Kish Beneficiation by Flotation

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Summary: This paper describes a bench scale investigation on the preparation of flake graphite concentrate from the steel making waste, obtained from Pakistan Steels, Karachi. The waste initially containing 65 % graphitic carbon has been upgraded by froth flotation technique to produce graphite concentrate containing 92.14 % graphitic carbon with 96.46 % recovery. This communication presents the results on the optimization of flotation parameters like pH, pulp density and reagents

Introduction

Graphite is marketed in grades by purity and fineness. Experts like G.S. Brady, have been reported that Number one flake should contain at least 90 % graphitic carbon. Crystalline graphite and flake graphite are synonymous terms for material of high graphite content as distinguished from amorphous [1-3]. Amorphous graphite is not amorphous in the correct sense of the term, but consists of particles so small as to give a compact non-crystalline appearance to the mass [4-5]. Flake graphite is not usually accepted for pencil manufacture as the material, even after very fine grinding, still preserves its flaky character.

The most important uses for graphite are for foundry facings and moulds, graphite crucibles, ladles, stoppers and nozzles, lubricants, paints, brushes for electrical machinery, dry batteries, stove polishes and electrodes. Smaller quantities are used in the manufacture of lead pencils, explosives, in electro-typing, rubber compositions, for preventing scale in boilers, shot polishing, etc [4].

Graphite is found in laminated, flaky aggregates and fine grained disseminations in schistose rocks. It also occurs in veins [6-7]. As with all natural minerals, the availability of graphite is diminishing and costs are rising. Furthermore, Pakistan is totally dependent on foreign sources for this vital material [8]. It is very important to note that this valuable commodity is generated during steel making process as a steel waste. This steel waste is known as Kish. It is a fine powder of graphite and iron but disposed off as landfill for decades. By applying mineral processing techniques of physical beneficiation and chemical purification, a range of graphite concentrates competitive with the natural mineral can be produced from Kish [9- 10].

The present paper mainly deals with the results of flotation study at laboratory scale on the beneficiation of waste Kish obtained from Pakistan Steels, Karachi.

Results and Discussion

The metallurgical balance given in Table- 1 indicates that Kish obtained from Pakistan Steels, Karachi, can be upgraded up to 80.53 % at rougher flotation stage with 97.46 % recovery. It is also important to note from Table-1 that cleaning and recleaning of the rougher concentrate have been ensured a final concentrate grade of 92.14 % with 96.46 % recovery. As number one flake should contain at least 90 % graphitic carbon, this concentrate of grade 92.14 % graphitic carbon can be considered to be good enough [4].

According to Table-2, upgradation of Kish at rougher flotation stage, have been obtained at a pulp pH of 7.5, pulp density 15 % solids, kerosene oil 0.05 Kg/ ton, pine oil 0.005 Kg/ ton, sodium silicate 0.02 Kg/ ton and starch 1.50 Kg/ ton of the feed. Consumption of reagents at rougher flotation stage seems to be quite reasonable [9]. Table-2 also indicates that cleaning and recleaning flotation of rougher concentrate have been done without addition of any flotation reagents. While grade of rougher

Table-1: Metallurgical Balance for Kish Flotation

Product	Wt. %	Grade %	Recovery %
Recleaner Concentrate	68.05	92.14	96.46
Recleaner Tailings	5.95	6.55	0.60
(Cleaner Concentrate)	(74.0)	(85.26)	(97.06)
Cleaner Tailings	4.67	5.49	0.40
(Rougher Concentrate)	(78.67)	(80.53)	(97.46)
Rougher Tailings	21.33	7.73	2.54
Calculated Head Sample	100	65.0	100.0

Table-2: Optimized Parameters for Kish Flotation.

Parameter	Flotation		
	Rougher	Cleaner	Recleaner
PH	7.5	7.5	7.5
Pulp density (% Solids)	15	12	10
Reagents (Kg/T):			
-Collector (Kerosene Oil)	0.05	Nil	Nil
-Gangue Depressant (Sodium Silicate)	0.02	Nil	Nil
-Iron Depressant (Starch)	1.5	Nil	Nil
-Frother (Pine Oil)	0.005	Nil	Nil
Conditioning time	05 min.	04 min.	03 min.
Flotation time	15 min.	12 min.	10 min.

Table-3: Effect of pH

pH	Wt. %	Grade% (Graphitic carbon)	Recovery %
6.5	78.00	75.00	90.0
7.0	78.65	80.50	97.41
7.5	78.67	80.53	97.46
8.0	78.70	80.35	97.29
8.5	78.40	80.40	96.98

Pulp Density= 15% solids, Kerosene oil = 0.05 kg/ T, Pine oil = 0.005 Kg/ T, Sodium Silicate = 0.02 Kg/ T, Starch = 1.5 Kg/ T, Rpm = 1200, Conditioning time = 5 min., Flotation Tim e= 15 min.

Table-4: Effect of Pulp Density.

Pulp Density % solids	Wt. %	Grade % (Graphitic carbon)	Recovery %
25	79.44	70.00	85.55
20	84.98	74.50	97.40
15	78.67	80.53	97.46
10	78.89	80.0	97.10

pH = 7.5, Kerosene oil = 0.05 kg/ T, Pine Oil = 0.005 Kg/ T, Sodium Silicate = 0.02 Kg/ T, Starch = 1.5 Kg/ T, rpm = 1200, Conditioning time = 5 min., Flotation Time = 15 min.

Table-5: Effect of Collector

Collector (Kerosene oil) Kg/ T	Wt. %	Grade % (Graphitic carbon)	Recovery %
0.03	84.37	70.03	90.90
0.04	79.55	75.50	92.40
0.05	78.67	80.53	97.46
0.06	80.03	78.03	96.07

pH = 7.5, Pulp Density = 15 % solids, Pine Oil = 0.005 Kg/ T, Sodium Silicate = 0.02 Kg/ T, Starch = 1.5 Kg/ T, rpm = 1200, Conditioning time = 5 min., Flotation Time = 15 min.

concentrate has been increased from 80.53 % to 92.14 % with only 1 % loss in recovery. It means froth flotation technique can be considered economical route for upgradation of Kish [9].

Table-3 indicates the effect of pH of the pulp on the % purity and recovery of the product. The maximum purity is achieved at a pH of 7.5. According to Taggart graphite floats in the alkaline baths as well as Majumdar and Yukio have obtained the best results after employing pH above 7.0. It is also clear from the Table-3 that an alkaline pH more than 7.5 adversely affect the optimum results.

Table-4 describes that the pulp density variation has a significant effect on the grade and the recovery of graphite. For flakey ores low pulp density is recommended [11]. It is clear from the results that as the pulp density is reduced from 25 to 15 % solids, the grade and recovery of the concentrate improve while the grade and recovery fall as the pulp becomes more dilute than 15 % solids. At pulp density of 15 % solids, the grade of the rougher concentrate is the best without any big difference in the recoveries in the range of 10-20 % solids.

It is obvious from Table-5 that an increase in the quantity of collector significantly affects the grade and recovery. A dose of 0.05 Kg/ ton of kerosene oil following by the frother (pine oil) dose of 0.005 Kg/ ton shows reasonable grade and recovery. Further increase in the dosage of frother (Table-6) lowers the recovery and does not have significant effect on grade and recovery.

Graphite, particularly of the flake variety, is readily floated. The difficulty in treatment is to keep down the gangue. The separation of gangue therefore requires treatment to effectively separate the useless from the useful mineral. In the light of results appearing in Tables-7 and 8, combination of starch (1.50 Kg/ ton) and sodium silicate (0.02 Kg/ ton) fulfills the purpose efficiently. It has been observed that excess of these reagents, however, caused the formation of brittle froth and consequently depressed even the required mineral particles.

Due to relatively coarse (100 mesh) size of Kish, speed of impeller seemed to be an important factor affecting the purity and recovery of the product [9]. Table-9 indicates that beyond a critical speed, it has adversely affected the purity and recovery of the

Table-6: Effect of Frother.

Frother (Pine Oil) Kg / T	Wt. %	Grade % (Graphitic Carbon)	Recovery %
Nil	80.15	78.50	96.80
0.003	80.10	79.00	97.35
0.005	78.67	80.53	97.46
0.007	78.77	79.30	96.10

pH = 7.5, Pulp Density = 15 % solids, Kerosene oil = 0.05 kg/ T, Sodium Silicate = 0.02 Kg/ T, Starch = 1.5 Kg/ T, rpm = 1200, Conditioning time = 5 min., Flotation Time = 15 min.

Table-7: Effect of Gangue Depressant.

Depressant (Na_2SiO_3) Kg/ T	Wt. %	Grade% (Graphitic Carbon)	Recovery %
Nil	84.0	75.06	97.0
0.005	82.65	76.44	97.20
0.020	78.67	80.53	97.46
0.035	79.27	79.02	96.37

pH = 7.5, Pulp Density = 15 % solids, Kerosene oil = 0.05 kg/ T, Pine Oil = 0.005 Kg/ T, Starch = 1.5 Kg/ T, rpm = 1200, Conditioning time = 5 min., Flotation Time= 15 min.

Table-8: Effect of Iron Depressant

Depressant (Starch) Kg / T	Wt. %	Grade % (Graphitic Carbon)	Recovery %
0.50	84.35	70.90	92.00
1.00	80.71	75.70	94.00
1.50	78.67	80.53	97.46
2.00	78.81	80.04	97.04
2.50	78.0	80.00	96.00

pH = 7.5, Pulp Density = 15 % solids, Kerosene oil = 0.05 kg/ T, Pine Oil = 0.005Kg/ T, Sodium Silicate = 0.02 Kg/ T, rpm = 1200, Conditioning time = 5 min., Flotation Time = 15 min.

Table-9: Effect of Speed

Speed (rpm)	Wt. %	Grade % (Graphitic Carbon)	Recovery %
900	75.03	76.31	86.91
1000	75.42	77.40	89.81
1100	75.18	79.00	91.37
1200	78.67	80.53	97.46
1300	75.03	78.03	90.07

pH = 7.5, Pulp Density = 15 % solids, Kerosene oil = 0.05 kg/ T, Pine Oil = 0.005 Kg/ T, Sodium Silicate = 0.02 Kg/ T, Starch = 1.5 Kg/ T, Conditioning time = 5 min., Flotation Tim e= 15 min.

Table-10: Effect of Conditioning Time.

Conditioning Time (minutes)	Wt. %	Grade % (Graphitic Carbon)	Recovery %
1	80.71	75.70	94.00
3	79.27	79.02	96.37
5	78.67	80.53	97.46
7	78.81	80.04	97.04

pH = 7.5, Pulp Density = 15 % solids, Kerosene oil = 0.05 kg/ T, Pine Oil = 0.005 Kg/ T, Sodium Silicate = 0.02 Kg/ T, Starch = 1.5 Kg/ T, rpm = 1200, Flotation time = 15 min.

concentrate. It is evident that the speed 1200 rpm brings about a good compromise between purity and recovery while further increase in speed again lowers the grade and recovery.

It may be seen from Table-10 that conditioning time has a pronounced effect on the grade and recovery of the concentrate. It appears that a prolonged conditioning time peels off conditioner coating on the gangue resulting in lower flotation grade and recoveries. A conditioning time of 5 minute was found to be sufficient for an optimum contact of the depressant with the mineral particles under the conditions existing in the flotation cell, for an optimum grade and recovery.

It may be seen from Table-11 that a flotation time of 15 minutes gives the best grade and recovery. While the grade of the concentrate falls by almost 5 % with 5 minutes increase in the froth collecting period.

The grade (graphitic carbon = 65 %) of Kish presented in Table-12 seems to be more than sufficient to upgrade this material on commercial scale to produce the graphite concentrate despite presence of other impurities. While the impurities can easily be removed either by flotation or acid leaching [10].

XRD of Kish (Fig. 1) presents powder photograph with data of diffracted X-rays which was recorded and drawn by XRD computer itself. Major peaks of Fig. 1 confirm the presence of maximum amount of flake graphite as two theta values of 26.43 and 54.56 corresponds with the standard d values, 3.37 and 1.68 of graphite. It was further identified by JCP.CAT search/ match programme provided with the X-ray Diffractometer D-5000 (Siemens, Germany) that the minor peaks corresponds with silica and magnetite. This programme contains d/ I values of about 60,000 Standards for material identification [12].

Fig. 2 shows that froth flotation circuit is comprised on three stages of flotation that is rougher, cleaning and recleaning. It is cleared that cleaning and recleaning operations after the rougher flotation have been ensured a final concentrate grade of 92.14 % with 96.46 % recovery.

Size analysis of Kish presented in Table-13 indicates that as the mesh number increases wt. %

Table-11: Effect of Flotation Time.

Flotation Time (minutes)	Wt. %	Grade % (Graphitic Carbon)	Recovery %
10	78.89	80.0	97.10
15	78.67	80.53	97.46
20	79.55	75.50	92.40
25	80.62	74.50	92.40

pH = 7.5, Pulp Density = 15 % solids, Kerosene oil = 0.05 kg/ T, Pine Oil = 0.005 Kg/ T, Sodium Silicate = 0.02 Kg/ T, Starch = 1.5 Kg/ T, rpm = 1200, Conditioning time = 5 min.

Table-12: Chemical Analysis of Kish.

Constituents	Percentage
Graphitic Carbon	65.0
SiO_2	5.40
Fe_2O_3	26.0
Al_2O_3	0.03
CaO	2.50
MgO	0.02
Na_2O	1.00
K_2O	0.05

Table-13: Size Analysis of Kish

Mesh No. (BSS)	Wt. %	Cum. Wt. % (Retained)	Cum. Wt. % (Passing)
25	2.5	2.50	97.50
50	17.5	20.0	80.0
100	30.0	50.0	50.0
150	32.5	82.50	17.50
200	10.0	92.50	7.50
250	5.0	97.50	2.50
-250	2.5	100.0	0.0

passing decreases. At 100 mesh size, 50 % retaining of material shows that it contains flaky graphite of coarse size. The graphite of this size is considered suitable for crucibles making and other refractory purposes [9].

Anyhow, the experimental results discussed in the proceeding pages have led us to the conclusion that it is possible to produce Number one quality flaky graphite concentrate (C = 92.14 %) by froth flotation from Kish, the steel making waste of Pakistan Steels, Karachi. However it is recommended that detailed study on large scale should be carried out on Kish to treat it as an ore and to produce a range of graphite products competitive with the natural mineral.

Experimental

To the representative sample of Kish from Pakistan steels, Karachi, conventional laboratory techniques of volumetric and gravimetric analysis were employed for chemical analysis. Size analysis

#	2theta	d	Rel. I	#	2theta	d	Rel. I
1	9.9860	6.8521	0.35	5	26.600	3.3245	66
2	23.924	3.7327	2.16	6	32.180	2.7799	1.71
3	26.100	3.4121	3.69	7	54.564	.6000	1.29
4	26.431	3.3700	100.0	8	54.695	.6771	2.36

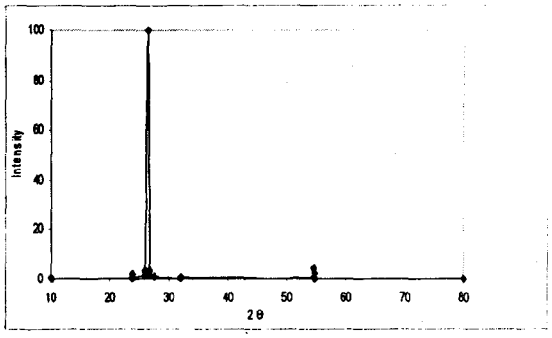

Fig. 1: XRD of Kish

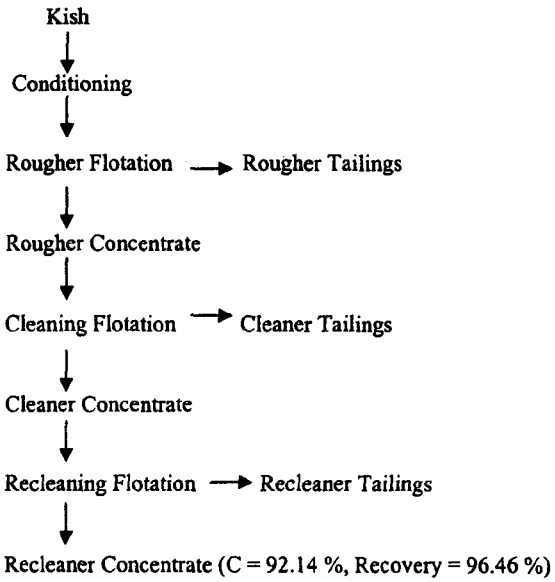

Fig. 2 Beneficiation Flow Sheet of Kish

of Kish was performed by using standard B.S.S. sieves.

Kish was also subjected to X-ray diffraction for phase determination. Phase analysis by XRD confirms the presence of flake graphite, silica and significant amount of iron as magnetite.

Flotation Tests

The flotation tests were carried out in a Denver D-12 flotation machine. Roughing, cleaning

and recleaning flotations were completed under different set of conditions for the optimization of flotation parameters. For roughing flotation the set of conditions was pH 6.5-8.5, pulp density 10-25 % solids, kerosene oil 0.03-0.06 Kg/ t (as collector), pine oil 0.0-0.007 Kg/ t (as frother), sodium silicate 0.0-0.035 Kg/ t (as gangue depressant) and starch 0.50-2.50 Kg/ t (as iron depressant) .

The conditioning time was 1-7 minutes and the froth was collected for 10-25 minutes as graphite rougher concentrate. The cleaning and recleaning flotations were carried out without addition of any flotation reagents.

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