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UPGRADATION OF KALABAGH IRON ORE BY HEAVY LIQUID AND FLOTATION TECHNIQUES

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The possibility of the beneficiation of Kalabagh iron ore was indicated by heavy liquid separation tests. The maximum grades of 51.7- 61.0% Fe with 4.5-13.0% SiO_2 was potentially obtainable from different horizons of the ore seams in Chichali area. Fatty acid flotation with sodium silicate and aluminium chloride as depressant produced in concentrate with 38.1% Fe and 6.1% SiO_2. The concentrate on pelletization and in duration at 1350° gave a concentrate with 55% Fe, 8.8% SiO_2 3.4% CaO and 2.4% MgO.

Key words: Kalabagh iron ore, Concentration, Flotation techniques.

Introduction

The iron bearing deposits at Kalabagh are by far the largest iron resources of the country [1, 2]. The deposits comprise of three main areas namely (i) Chichali, (ii) Makerwal and (iii) Kutch. The estimated total reserves at Kalabagh exceed 400 million tonnes. The Chichali deposit which is the largest of the group was estimated at 292 million tonnes, but has not been brought into production because of the metallurgical problems encountered in recovering the iron. Consequently, the ore was re-investigated and the factors relevant to the behaviour of this ore towards beneficiation were evaluated.

Extensive studies on Kalabagh deposits were one at home and abroad from 1949-1974 by a number of organisations to examine the expediency of establishing iron and steel works in Pakistan from domestic raw materials. These studies were generally based on three major approaches, (a) beneficiation of the ore after reduction roasting in a rotary furnace or fluidized bed [3-5]. In the Krupp process [3] 90% iron recovery was achieved corresponding to 87% metallization. The product was impure iron containing most of the sulfur and phosphorus needing further melting and refining in electric arc furnace. (b) Sintering and smelting tests [6-9]. The studies conducted in France, Germany and USSR has shown that the fuel consumption in these process was significantly high and the process recommended by Saltzgitter [8] in 1967 could not be utilized at the present cost of coke and gas. The presence of large amount of gangue minerals in the ore cause the formation of huge amount of slag and fuel consumption. Gepramez [9] conclusion that, "the establishment of iron and steel works based on Chichali ore would only result to very low efficiency", led to the establishment of integrated steel mills in Karachi based on 100% imported ore and cooking coal. (c) Ore dressing methods: Flotation of non-magnetic iron ores has been the subject of substantial research and development for the last about half a century and 1954 first commercial plant was placed by Humboldt Mining Co. In Machigan, USA. Several ore dressing methods were employed to upgrade Kalabagh iron ores including flotation, high intensity magnetic separation, magnetised roasting followed by magnetic separation. Earlier works [10-12] at PCSIR, BISRA, US Bureau of Mines, Energy and Mineral Resources in Canada were sceptical to physical methods of beneficiation because of fine distribution of iron minerals in the gangue matrix. However, recent studies [13-15] at PCSIR Laboratories, Lahore. Minsesota Mineral Resource Research Centre, USA and Warren Spring Laboratories in UK showed the possibility of extraction of iron rich concentrate by physical beneficiation techniques. The break through was achieved by detailed investigation of Mineralogical and beneficiation characteristics of the ore using ore microscopy and sink float techniques [13]. The limitation of the previous works were described by Khan and Qazi [13]. MRRC [14] reported flotation tests at 95° using fatty acids with high calcium and magnesium contents in the concentrate. Further studies to improve iron recovery was required and the present work was conducted to establish the conditions of flotation of Chichali ore particularly at room temperatures. The paper deals with mineralogical characteristics that affect the behaviour of the material in ore dressing. The flotation investigation involved testing by reverse flotation, direct flotation and desliming. Better results were achieved by direct flotation of deslimed products and is described in detail in this paper.

Experimental

Collection of samples. The ore beds occur in Belemnite beds of lower Cretaceous age and forms two distinct seams namely "upper seam" and " lower seam".

Channel samples were taken from western main adit of Chichali pass exploratory mine, which have been driven horizontally following the strike direction. The tunnel is of about

2-3 m diameter and at 24,37,46 and 55 meters respectively from the mine mouth horizontal cross cuts 1-4 were dug.

Channel sampling were undertaken from cross cuts 3 and 4 to represent the entire exposed thickness of upper iron ore seam and the variation across the thickness in cross cut 3 and 4. These samples taken from a 0.5 m deep channel running horizontally across the rock in the tunnel and cross cuts from the ore and bed which dips more or less 40 degree were:

Sample CH-1 of cross cut 4 representing 1.5 m thickness of greenish glaunitic roof ore in the cross cut lying at the right hand side on entering the mine.

Sample CH-2 representing 2m thickness of the greenish glauconitic iron ore in the tunnel.

Sample CH-3 was yellowish brown iron ore lying below the fossiliferous zone upto the beginning of cross cut at the left it was about 2.5m thick.

Sample CH-5 was the yellowish brown iron ore in the left of cross cut and overlied greenish glauconitic shale. It was about 2 m thick.

The strike is divided by a layer of fossils about 2.8 m from the top. The ore above the fossil layer were called, "Upper Horizon" (UH) and the ore below as "Lower Horizon" (LH). Although difference in behaviour and composition between UH and LH samples, have been found previously but keeping in view that it would not be possible to mine these layers separately a composite sample of the two horizones representative of the ore body was also sampled Tabled CH-Mix).

Two samples were also taken from cross cut 3, labelled CH-3 (3 m in thickness from UH) and CH-7 (3 m in thickness).

Mineralogical studies of Chichali ores. A combination of techniques were employed to establish the mineralogical characteristics of Chichali iron ore that affect ore dressing. The techniques used were X-ray analysis, DTA, TGA, petrological and ore microscopic studies of polished samples. Thin sections and crushed ore particles in different sizes. Mesh of liberation, model analyses particle counting and heavy liquid separation were conducted using the method of Muller and Burton [16]. In this method the ore was crushed to below the liberation size and centrifuged in a density gradient. The various minerals separated into bands at their density levels from which they were extracted and weighed. The technique was also used for establishing liberation size when grinding at different sizes were subjected to density separation. The liberation size produced sharply defined layer fractions whereas coars grinding produced diffuse fuzzy layers.

Flotation technique. The ore was first subjected to stage grinding in order to reduce the generation of fines by repeated sieving during grinding to get maximum amount of liberated sand. By this technique generally 70% sand (-150 mesh) was recovered and 30% slimes were generated.

A batch of 200 g of ore sample was wet ground to pass 80 percent of it through 150 mesh screen, then deslimed by using hydro-separator. The deslimed sand was taken in flotation cell (Denver D-12 cell) and was conditioned at room temperature with a collector at high pulp density (55-60%). The collectors used for the flotation of iron minerals and reverse flotation of gangue were fatty acids, their salts, sulphonates, tall oils and amines.

The pH was adjusted between 5-10 by addition of H_2SO_4 and sodium carbonate. Various types of depressents and modifiers were tried to depress the silicates. Small amounts of collector was added at different intervals when the froth seemed to subside. Froth was collected separately after every addition of collectors, so as to find the optimum quantity of collector required. Several schemes of flotation were tested in order to find the ore most suitable for the beneficiation of Chichali iron ore. The variables employed in these tests were different pulp densities, pH, conditioning time, concentration of surfactants, depressents and modifiers.

Results

Mineralogical composition of upper horizon rocks . These rocks consist of siderite and chlorite in major amounts and guartz, goethite/limonite and glauconite in minor amounts. In thin sections, iron silicate mineral is present as large irregularly shaped grains with aggregate of siderite grains distributed as matrix. Fine grained reddish to opaque minerals of iron oxide are disseminated in the matrix. Miner amounts of quartz and traces of calcite, apatite, feldspar, pyrite and clay mineral are also noticed. The overall texture of the rock is mossaic with low grain-cohesion index due to the setting of large crystals in fine grained matrix and will, therefore, tend to be easily crushed on milling. The grain size of different constituents are shown in Table 1.

TABLE 1. GENERAL GRAIN SIZE ANALYSIS OF ROCKS.

Sample	Mineral	Abundance	Size range (microns)	Common (size)	Remarks
UH	Chlorite	High	150-800	350	Size of the siderite
rock	Siderite	High	50-200	100	aggregation several
	Quartz	Low	50-200	150	times greater than
	Goethite	Trace	25-100	25	100 microns
LH rocks	Chamosite	Moderate	100-350	200	
less	Goethite	Moderate	150-500	300	
ltered	Siderite	Moderate	50-150	Variable	
	Quartz	Low	50-300	150	
LH rocks	Chamosite	Moderate	100-250	_	
	Opaque ore	high	200-400	_	High goethite/siderite
more	Siderite	Moderate	50-150	_	ratio and abundant Sec
altered					iron silicate

Mineralogical composition of lower horizon rocks. The rocks in the lower horizon are yellowish brown to dark greenish brown and consist of siderite-goethite/limonite, chamosite (chlorite), glauconite mineral assemblage. The crystalloids of goethite are pseudomorphic to chamosite, having been changed by later decomposition or replacement as indicated by the relict structure. The original chamosite is larger in size than the fresh looking small grained iron silicate mineral which is of secondary origin.

Variation in the proportion of major minerals is observed in different specimen which ranges from major chamosite, siderite mineral assemblage to substantial increase (upto 50 percent of the total mass) in goethite and opaque iron oxide consituents formed at the expense of chamosite and siderite. In the less altered rocks dark coloured iron-rich nucleous is formed in chamosite and reddish brown iron oxide colouration is produced in siderite matrix. The grains size of different minerals are widely variable and are summerized in Table 1.

Chemical composition. The chemical analyses (Table 2) show that ferrous iron, ignition loss and calcium contents are significantly higher in the samples of UR whereas ferric iron is higher in LH due to high siderite content in UH and high iron oxide in LH. Silica and silicates remain more or less constant as shown in this section studies.

TABLE 2. CHEMICAL COMPOSITION OF CHICHALI IRON ORE.

Sample No	L.I.	SiO_2	FeO	Fe_2O_3	Al_2O_3	CaO	MgO	S	P
CH-1	23.82	16.46	28.98	17.46	2.56	8.04	1.12	1.89	0.22
CH-2	23.98	17.38	31.37	14.69	5.16	6.17	Nil	1.56	0.30
CH-3	18.94	17.76	22.34	28.51	4.05	5.10	1.85	1.55	0.38
CH-5	14.62	28.36	13.68	30.36	5.06	5.05	1.21	1.30	0.37
CH-6	21.60	21.56	23.78	21.66	3.76	5.17	1.62	1.34	0.20
CH-7	18.25	18.30	14.78	35.27	5.15	5.60	2.05	1.05	0.45
CH-Mix	19.97	20.72	29.62	15.23	1.72	4.48	2.85	_	0.38

Mesh of liberation. The samples were crushed and ground to pass 60 mesh sieve (BSS) and sized using 30, 100, 120, 150 mesh sieves. Quantitative estimate of locked and free ore and gangue, particles by microscopic counting method reveals that above 120 mesh size the locked ore and gangue particles are more than 30 percent. On further grinding to below 150 mesh size the extent of ore-gangue locked particles reduce to 3-6 percent.

The iron ore minerals are transitional in nature, probably formed by the alteration of siderite to hydrogoethite and goethite. The resultant particles are mostly composite of ore between 150-250 mesh size.

Model analysis. Model percentages were evaluated quantitatively by using microscope-particle-counting on heavy liquid fractions (HLS) of different density and are summarized in Table 3.

The gangue was appreciably separable from ore minerals. For example in sample CH-1, 34.2 percent chlorite was recovered in lighter fraction HLS-3 with 72 percent free and 16 percent locked chlorite particles, while the ore concentrate (HLS-1) which was 56.9 percent by weight contain only 6 percent free and locked particles of chlorite. The heavy fraction of CH-1 had 42 percent total Fe, corresponding to about 70 percent recovery, which on ignition can yield pellets containing 86 percent Fe_2O_3. In sample CH-5 the maximum calculated content of Fe_2O_3 in ignited concentrate come to about 75 percent. However, considering the amount and composition of middling the calculated iron concentration will reduce in mixed concentrate although at a higher Fe recovery.

TABLE 3. MINERALOGY OF HEAVY LIQUID GRAVITY FRACTIONS (PARTICLE SIZE - 150+345 MESH) (B.S.S.)

Sample No.	CH-1 HLS1	CH-2 HLS1	CH-3 HLS1	CH-5 HLS1	CH-1 HLS2	CH-2 HLS2	CH-3 HLS2	CH-5 HLS2	CH-1 HLS3	CH-2 HLS3	CH-3 HLS3	CH-5 HLS3	CH-5 HLS4
Density g ml^{-1}	+3.45	+3.45	+3.45	+3.99	3.45-3.18	3.45-3.24	3.45-3.18	3.99-3.37	3.18-2.8	3.24-2.6	3.18-2.8	3.37-2.8	2.80-2.0
Weight Percent	56.9	52.2	52.8	39.7	9.1	10.2	31.8	47.9	34.2	37.6	15.7	5.8	6.6
Mineral Percentage													
Chlorite±	1	1	0	1	4	11	8	10	72	74	46	7	2
Glaucotine											4		71
Quartz+Calcite													
Locked Ch1. -ore	5	6	11	11	55	27	46	40	16	16	27	65	-
Opaque ore	5	5	15	19	8	5	14	11	2	1	2	4	-
Geothite+Siderite	41	37	19	28	12	25	12	10	7	4	12	9	-
Siderite	19	23	11	10	8	18	8	4	5	4	2	10	24
Composite iron oxide	29	28	34	26	18	14	12	25	2	4	7	5	3
Partial chemiaal analysis													
SiO_2	4.14	3.10	7.62	10.26	10.20	9.15	24.10	31.88	37.9	36.98	33.80	41.75	88.30
Fe^{+3}	9.80	9.24	18.9	24.08	12.60	12.6	20.	23.1	14.70	18.85	17.92	12.25	1.25
Fe^{+2}	33.20	32.76	23.10	16.52	25.90	28.00	6.72	9.24	5.74	5.74	4.48	6.30	2.24
L.I.	29.86	31.22	25.00	21.50	14.66	30.50	15.50	13.40	11.30	16.30	12.20	11.10	9.25

The foregoing studies have indicate that over 80 percent iron ore minerals can be liberated from the silicate matrix on grinding to a size finer than 150 mesh. Also, that the ore after this grind size is amenable to beneficiation using mineral dressing methods separately or in combination.

Flotation studies. Several procedures were tested to find the most suitable for the beneficiation of Chichali iron ore. Flotation with FA-2 (fatty acid) showed better results among the collectors used for the flotation of iron minerals and reverse flotation of gangue such as, fatty acids and their salts, sulfonated tall oil and amines. The results of flotation at different pH (Table 4) showed that in the presence of FA-2 as collector and D-128 and R-422 (of Flotbell) as depressent, optimum grade recovery of iron ore minerals were achieved at pH 10. Other depressent such as D-60, causterised starch, calcium chloride and sodium silicate were also tried. Sodium silicate was found to be the best depressent for the silicate in the quantities of 2-5 ml of 2.5 percent solution. To supress the alkaline earth carbonates, trivalent metal salt such as iron and aluminium were used in presence of fatty acid and sodium silicate. The aluminium chloride reduced CaO and MgO contents to half in the concentrate. The result of flotation of Chichali iron ores from upper and lower horizon using fatty acid with sodium silicate and aluminium chloride is reported in Table 5. The final rougher concentrate having the following composition Fe 38.1% SiO_2 6.2%, CaO 2.4%, MgO 2.0% and P 0.32% was produced using CH-Mix ore. The products was rolled into pellets after mixing with 0.5% of bentonite and indurated at 1350° to obtain fired pellets containing 55% Fe, 8.8% SiO_2, 3.4% CaO and 2.4% MgO.

Discussion

Low iron content and the presence of prohibitive amount of undesirable impurities in Chichali ore necessitates prior beneficiation of the ore for iron and steel manufacture. The present investigation has demonstrated that the results of heavy liquid separation are significant in regard to the possibility of beneficiating the ore. The density product HLS-1 and HLS-2 for upper horizon sample and HLS-1 for lower horizon sample does given a realistic indication of what might be recoverable as iron concentrate. By substituting the loss on ignition (Ll) component, the value for the total iron percentage in the fired pellets could be calculated and is given in Table 6. The maximum grade of 37.4-61.0% Fe with 4.5-6.9% SiO_2 is obtainable from upper horizon ore and 51.7-56.0% Fe with 10-13% SiO_2 potentially obtainable in lower horizon ore. The lower value in the later case is due to goethite/chlorite (alumino silicate) intergrowth which contaminates the products. The recovery of the locked goethite in chlorite, however, needed further studies. The chlorite/chamosite phase is found to be of two paragenetic type, one altered and early variety may be termed as chamosite and the younger green coloured mineral as chlorite.

Table 4. Typical Flotation Results of CH-3 Ore with Varying Conditions.

pH	Collector	Depressant	Wt. recovery %	Fe % in conc.	SiO_2 in conc.
7	FA_2 0.3 ml	4 ml Soda silicat 10 ml D-128, 1%	6	35	9.48
8	FA_2 0.3 ml	10 ml D-128, 0.2% 2 ml Flotebll R-422 4%	35	35.35	7.88
9.	FA_2 0.3 ml	10 ml D-128, 0.2% 2 ml Flotbell R-422 4%	36	35.70	8.72
10	FA_2 0.3 ml	-do-	40	37.10	8.98
11	FA_2 0.3 ml	-do-	45.5	36.05	9.80
10	FA_2 0.3 ml	2 ml 21/2% $NaSiO_3$	R Conc.1 24.0 R Conc.2 30.0	35.90 35.20	5.80 10.80
10	FA^2-0.3 ml	2 ml 2 1/2% $NaSiO_3$ +0.2 g $AlCl_3$	R Conc.1 42.5 R Conc. 2 12.5	36.10 30.10	4.50 5.90

TABLE 5. FLOTATION OF CHICHALI IRON ORES FROM UPPER AND LOWER MIXED HORIZONS.

	Sample Product	Weight %	Analysis% Fe	SiO_2	Distribution% Fe	SiO_2
Upper Horizon	1. R.Conc.1	24.6	37.8	5.8	26.9	8.4
	R. Conc.2	22.4	35.9	10.8	23.6	14.3
	Tailings	30.0	29.8	24.6	25.7	43.5
	Slimes	23.0	32.4	24.2	23.8	33.8
	2. R.Conc.	47.8	37.3	7.5	51.7	21.2
	Tailings	24.0	29.8	27.0	20.7	38.2
	Slimes	28.0	32.9	24.4	27.6	40.6
Lower Horizon	1. R.Conc.1	23.8	40.9	4.9	26.2	6.7
	R. Conc.2	21.5	38.9	7.8	22.5	9.4
	Tailing	28.5	34.2	29.3	26.4	47.2
	Slimes	26.2	35.5	24.8	24.9	36.7
	2. R.Conc.1	24.4	40.4	4.7	26.4	6.5
	R.Conc.2	21.8	38.6	11.9	22.5	14.6
	Tailing	24.4	33.5	29.4	22.1	40.7
	Slimes	29.4	34.9	23.2	28.0	38.2
Mixed Horizon	R. Conc.	46.8	38.1	6.2	52.9	14.0
	Tailing	25.7	27.8	41.8	21.2	51.9
	Slimes	27.5	31.6	25.7	25.9	34.1

TABLE 6. CALCULATED RESULTS OF WEIGHT RECOVERY SiO_2 AND FE PERCENTAGE OF PRODUCTS AFTER INDURATION.

Sample No.	CH-1		CH-2		CH-3	CH-5
	HLS1	HLS+2	HLS1	HLS+2	HLS-1	HLS-1
Density gm^{-1}	+3.45	+3.9	+3.43	+3.2	43.45	43.99
Wt.recovery	56.9	66.0	52	62.4	52.60	39.70
SiO_2	5.9	6.9	4.50	5.6	10.16	13.10
Fe (t)	59.9	37.4	61.10	60.6	56	51.70

Although the ore feed prior to beneficiation apparently contained 32.5% iron, but about one third of this iron is tied up in gangue minerals. The recoverable iron in the ore accordingly, is about 32 percent. Keeping this in view, the iron recovery was found to be about 85 percent corresponding to an overall recovery of 60 percent by flotation. The flotation procedure developed using fatty acid as collector and sodium silicate and $AlCl_3$ as depressent for silica, CaO and MgO at pH-10 is, however, based on tunnel samples of old PIDC mine at Chichali. The need for fresh sampling for further refining of the mineralogical and flotation methods is apparent as the flotation technique depends upon the mineralogical composition particularly of the fresh surface of the ore particles. The previous work, though enourmous, do not provide sufficient data on the mineral composition, grain size variation and flotation characteristics. The flotation method developed during this investigation may be the most economical method for concentrating this ore, especially because it is relatively inexpensive and lend itself easily to high capacity production.

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