

Laboratory Column flotation studies for reduction of alumina and silica in iron ore slimes of an operating plant

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Abstract

A typical iron ore washing plant for treating iron ore fines (<10 mm) consist of sizing of the ore by dry / wet screening, washing, classification by screw classifiers followed by single or multi-stage hydrocycloning of screw classifiers overflow. The underflow of the hydrocyclones forms the concentrate which is suitable for pellets making. Since the cut-point of hydrocyclones is usually at 20 µm or less, relatively lower diameter cyclones in a cluster with parallel feeding are used. This is found to, often, result in choking of the spigots, at times, by extraneous material reporting along with the slurry. It is observed to lead to sub-optimum performance of the cyclones with loss of iron values into cyclone overflow and thereafter into tailings. Analysis of the data of an operating beneficiation plant (Beneficiation plant - 1) of M/s Jindal South West (JSW) Steel Limited, Karnataka, India over a period of one year indicated that weight recovery of the concentrate from two-stage hydrocycloning was 45.9% (with respect to feed to the cyclones) and assaying 63.43% Fe, 4.43% SiO₂ and 2.23% Al₂O₃. Considering the fineness of the slimes (d₈₀: 40.5 µm), generated as screw classifier overflow, flotation is thought to be better alternative vis-à-vis hydrocyclones to recover iron values from it. The present study aims at improving the recovery of the iron values from the screw classifier overflow by adopting reverse cationic column flotation technology.

After selecting a suitable collector, process parameters like collector and depressant dosages are optimized by statistically designed experiments on a Denver D12 flotation cell. Later, the effect of important operating parameters of flotation column like air flow-rate, froth depth and wash water on the separation process is studied and optimized. It was established that a typical screw classifier overflow analyzing 60.43% Fe, 6.88% SiO₂ and 3.26% Al₂O₃ could be improved to 63.30% Fe, 4.04% SiO₂ and 2.32% Al₂O₃ with 59.10% weight and 61.70% Fe recovery. This is an improvement of 29% in weight recovery of the concentrate at equivalent metallurgy compared to what is being obtained, by two-stage cycloning.

Keywords: Iron ore; slimes; column flotation; alumina reduction; reverse flotation

1. Introduction

The Government of India highlighted Processing and utilization of iron ore fines as a pre-requisite to meet the domestic raw material requirements for the projected 110 million tonnes of domestic steel production by the year 2019-20 in the National Steel Policy announced in November 2005 (Sharma, 2005). This would require 190 million tonnes of iron ore. The policy lays emphasis in encouraging investments in adding value to iron ore fines. It was indicated that 60% of the iron ore produced in India comes in the form of fines during the course of mining operations itself. Further, 10-12% lumps become fines while handling, loading / unloading (particularly in Bellary – Hospet sector where the ore is soft and friable) and while converting them into calibrated lump ore (CLO) for sponge / pig iron plants / exports. Also, as mining depths increase, the ores are becoming softer and increased moisture content resulting in lower grade ores which need to be processed for their optimal utilization. The fines (especially ultrafines or concentrate; <150µm) can only be made into pellets suitable to blast furnace / COREX charge and they should conform to stringent and consistent specifications.

The iron ore deposits of Bellary – Hospet sector are considered to be one of the richest iron ore deposits next to those in Orissa, Bihar and Chattisgarh. The ore bearing terrain is just south of the Bellary-Hospet railway line and comprises of Ramandurg, Kumaraswamy, Donimalai, Timmappanagudi and Devadarigudda sections along the eastern and western ranges of Sandur hills. The principle ore bearing minerals of normative composition averaged over a number of deposits of this area are hematite 70-75%, goethite/limonite 15-20% and martite 5-15% are highly oxidized. Some of the salient features of these ores are

- relatively soft nature which generate excess fines during mining, handling and processing, at times, beyond acceptable limits for subsequent processes
- high alumina content
- intrinsic association of alumina with iron bearing minerals at $-25\mu\text{m}$ rendering selective recovery of iron values almost impossible at this size range.

The typical chemical composition of these ores is given in the following Table 1.

Table 1: Typical chemical composition of ore

Constituent	Weight, %
Fe	55.0-69.0
SiO ₂	0.2-10.0
Al ₂ O ₃	0.9-15.0
TiO ₂	0.2-1.2
MnO	0.1-2.1
P	0.02-0.16
S	0.005-0.05
CaO	0.05-0.2
MgO	0.03-2.1
H ₂ O	2.0-12.0

M/s JSW Steel Limited, one of the leading producers of Steel in India outsources iron ore fines for its beneficiation plant from the above mentioned eastern and western ranges of Sandur hills. It has been established that the ore from different sources vary widely in mineralogy, chemical composition, particle size distribution and response to washability for reduction of alumina. Accordingly they have been classified as preferred, tolerable and not amenable for processing. Based on this, necessary caution is being exercised while procuring the iron ore fines for their beneficiation plant. A 3.0 Mtpa beneficiation plant has been in operation with the primary objective of reducing alumina in iron ore fines. It has two parallel streams, each with a

rated capacity of 300 tph. The unit operations in each stream (Figure 1) comprise of wet screening, classification of undersize of wet screening by a set of screw classifiers followed by 2-stage hydro-cycloning of screw classifiers' overflow at 20-microns cut-point. The underflow of 2-stage hydro-cycloning forms the concentrate which is fed to the pellet plant after dewatering by horizontal belt filter. The oversize material from screen and screw classifier fines are stock piled in the raw material yard for their usage in the downstream processes.

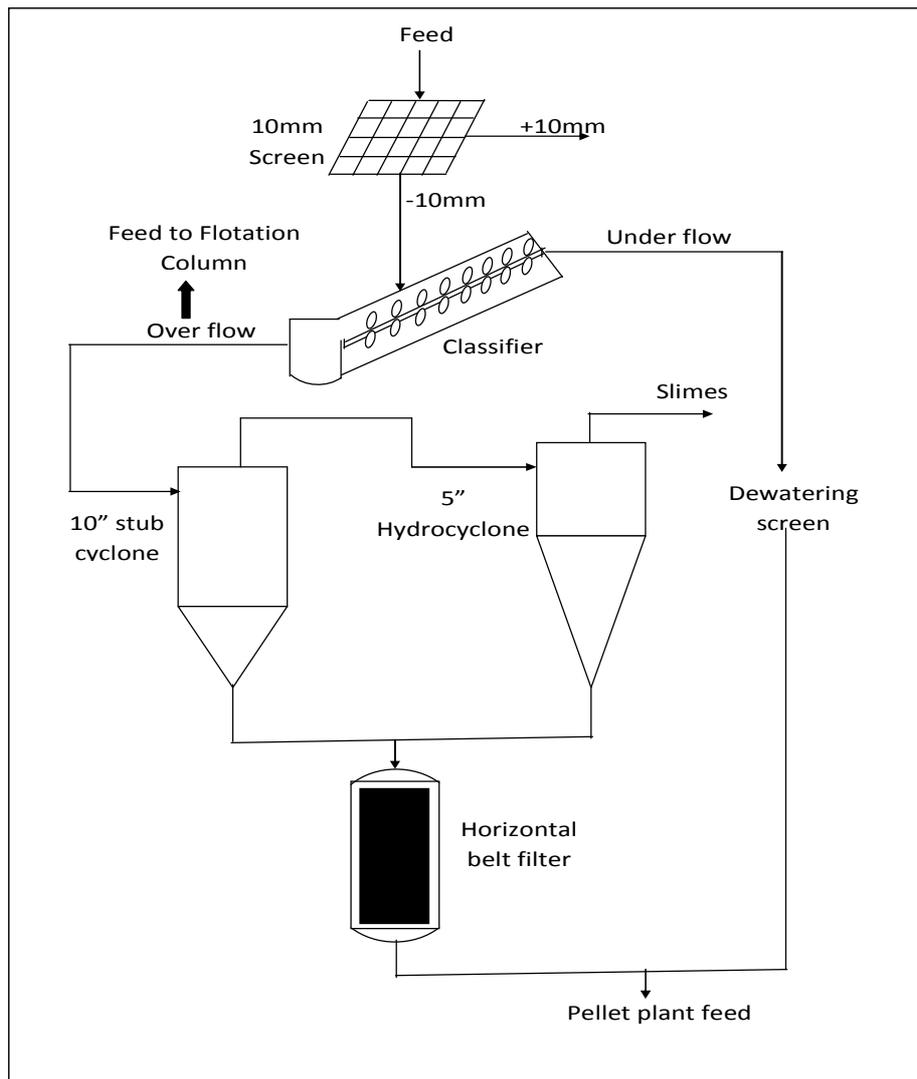


Fig.1 Flow diagram of one stream of beneficiation plant - 1

Analysis of the plant performance data over a period of one year provided the following information.

- Screen oversize (64.04% Fe & 2.31% Al₂O₃) and screw classifier fines (64.70% Fe & 2.04% Al₂O₃) were of good quality to be utilized in the downstream processes
- Screw classifier overflow (59.84% Fe & 4.16% Al₂O₃) is being subjected to 2-stage hydrocycloning to obtain a concentrate (63.43% Fe & 2.32% Al₂O₃) with weight recovery of 45%.

Since the cut-point of hydro-cyclones was at 20µm, relatively lower diameter cyclones in a cluster with parallel feeding were being used. This often resulted in choking of the spigots, at times, by extraneous material reporting along with the slurry, leading to sub-optimum performance with loss of iron values into cyclone overflow and thereafter into tailings.

Different aspects of column flotation are reviewed and its advantages over conventional flotation are well documented (Dobby and Finch, 1991; Finch, 1995; Finch and Dobby, 1990, 1991; Finch et al., 1994; Rubinstein, 1995). Similarly, iron ore flotation and application of column flotation for improvement of grade of the fines / slimes of different origin, mineral compositions and under different physico-chemical environments was studied by several investigators (Araujo et al., 2005; Eisele and Kawatra, 2007; Filippov et al., 2010; Flint et al., 1992; Houot, 1983; Lima et al., 2013; Ma et al., 2011; Pradip, 2006; Rocha et al., 2010; Rodrigues et al., 2013; Shibata and Fuerstenau, 2003; Turrer and Peres., 2010; Turrer et al., 2007; Wyslouzil et al., 1994; Yuhua and Jianwei, 2005)

Earlier studies (Rao et al., 2011; Vijaya Kumar et al., 2005a, 2005 b, 2010) on iron ores of India indicated that silica and alumina could be reduced by reverse cationic column flotation as a value addition step on a pre-concentrate obtained from an another operating beneficiation plant.

In this present study, the authors have attempted to beneficiate and recover the iron values from the screw classifier overflow slimes from the above mentioned operating iron ore washing plant by means of laboratory scale flotation column with a view to maximize the recovery of iron values at equivalent or better grade as compared to that from existing practice.

2. Materials and methods

2.1. Materials

Cationic amine collectors which are generically same but compositionally different from each other are manufactured and supplied by M/s Somu Organo-Chem Pvt. Ltd., Bengaluru, India. These cationic amine collectors are proprietary in nature and their detailed chemical composition is not revealed, but are said to be ether amine based. Their performance was evaluated and the best among them was chosen for lowering alumina content and optimizing the process parameters. Causticised maize starch was used as depressant for iron bearing minerals. It was supplied by M/s Riddhi Siddhi Gluco Biols Ltd., Ahmedabad, India. Commercial grade sodium hydroxide was used as pH regulator.

2.2 Laboratory flotation tests for evaluation and selection of collector

Flotation tests are conducted in a laboratory model Denver D12 flotation cell. Conditioning of the ore slurry is done at pH 9.5 - 10.0 and pulp density of 50% solids by adding 1.0 kg/t of causticised starch for a period of 5 minutes. The designated collector is, then, added in three stages of 0.20, 0.10 and 0.10 kg/t respectively. Before introducing the air for flotation to take place, the pulp density is reduced to 40% solids by adding additional water. After each stage-wise addition, conditioning is done for 5 minutes. Flotation is carried out for 10, 10 and 5 minutes respectively corresponding to 1st, 2nd and 3rd stages of addition of amine collector. The iron ore concentrate, remaining in the flotation cell at the end of the test and the tailings collected as froth are analysed for iron, silica and alumina.

2.3. Laboratory column flotation tests

The automated flotation column with an internal diameter of 74 mm designed and developed by CSIR -NML Madras Centre is used in the present study. The details of the flotation column are shown in Figure 2. The column shells are made up of flanged sections consisting of air injection zone, collection zone, feed point and cleaning zone/froth zone. Electronically controlled metering pumps are used to feed and discharge the slurry. The pumps are designed to deliver an accurately measured volume of slurry with an error of +/- 2%. Slurry/froth interface is maintained using Differential Pressure Transmitter (DPT). The output signal of the DPT is looped with the stroke controller of the tailing pump so that the pumping rate could be automatically varied to maintain the interface level at a fixed froth depth. Under steady state

conditions, the interface level could be maintained at a constant height to within +/- 1cm. Purge rotameters with differential pressure regulators are used to control the flow of air and water.

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. After stabilization with water, the reagents' conditioned slurry is pumped into the column at desired flow rate. The slurry at 15% solids is conditioned initially with sodium hydroxide in a conditioner with a conditioning time of 180 seconds. In the second stage, starch is added and conditioned for further 180 seconds. This slurry is further conditioned with amine collector. The reagentized slurry is fed to the column and allowed to run for a minimum period of 3 - 4 residence times. Samples are drawn under near-steady state conditions. Both the process parameters and column operating conditions are recorded before collecting the samples. Samples are analyzed for Fe, SiO₂, Al₂O₃ and loss on ignition (LOI) by adopting standard methods of analyses

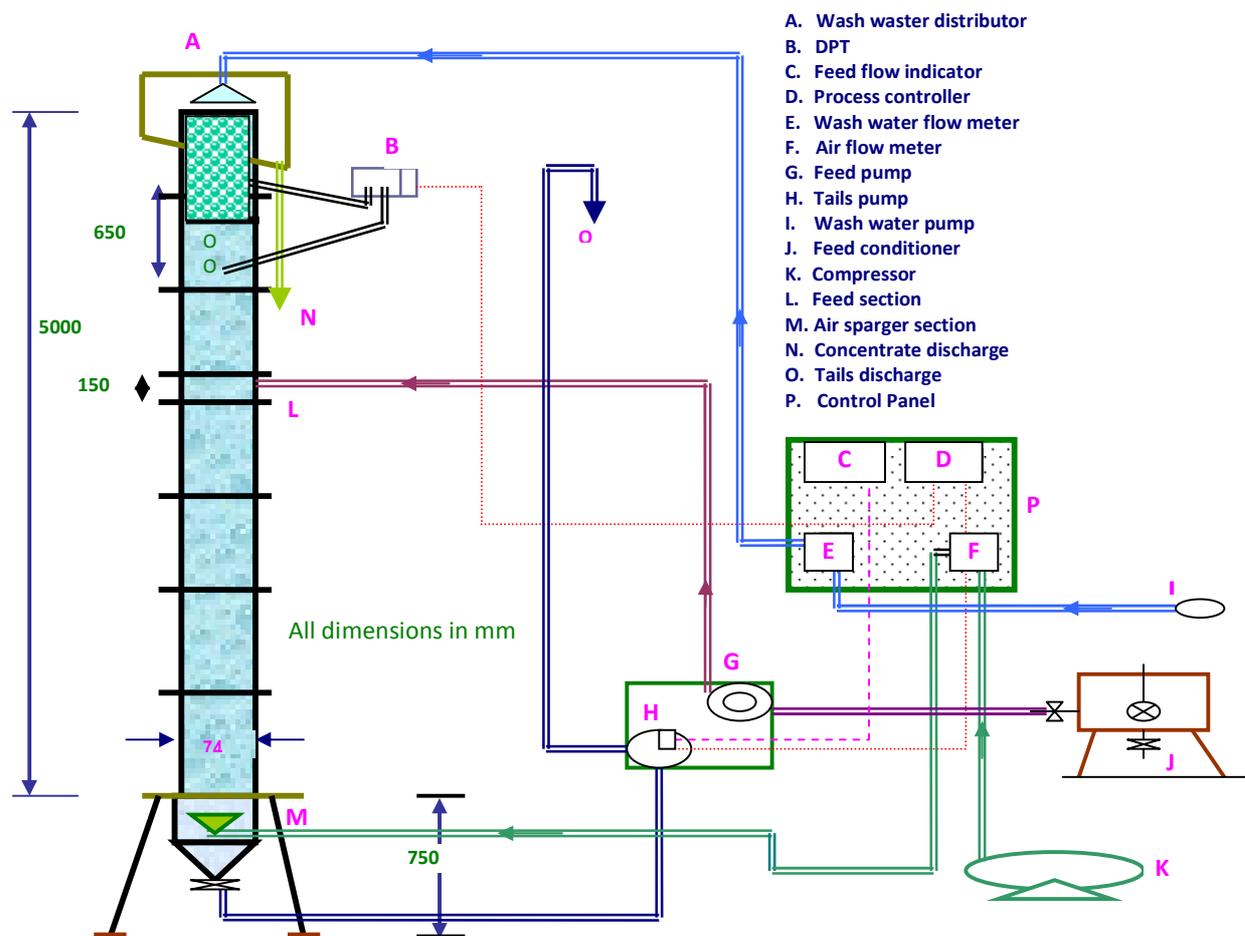


Fig. 2 Schematic diagram of CSIR – NML laboratory scale flotation column

3. Results and Discussion

3.1 Evaluation & selection of collector

Selection of appropriate reagents depends on various factors such as technical performance, price and availability. The chemical composition of the collector has a vital role to play in the reduction of alumina and silica in general and alumina in particular in the iron ore fines. However, the dosage and efficiency of the reagent are of utmost importance. The manufacturer of Sokem family of cationic collectors developed alumina-specific cationic collectors to suit the type of iron ore slimes under investigation in technical collaboration with CSIR-NML Madras Centre (CSIR-NMLMC). Development of new reagents has been taken up by Somu Organo-Chem Pvt. Ltd., based on the feedback received from NMLMC on the efficacy of collectors supplied by them from time to time during the course of the investigation. Eight

cationic collectors which are generically same but of varying chemical composition were evaluated and tested for their performance. Basically they are ether amines, each of them varying in carbon chain length of fatty amines. The performance of the reagents was evaluated based on modified version of Selectivity Index (SI) derived by Douglas (1962). According to his definition, an index of 100 is indicative of a perfect separation between the valuable minerals and the gangue; an index of zero indicates no separation. The numerical value of this selectivity index, as defined, is adjusted for variations in head assay. As such, therefore, it served as a useful measure of the efficacy of the separation process on a number of different samples as well. It is given by

$$SI = [(R-C) * (c-f) * 100] / [(100-C) * (c_{max} - f)]$$

Where

C = % weight of the concentrate

R = % recovery of iron in the concentrate

c = % iron of the concentrate

c_{max} = maximum (theoretical) iron in the concentrate

f = % iron of the feed

For each collector, four repeat experiments were carried out, and the average of them was considered for calculating the SI. The details of the results are given elsewhere (Vijaya Kumar and Gopalkrishna, 2012). The relative values of SI for different collectors are presented in Figure 3.

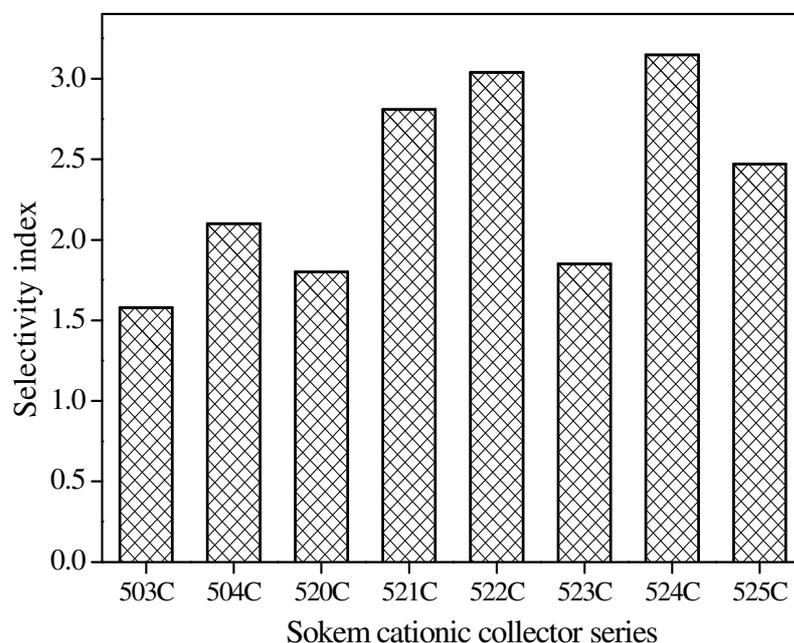


Figure 3 Comparison of performance of Sokem series of cationic collectors

All the collectors were not evaluated at a time, but the development and improvement on the reagents' performance front were made sequentially as shown in the above figure based on the feedback received as the investigation was progressing. During the initial stages of investigation on reagents' dosage optimization, Sokem 521C was proved to be better. By the time the laboratory and pilot scale flotation column studies were planned, Sokem 522C was found to have better selectivity and frothing characteristics and hence was preferred to 521C during the laboratory and pilot scale flotation column studies. By the fag end of the investigation, another collector in this suite, Sokem 524C, was developed which proved to be the best among all. This, however, could not be tested on a pilot scale studies due to paucity of time. Superior performance of these reagents over the rest could be attributed to relatively better hydrophobicity imparted to the gangue in the ore under investigation, thereby affecting the selectivity of separation.

Important process parameters such as collector's dosage (Sokem 521C) and depressant dosage (starch) were optimized at 0.3 kg/t and 1.0 kg/t respectively in the pH range of 9.5 - 10.0 on bench scale studies using Denver D12 flotation cell and statistically designed experiments.

Having established the optimum reagent conditions, tests were conducted on laboratory scale 74mm diameter flotation column using the same screw classifier overflow material. Effect of variation of column operating parameters like air flow rate, froth depth and wash water was studied with an intention to optimize them. By now, the collector Sokem 522C has been evaluated and found to be at par with Sokem 521C. Hence, it was used in this test work because of its better frothing characteristics.

3.2 Effect of variation in air flow rate

Table - 2 shows the effect of variation of superficial air velocity (or air flow rate) on column flotation of iron ore fines. The experimental conditions and the test results were shown in the table.

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. It is to be noted that column should operate in a bubbly flow regime, where gas hold-up varies linearly with air rate. It was observed that the superficial air velocity of 1.17 cm/s was found to be optimum to obtain better grade and recovery, as also reflected by the better selectivity index of 1.81. Above the superficial air velocity of 1.17 cm/s, the signs of turbulent flow regime having set in were appearing at 1.56 cm/s and this is reflected in the decrease of selectivity index to 1.65.

3.3 Effect of variation in froth depth

Froth depth was adjusted to the required value using YS 170 controller looped to pressure transmitter. Experiments were conducted at different froth depths ranging from 100 mm to 500 mm and the results obtained were presented in Table - 3. The rejection of entrained particles, 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 not enough froth depth, these particles will be carried along with gangue and thus the recoveries may 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.

There was not much variation in the grade of the concentrate as the froth depth was increased from 100 mm to 500 mm. But the weight recovery of the concentrate decreased from 68.80% to 56.30% during this variation in the froth depth which was also reflected in the decrease of the selectivity index from 1.25 at 100 mm to 1.04 at 500 mm froth depth. This could have been due to froth drop back into slurry phase across the froth / slurry interface at relatively higher froth depth. Hence, it follows that maintaining deeper froth depths may not be advantageous in this system and hence shallow froth depths from 100 mm to 300 mm, in this case, need to be maintained to obtain better recovery at equivalent grade.

Table 3 Flotation Tests on lab column to optimize froth depth

<u>Process parameters</u>	<u>Column parameters</u>
pH: 9.5 - 10.0	Superficial feed vel.: 0.5 cm/s
Starch: 1.0 kg/t	Superficial air vel. : 1.17 cm/s
Sokem 522C: 0.3 kg/t	Superficial wash water velocity : Nil

Conditioning at 50% solids for 5 mins. for each reagent addition

Flotation at 20% solids

Froth depth (mm)	Selectivity Index	Product	Wt. %	Assay, %			Distribution, %		
				Fe	SiO ₂	Al ₂ O ₃	Fe	SiO ₂	Al ₂ O ₃
100	1.25	Tailings	31.20	57.75	8.17	4.54	29.5	43.8	44.9
		Conc.	68.80	62.70	4.76	2.53	70.5	56.2	55.1
		Head (Calc.)		61.16	5.82	3.16	100.0	100.0	100.0
		Head (Assay)		60.43	6.88	3.26			
300	1.29	Tailings	33.50	56.98	8.78	5.08	31.5	47.4	49.0
		Conc.	66.50	62.57	4.91	2.66	68.5	52.6	51.0
		Head (Calc.)		60.70	6.21	3.47	100.0	100.0	100.0
		Head (Assay)		60.43	6.88	3.26			
500	1.04	Tailings	43.70	57.39	8.69	5.02	41.6	57.8	60.1
		Conc.	56.30	62.60	4.92	2.59	58.4	42.2	39.9
		Head (Calc.)		60.32	6.57	3.65	100.0	100.0	100.0
		Head (Assay)		60.43	6.88	3.26			

3.4 Effect of variation in wash water addition

Wash water was introduced through shower- like arrangement fixed at a distance of 30 mm from the top-end of the column so as to have even distribution of sprinkling water across the column cross sectional area.

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. Thus the wash water rate controls the recovery by replacing feed water in the froth and thereby hydrophilic iron ore particles. Usually, wash water rates are kept as low as possible to minimize unnecessary dilution of reagents. Since the nature of collector adsorption is of physical in nature, excess wash water may desorb the reagent and silica and alumina bearing minerals would report to slurry phase affecting the grade.

The results obtained when wash water addition rate was varied at optimized superficial air velocity and froth depth were shown in Table - 4. There appears to be no positive impact on

the selectivity or improvement in the metallurgy of the concentrate when wash water was added. Instead, it had negative effect on the process as reflected in the decrease of selectivity index as superficial wash water velocity was increased. This could have been due to reporting of alumina and silica back into the slurry phase from the froth phase as the wash water was increased which resulted in diluting the grade of the concentrate. 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 where it was not.

Table 4: Flotation Tests on lab column to optimize wash water addition

<u>Process parameters</u>				<u>Column parameters</u>					
pH: 9.5 - 10.0				Superficial feed vel.: 0.5 cm/s					
Starch: 1.0 kg/t				Superficial air vel. : 1.17 cm/s					
Sokem 522C: 0.3 kg/t				Froth depth : 300 mm					
Conditioning at 50% solids for 5 mins for each reagent addition.									
Flotation at 20% solids									
Wash water superficial velocity (cm/s)	Selectivity Index	Product	Wt. %	Assay, %			Distribution, %		
				Fe	SiO ₂	Al ₂ O ₃	Fe	SiO ₂	Al ₂ O ₃
Nil	1.29	Tailings	33.50	56.98	8.78	5.08	31.5	47.4	49.0
		Conc.	66.50	62.57	4.91	2.66	68.5	52.6	51.0
		Head (Calc.)		60.70	6.21	3.47	100.0	100.0	100.0
		Head (Assay)		60.43	6.88	3.26			
0.30	1.05	Tailings	22.60	55.90	9.45	5.62	20.9	33.3	36.1
		Conc.	77.40	61.83	5.53	2.91	79.1	66.7	63.9
		Head (Calc.)		60.49	6.42	3.52	100.0	100.0	100.0
		Head (Assay)		60.43	6.88	3.26			
0.36	0.53	Tailings	26.50	56.96	8.95	5.22	25.1	37.3	39.6
		Conc.	73.50	61.41	5.43	2.87	74.9	62.7	60.4
		Head (Calc.)		60.23	6.36	3.49	100.0	100.0	100.0
		Head (Assay)		60.43	6.88	3.26			

Table 5 provides the performance of laboratory scale flotation column at optimized process and column operating parameters.

<u>Process parameters</u>	<u>Column parameters</u>
pH: 9.5 - 10.0	Froth depth : 300 mm
Starch: 1.0 kg/t	Superficial air velocity : 1.17 cm/s
Sokem 522C: 0.3 kg/t	Superficial wash water velocity : Nil
Feed pulp density : 20% solids	Superficial feed velocity : 0.50 cm/s

Table 5: Performance of laboratory flotation column at optimized conditions

Selectivity Index	Product	Weight, %	Assay, %			Distribution, %		
			Fe	SiO ₂	Al ₂ O ₃	Fe	SiO ₂	Al ₂ O ₃
1.81	Tailings	40.90	56.73	9.05	5.29	38.3	60.8	61.2
	Concentrate	59.10	63.30	4.04	2.32	61.7	39.2	38.8
	Head (Calculated)		60.61	6.09	3.53	100.0	100.0	100.0
	Head (Assay)		60.43	6.88	3.26			

Table 6 shows the comparison of recovery and grade of the concentrates obtained from two-stage cyclones and flotation column operation.

Table 6: Comparison of recovery & grade of concentrates from cyclones & flotation column

Product	Weight recovery, %	Assay, %			Recovery, % Fe
		Fe	SiO ₂	Al ₂ O ₃	
Cyclone underflow	45.9	63.43	4.43	2.32	48.65
Lab.column Concentrate	59.1	63.30	4.04	2.32	61.70
Feed		60.43	6.88	3.26	

4. Conclusions

The amenability of reverse cationic column flotation technology for recovery of iron values from screw classifier overflow or slimes (d_{80} : 40.5 μm) of an operating washing plant has been established by laboratory scale studies. Selection of proper collector plays a key role and needs to be evaluated based on the type of ore and nature of the gangue. A typical screw classifier overflow analyzing 60.43% Fe, 6.88% SiO_2 and 3.26% Al_2O_3 could be improved to 63.30% Fe, 4.04% SiO_2 and 2.32% Al_2O_3 with 59.10% weight and 61.70% Fe recovery. This is an improvement of 29% in weight recovery of the concentrate at equivalent metallurgy compared to what is being obtained, by two-stage cycloning.

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