 60	8.55	60.00
— 60+ 85	5.04	57.00
— 85+120	8.29	57.00
—120+150	4.18	56.00
—150+170	5.04	56.10
—170+200	5.04	58.00
—200	17.09	54.00

Table 4. Effect of pH on Flotation of Graphite on Grade 2 Ore.

pH	Fixed carbon in the concentrate (%)	Recovery (%)
2	36.30	48.00
3	30.68	15.00
4	48.00	70.00
5	48.75	61.00
6	25.60	65.00
7	49.75	80.00
8	46.50	79.00
9	45.12	71.00

Table 5. Effect of Pulp Density on Flotation.

Pulp density (%)	Fixed carbon in concentrate (%)	Recovery (%)
10	54.30	76.00
15	60.57	87.50
20	60.23	69.00
25	58.14	65.90
30	53.38	71.20

TABLE 6. SCREEN ANALYSIS OF SECOND STAGE CONCENTRATE WHICH HAS NOT BEEN SUBJECTED TO INTERMEDIATE GRINDING.

Screen size	Weight (%)	Fixed carbon (%)
—120+150	29.63	61.23
—150+170	25.98	59.74
—170+200	38.97	57.44
—200	5.42	53.94

TABLE 7. SCREEN ANALYSIS OF SECOND STAGE CONCENTRATE AFTER SUBJECTION FOR 15 MIN INTERMEDIATE GRINDING.

Screen size	Weight (%)	Fixed carbon (%)
—120+150	21.25	65.25
—150+170	26.25	63.30
—170+200	42.50	59.25
—200	10.00	54.68

TABLE 8. EFFECT OF INTERMEDIATE GRINDING IN A TWO-STAGE FLOTATION TREATMENT AT pH 7.

Time for intermediate grinding (min)	Final concentrate	
	Fixed carbon (%)	Final recovery(%)
0	59.40	55.59
15	61.25	48.60
30	66.76	45.50
40	68.85	42.81

centrate. Results in Tables 6 and 7 show the sieve analysis of concentrates with and without intermediate grinding treatment.

The effect of intermediate grinding was studied for different time on the recovery and carbon content in a two-stage flotation treatment. The results are summarised in Table 8.

Discussion

The results of this investigation indicate that the recovery of graphite particularly depends on the particle size and the texture which have direct bearing on the liberation trend of host minerals. The reduction in the particle size not only causes an increase in the liberation of graphite but also in the surface area of the ground ore. In the particle size range upto + 170 the results indicate that both these factors contribute significantly to the recovery and, therefore, a decrease in particle size in this range results in a rapid increase in the recovery In the — 170 + 200 particle-size range there may be some tendency of agglomeration and hence the effect of specific surface may not be significant.

Pulp density (P.D.) variation has a marked effect on the percentage recovery of graphite. On the contrary, little change has been observed in fixed carbon content in the pulp density range of 15–25%. Sudden fall of recovery beyond the (P.D.) of 15% may be due to the lack of rearrangement of particles as a result of over thickening of the pulp.

The use of terpineol to float graphite was reported by U.S. Bureau of Mines.[5] The results show that much better flotation occurs with neutral pulps, the reason for which is not completely known. However, the effectiveness of high hydrogen ion concentration (low pH value) in suppressing flotation of graphite may be interpretted as the result of competition between the effective collector anion and the hydrogen ions for the mineral surface. It may be inferred that the mineral surface retains both of these ions in a proportion defined by affinities of each ion for graphite and by the concentration of hydrogen and collector ions in the liquor. Over concentration of hydroxyl ion itself acts as a specific depressant for graphite. That is why a fall in fixed carbon as well as recovery of graphite is noted beyond pH 7.

Improved concentrates were obtained by regrinding and floating the primary concentrate. Intermediate grinding produces increase in the fixed carbon content of concentrate. Microscopic examination indicated that concentrate from one stage flotation contained many grains in which graphite and gangue were interlocked due to insufficient grinding. Intermediate grinding caused liberation of the locked graphite which was recoverable in the second stage flotation.

From the investigation carried out regarding the upgrading of graphite by froth flotation, it can be concluded that there is no difficulty in obtaining commercial grade concentrate,[6] with reasonable fixed carbon content, and fairly high recovery rate, from Tirah graphite ore by grinding the primary flotation concentrate and reflotation in the second stage. The product obtained can be utilized in foundry, paints[7] and in the manufacture of refractory articles.

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