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COLUMN FLOTATION STUDIES ON LOW GRADE IRON ORE SLIMES OF AN OPERATING PLANT IN INDIA

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Abstract

The present investigation deals with the recovery of iron values from the screw classifier overflow slimes of an operating iron ore washing plant using pilot scale flotation column (0.5 m diameter). Initially, selection of collector and optimization of process parameters like the dosages of collector and depressant and solids to liquid ratio during 'reverse flotation tests' was carried out on bench scale laboratory flotation cell. During the on-site pilot plant trials, operating parameters of flotation column such as air flow rate, froth depth, feed flow rate and wash water rate were optimized. Feed assaying Fe - 58.00%, SiO_2 - 6.21%, Al_2O_3 - 5.70% could be upgraded to Fe - 58.54%, SiO_2 - 5.29%, Al_2O_3 - 4.77% with weight recovery of 86.90% and Fe recovery of 88.97%. Iron oxide and alumina have identical crystal structure and at times exist in solid solution (one form of interlocked state). It appears that this contributed to limitations in separation of alumina from iron bearing minerals from these slimes.

Key words: *iron ore slimes; cationic collector; reverse flotation; column flotation; alumina reduction.*

1. Introduction

Iron ore, serving as the chief raw material for iron and steel industries, is beneficiated to lower alumina to silica ratio. However, each and every deposit of Iron ore has its own distinct mineralogical characteristics and needs a specific beneficiation route to get the best possible iron ore concentrate from it. On the other hand, the beneficiation process to be adopted and its successful operation also depend on the mineralogical nature of the gangue and its textural association with the valuable iron bearing minerals. Sizing, washing, classification, jigging, magnetic separation, advanced gravity separation techniques and/or flotation are being normally practiced in India to enhance the iron ore quality. During sizing and washing of the ore, huge quantities of slimes are generated. It is estimated that around 10 million tonnes of slimes are being generated every year during the processing of hematite ore containing around 48-62% of Fe. The slimes, as such, are not suitable in iron making due to the presence of higher amount of gangue (alumina and silica), but are attractive from granulometry point of view. In earlier days, these were discarded into the tailing ponds. Of late, these are being treated in hydrocyclones for recovery of iron values, as an extension of the existing washing circuit. However, beneficiation and utilization of these slimes still remains a challenging task due to the sub-optimal recovery of values in hydrocyclones. In the past, several beneficiation techniques

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have been tried by several research groups from time to time to reduce the gangue [1, 9, 15]. These include separation in a hydro-cyclone [2], flocculation techniques [4, 5, 7], selective dispersion – flocculation – flotation [6], wet high-intensity magnetic separators (WHIMS) followed by classification in hydrocyclone [11], classification by hydro-cyclone followed by high-intensity magnetic separation [1], classification in a hydro-cyclone followed by spiral concentration [16], enhanced gravity techniques by CSIR-AMPRI, Bhopal [3], floatex density separator followed by gravity / magnetic separator [12], hydrocyclone followed by wilfley table, WHIMS and flotation [8] and hydrocyclone, WHIMS and flotation [17]. TATA Steel developed cold bonded briquettes, iron ore nuggets, bricks, tiles and pavement blocks out of the poor grade slimes [21]. Earlier studies [13, 18, 19, 20] on iron ores of India indicated that slilica and alumina could be reduced by reverse cationic column flotation of fine iron ore concentrate as a value addition step on a pre-concentrate obtained from an operating beneficiation plant. Rocha et al., [14] also successfully applied reverse cationic column flotation process to beneficiate iron ore slimes of Brazilian origin. In the Indian context, most of the above studies are based on physical separation techniques. It is a well known fact that the efficiency of physical separation techniques is limited while treating slimes, as the physico-chemical properties of slimes start dominate over their physical properties. This generally results in poor recoveries. Flotation in general and column flotation in particular is thought to be apt to treat slimes to realize better selectivity of separation, grade and recovery [18]. In this present study, the authors have attempted to beneficiate and recover the iron values from the screw classifier overflow slimes from an operating iron ore washing plant by means of pilot scale flotation column.

2. Materials and Methods

2.1. Materials

Cationic amine collectors which are generically same but compositionally different from each other were manufactured and supplied by M/s Somu Organo-Chem Pvt. Ltd., Bengaluru, India. These cationic collectors are proprietary in nature and their chemical composition is not revealed from intellectual property point of view. They are said to be ether amine based and coded as SOKEM 521C, SOKEM 522C and SOKEM 524C. Their performance was evaluated and SOKEM 524C was chosen as the best among these three for lowering alumina content and optimizing flotation process parameters [20]. Causticised maize starch, used as depressant for iron bearing minerals, was supplied by Riddhi Siddhi Gluco Biols Ltd., Ahmedabad, India. Commercial grade sodium hydroxide was used as pH regulator.

2.2. 0.5m diameter NML flotation column

0.5m diameter flotation column designed and developed by CSIR-NML Madras Centre, India was shifted to the beneficiation plant of M/s TATA Steel Limited at Joda East Iron Mines (JEIM), Joda, Odisha, India. It was erected at a suitable location so as to facilitate feeding of screw classifier overflow to the flotation column through three conditioners. The details of the flotation column and the sequence of addition of reagents are shown in Figure 1. The column shells are made up of mild steel and can be mounted one over the other by joining these flanged shells. Slurry/froth interface is maintained using Differential Pressure Transmitter (DPT) mounted over one of the shells. The output signal of the DPT is looped to an electro-pneumatic discharge valve through a YS 170 controller. Based on the signal (4-20 mA) given by DPT, the discharge valve will be automatically actuated and accordingly the slurry discharge is maintained. The froth depth could be altered between 50-2000 mm by changing the set-point in the controller. One-phase internal porous spargers, made of sintered silicon carbide and designed by CSIR-NML Madras Centre were used. Air flow rate is controlled and monitored by purge rotameter while the feed slurry flow rate is regulated by variable frequency driven feed pump and monitored by using magnetic flow meter.

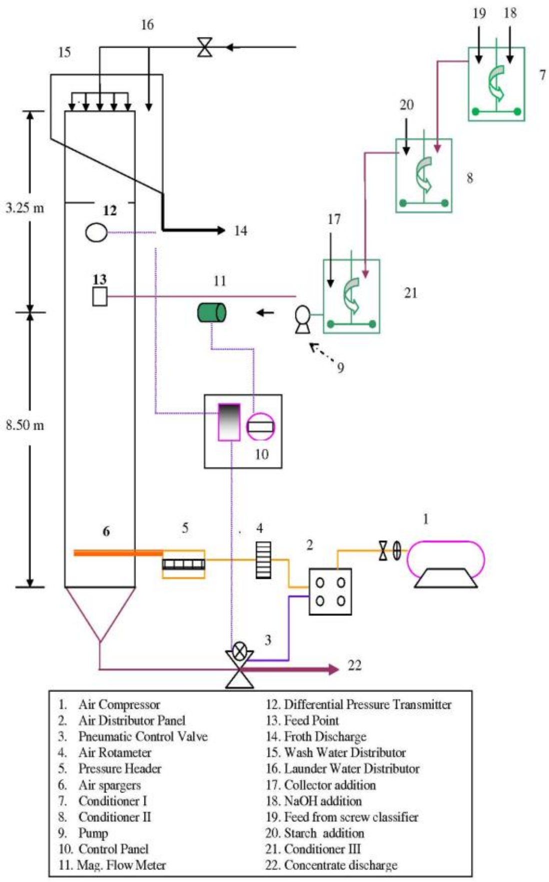

Figure 1. Schematic diagram of the 0.5 m dia flotation column

The air from the compressor is let into the column at a desired flow rate. The column is filled with water and stabilized at required froth depth at a constant wash water addition. After stabilization with water, the reagents-conditioned iron ore slimes are pumped into the column at desired flow rate. Minimum residence time of 10 minutes is maintained in all the conditioners. Then the slurry is pumped into the column through a variable frequency driven feed pump. The column is allowed to run for a minimum period of 3 - 4 residence times. Concentrate and tailings samples are drawn under near-steady state conditions. Both the process and column operating parameters are recorded before collecting samples. The collected samples are measured for pulp density, volume of the slurry, weight of the slurry and solids after drying. Dried samples were analyzed for Fe, SiO2, Al2O3 by chemical analysis wing at JEIM.

3. Results and Discussions

In plant practice, the characteristics of the feed to the beneficiation plant keep changing from time to time. Hence, it was thought prudent to erect 0.5m diameter pilot scale flotation column at the plant site and carry out trials under varied plant operating conditions with the objective of optimizing the operating parameters of the flotation column and run the flotation column continuously under these conditions.

3.1. Characterisation

Screw classifier overflow was characterized in terms of its fractional size analysis and X-Ray Diffraction for identification of mineral phases in them. The sample is extremely fine in nature (d_{80} = 45.6 µm). The +150, +125, +106, +90 and +75 µm sieve fractions were chosen for the XRD study as they contain higher alumina. The diffractogram is shown in the Fig. 2.

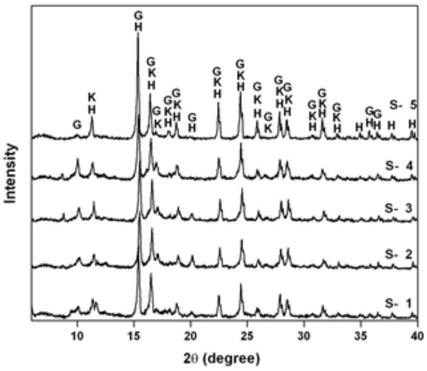

Figure 2. XRD pattern of +150 µm (S-1), +125 µm (S-2), +106 µm (S-3), +90 µm (S-4) and +75 µm (S-5) sieve fractions. H=Hematite, G=Goethite, K=kaolinite.

From the figure, it is clear that hematite and goethite are the iron bearing mineral phases. Kaolinite occurs as minor gangue phase. Though quartz is present in minor quantities but it was not reflected in the XRD patterns. These quartz and kaolinite are the silicate gangue mainly contributing towards the silica and alumina respectively in the sample. Even though the diffractograms corresponding to five size fractions appear to be more or less similar, heavy media separation tests (or float - sink tests) on these size fractions using tetrabromoethane indicated that most of the mineral phases are in interlocked state indicating, prima facie, a poor separation. There was no segregation of any particular mineral phase in significant amount in any size fraction, which otherwise, might have aided better separation. Specific gravity of the representative sample was determined and found to be 3.80. The details of the characterisation studies are reported elsewhere [20].

3.2. Effect of variation in air flow rate

Generally, superficial air velocity should be as large as possible to ensure a high throughput. At the same time, if the superficial air velocity is too high, the flow pattern will be disturbed and there is every possibility to lose bubbly swarm. Performance data are obtained for superficial air velocities ranging from 0.68 cm/s to 1.27 cm/s. The results are presented in Table 1.

Table 1. Flotation tests on pilot scale column to optimize air flow rate

Superficial air velocity (cm/s)	Product	Weight, %	Assay, %			Distribution, %		
			Fe	SiO_2	Al_2O_3	Fe	SiO_2	Al_2O_3
0.68	Tailings	2.4	39.30	20.00	11.63	1.88	3.64	3.46
	Conc.	97.6	50.50	13.02	7.97	98.12	96.36	96.54
	Head (Calc.) Head (Assay)		50.23 46.25	13.18 14.77	8.08 9.77	100.0	100.0	100.0
0.93	Tailings	9.5	42.42	15.56	12.06	7.93	14.36	13.14
	Conc.	90.5	51.73	9.74	8.37	92.07	85.64	86.86
	Head (Calc.) Head (Assay)		50.85 47.78	10.29 13.31	8.72 9.89	100.0	100.0	100.0
1.27	Tailings	12.2	53.53	8.23	7.84	11.48	16.40	16.09
	Conc.	87.8	57.33	5.83	5.68	88.52	83.60	83.91
	Head (Calc.) Head (Assay)		56.87 56.84	6.12 5.70	5.94 6.47	100.0	100.0	100.0

Process parameters: pH: 9.5; Starch: 1.0 kg/t; Sokem 524C: 0.3 kg/t; Feed pulp density: 1.07
Column parameters: Superficial feed velocity: 0.64 cm/s; Froth depth: 200 mm; Superficial wash water velocity: Nil

Air flow rates, expressed as the superficial velocity (cm/s) had a significant influence on percent alumina removal from the feed (screw classifier overflow slimes). Superficial air velocity of 1.27 cm/s was found to be optimum. Beyond superficial air velocity of 1.27 cm/s, turbulent flow regime had set in causing mixing in the system which is undesirable. It is to be noted that at this superficial air velocity of 1.27 cm/s, maximum removal of % Al_2O_3 (16.09%) took place. Feed assaying Fe - 56.84%, SiO_2 - 5.70%, Al_2O_3 - 6.47% could be upgraded to Fe - 57.33%, SiO_2 - 5.83%, Al_2O_3 - 5.68% with weight recovery of 87.80% and %Fe recovery of 88.52%.

3.3. Effect of variation in froth depth

Tests were conducted at different froth depths ranging from 200 mm to 1000 mm. The rejection of entrained particles in the froth, in this case iron bearing minerals, depends on froth depth. Due to high superficial air velocities, some of the fine sized iron values might be misplaced into the froth phase. If there is no enough froth depth, these particles will be carried along with gangue and thus the recoveries could be affected. In the froth zone, particles are subjected to repeated detachment / reattachment events due to coalescence. During this process, particles with sufficient hydrophobicity only will float. Particles with less hydrophobicity will ultimately report to slurry phase. Table 2 shows the results of the effect of variation in froth depth. Alumina and silica are sluggishly floatable as could be seen in the conventional batch flotation cell test work wherein longer duration of flotation times of 20 - 25 minutes are required for the removal of alumina and silica to considerable extent.

Table 2. Flotation tests on pilot scale column to optimize froth depth

Froth depth (mm)	Product	Weight %	Assay, %			Distribution, %		
			Fe	SiO_2	Al_2O_3	Fe	SiO_2	Al_2O_3
200	Tailings	12.2	53.53	8.23	7.84	11.48	16.40	16.09
	Conc.	87.8	57.33	5.83	5.68	88.52	83.60	83.91
	Head (Calc.)		56.87	6.12	5.94	100.0	100.0	100.0
	Head (Assay)		56.84	5.70	6.47			
400	Tailings	2.5	48.60	12.18	9.72	2.09	4.59	4.31
	Conc.	97.5	58.30	6.49	5.53	97.91	95.41	95.69
	Head (Calc.)		58.06	6.63	5.64	100.0	100.0	100.0
	Head (Assay)		57.00	3.38	5.59			
600	Tailings	1.5	48.17	12.21	10.14	1.24	2.92	2.75
	Conc.	98.5	58.28	6.18	5.45	98.76	97.08	97.25
	Head (Calc.)		58.13	6.27	5.52	100.0	100.0	100.0
	Head (Assay)		57.90	6.30	5.28			
800	Tailings	3.0	51.12	10.96	8.87	2.60	5.03	5.12
	Conc.	97.0	59.18	6.40	5.08	97.40	94.97	94.88
	Head (Calc.)		58.94	6.54	5.19	100.0	100.0	100.0
	Head (Assay)		59.15	6.28	5.00			
1000	Tailings	1.6	47.20	13.08	11.19	1.28	3.14	3.34
	Conc.	98.4	59.30	6.57	5.26	98.72	96.86	96.66
	Head (Calc.)		59.11	6.67	5.36	100.0	100.0	100.0
	Head (Assay)		55.85	8.27	6.85			

Process parameters: pH: 9.5; Starch: 1.0 kg/t; Sokem 524C: 0.3 kg/t; Feed pulp density: 1.07
Column parameters: Superficial feed velocity: 0.64 cm/s; Superficial air velocity: 1.27 cm/s; Superficial wash water velocity: Nil

Shallow froth depth of 200 mm was found to be ideal. At relatively higher froth depths, froth drop back into slurry phase was found to take place across the froth / slurry interface. Hence, it follows that maintaining deeper froth depths may not be advantageous in this system and hence shallow froth depth of 200 mm was maintained for optimum performance.

3.4. Effect of variation in feed flow rate

Change in feed flow rate usually affects grade and recovery. Sufficient residence time is to be maintained so that entire gangue could be collected and separated. Feed flow velocities were varied from 0.43 to 1.28 cm/s and the results are presented in Table 3. Results indicate that at slurry residence time of 24 minutes corresponding to superficial feed velocity of 0.64 cm/s, concentrate assaying Fe - 57.33%, SiO_2 - 5.83%, Al_2O_3 - 5.68% with weight recovery of 87.8% could only be achieved. At higher superficial velocities of feed rate or lower residence times, lesser percent of alumina removal takes place due to insufficient retention time resulting in poor separation (Table 3).

Table 3. Flotation tests on pilot scale column to optimize feed flow rate

Superficial feed velocity (cm/s)	Product	Weight %	Assay, %			Distribution, %		
			Fe	SiO_2	Al_2O_3	Fe	SiO_2	Al_2O_3
0.43	Tailings	3.8	42.00	15.65	12.60	2.84	7.61	7.53
	Conc.	96.2	56.70	7.51	6.11	97.16	92.39	92.47
	Head (Calc.)		56.14	7.82	6.36	100.0	100.0	100.0
	Head (Assay)		53.50	9.27	7.69			
0.64	Tailings	12.2	53.53	8.23	7.84	11.48	16.40	16.09
	Conc.	87.8	57.33	5.83	5.68	88.52	83.60	83.91
	Head (Calc.)		56.87	6.12	5.94	100.0	100.0	100.0
	Head (Assay)		56.84	5.70	6.47			
0.85	Tailings	1.4	48.10	13.15	10.00	1.17	2.67	2.52
	Conc.	98.6	57.60	6.80	5.50	98.83	97.33	97.48
	Head (Calc.)		57.46	6.89	5.56	100.0	100.0	100.0
	Head (Assay)		56.00	8.30	6.69			
1.28	Tailings	1.1	48.20	9.95	9.94	0.94	1.95	1.72
	Conc.	98.9	56.90	5.57	6.32	99.06	98.05	98.28
	Head (Calc.)		56.80	5.62	6.36	100.0	100.0	100.0
	Head (Assay)		55.70	5.63	6.34			

Process parameters: pH: 9.5; Starch: 1.0 kg/t; Sokem 524C: 0.3 kg/t; Feed pulp density: 1.07
Column parameters: Froth depth: 200 mm; Superficial air velocity: 1.27 cm/s; Superficial wash water velocity: Nil

3.5. Effect of variation in wash water addition

In column flotation technology, wash water provides the bias water and the water necessary to transfer the collected solids into the launder. The bias water replaces the water draining naturally from the froth and promotes froth stability. The results obtained when wash water addition rate is varied are shown in Table 4. There appears to be no positive impact on the selectivity or improvement of the concentrate when wash water is added. Instead, it had negative effect on the process as reflected in the decrease of percent removal of alumina as superficial wash water velocity is increased. Since the nature of collector adsorption is of physical in nature, excess wash water may desorb the reagent and silica and alumina would report to slurry phase affecting the grade. An evidence of this could be seen from the relatively higher distribution of alumina and silica in the concentrate when wash water was added as against the case when it was not added.

Table 4. Flotation tests on pilot scale column to optimize wash water rate

Superficial wash water velocity (cm/s)	Product	Weight %	Assay, %			Distribution, %		
			Fe	SiO_2	Al_2O_3	Fe	SiO_2	Al_2O_3
Nil	Tailings	12.2	53.53	8.23	7.84	11.48	16.40	16.09
	Conc.	87.8	57.33	5.83	5.68	88.52	83.60	83.91
	Head (Calc.) Head (Assay)		56.87 56.84	6.12 5.70	5.94 6.47	100.0	100.0	100.0
0.03	Tailings	11.4	53.25	8.71	8.51	10.70	15.72	13.75
	Conc.	88.6	57.20	6.01	6.87	89.30	84.28	86.25
	Head (Calc.) Head (Assay)		56.75 55.78	6.32 6.14	7.07 7.21	100.0	100.0	100.0
0.06	Tailings	8.1	48.60	11.40	10.40	6.92	17.19	13.85
	Conc.	91.9	57.60	4.84	5.70	93.08	82.81	86.15
	Head (Calc.) Head (Assay)		56.87 55.78	5.37 6.14	6.08 7.21	100.0	100.0	100.0
0.12	Tailings	2.2	37.50	18.03	15.12	1.47	5.84	4.65
	Conc.	97.8	56.40	6.54	6.98	98.53	94.16	95.35
	Head (Calc.) Head (Assay)		55.98 55.80	6.79 6.76	7.16 7.18	100.0	100.0	100.0
0.15	Tailings	3.0	40.20	16.30	13.43	2.14	7.45	5.90
	Conc.	97.0	56.80	6.26	6.62	97.86	92.55	94.10
	Head (Calc.) Head (Assay)		56.30 54.50	6.56 7.72	6.82 7.58	100.0	100.0	100.0

Process parameters: pH: 9.5; Starch: 1.0 kg/t; Sokem 524C: 0.3 kg/t; Feed pulp density: 1.07
Column parameters: Superficial feed velocity: 0.64 cm/; Superficial air velocity: 1.27 cm/s;
Froth depth: 200 mm

By and large, a combination of maintaining a shallow froth depth, air flow rate that results in bubbly regime only, feed flow rate that provides enough retention time to separate gangue from values and no wash water addition should provide ideal separation conditions for this particular case.

An inter-play between all the above optimized parameters, which vary from case to case and has to be established by test work only, is the key in realizing optimum separation. Any deviation in any one of these during operation would drastically affect the separation.

3.6. Trials at optimized conditions

Table 5 provides the results obtained on the pilot scale flotation column at the optimized process and column operating parameters. This takes into account the variation of feed quality at different times and its effect on flotation column performance. It is to be noted, however, that mild variations needed to be made to the process and column operating parameters as the situation demanded to obtain the best possible grade and recovery during the flotation column operation.

Table 5. Performance of flotation column at optimized conditions

Product	Wt. %	Assay, %			Distribution, %		
		Fe	SiO_2	Al_2O_3	Fe	SiO_2	Al_2O_3
Tailings	13.1	48.15	11.80	9.88	11.03	25.16	23.79
Conc.	86.9	58.54	5.29	4.77	88.97	74.84	76.21
Head (Calc.)		57.18	6.14	5.44	100.0	100.0	100.0
Head (Assay)		58.00	6.21	5.70			

Process parameters: pH: 9.5; Starch: 1.0 kg/t; Sokem 524C: 0.3 kg/t; Feed pulp density: 1.07
Column parameters: Froth depth: 200 mm; Superficial air velocity: 1.27 cm/s; Superficial wash water velocity: Nil; Superficial feed velocity: 0.64 cm/s;

It could be noticed that the grade improvement was not substantial. Further improvement in grade of the concentrate was limited by issues related to liberation. In a detailed liberation study of the different size fractions of the sample under investigation by heavy media separation, it was observed that iron oxide mineral phases are in fairly interlocked state with alumina bearing minerals [20]. One of the important findings of earlier investigations [9] is that alumina in Indian iron ore slimes occurs in the form of two distinct mineral constituents namely, gibbsite (hydrated aluminium oxides) and kaolinite (and other clay minerals in minor quantities). It is well known that iron oxide and alumina have identical crystal structure and at times exist in solid solution (one form of interlocked state). It appears that this also had contributed to limitations in separation of alumina from iron bearing minerals from these slimes.

4. Conclusions

Screw classifier overflow slimes from an operating iron ore washing plant were subjected to reverse cationic column flotation process. On-site trials were conducted on a 0.5 meter diameter pilot scale flotation column at optimized process and operating parameters. Superficial air velocity and slurry feed flow rate / residence time were found to have significant effect on the separation. Feed assaying Fe - 58.00%, SiO_2 - 6.21%, Al_2O_3 - 5.70% could be upgraded to Fe - 58.54%, SiO_2 - 5.29%, Al_2O_3 - 4.77% with weight recovery of 86.90% and Fe recovery of 88.97%. Iron oxide and alumina have identical crystal structure and at times exist in solid solution (one form of interlocked state). It appears that this contributed to limitations in separation of alumina from iron bearing minerals from these slimes.

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