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T.V. Vijaya Kumar, D.S. Rao, S. Subba Rao, P. More, Y.S. Reddy, S. Prabhakar and G. Bhaskar Raju, India

Beneficiation of Iron Ore Fines by Conventional Flotation, Flotation Column and Dual Extraction column – A pilot scale study

Abstract

Goa state in India is endowed with large deposits of low-grade iron ores, geologically formed from lower Dharwar super group of Archaean rocks. Petrological studies have revealed that silica and alumina are the main gangue minerals where alumina exists as fine clay and adherent material interspersed in the ore body and in some cases both silica and alumina interlocked with iron ore particles. Efficiency of conventional flotation, column flotation and dual extraction column for separation of gangue was studied on a pilot scale at Greater Ferromet Beneficiation Plant of M/s. Sociedade De Fomento Industrial Ltd., Goa. Reverse flotation process was adopted wherein gangue minerals silica and alumina were floated using cationic amine collector. Sodium hydroxide and starch were used to regulate the slurry pH and to depress iron ore, respectively. Various iron ore samples drawn from different mines and their mixtures were subjected to the test work. Results of pilot scale tests clearly indicate the superiority of flotation column vis-à-vis conventional cells and dual extraction column. Iron ore concentrate assaying 67 % Fe and 2 % SiO_2 and Al_2O_3 with a recovery of 85 - 90 % could be obtained by single stage flotation column operation.

1 Introduction

The centenary of flotation process is going to be celebrated by the Australian Institute of Mining and Metallurgy in June 2005. The process had made major strides in the development of different types of flotation cells in this century-long history (Finch, 1995; Rubio et al., 2002). The flotation column developed by Boutin was the first in the new generation of apparatuses based on a countercurrent flow of slurry and air bubbles. The advantages of flotation columns over conventional cells have been well documented in the literature (Boutin and Wheeler, 1967; Coffin and Miszczak, 1982; Michael and Ian, 1989; Donald and Harold, 1991; Bhaskar Raju et al., 1993, 1994; Prabhakar and Bhaskar Raju, 1998; Bhaskar Raju and Prabhakar, 2000). Since then, different types of flotation columns like Cyclonic Flotation Column, Dual Extraction Column etc., have been developed for special applications in coal and mineral separation.

Recently there is a marked shift from iron ore lumps to fines in iron making. The processes, which permit the use of ore fines, have become commonplace in iron and steel making industry. Further, recovery of values from fines generated during mining, milling and washing wastes is being practiced at all mines. Usual physical separation techniques like Low Intensity Magnetic Separation (LIMS), Medium Intensity Magnetic Separation (MIMS) and High Gradient Magnetic Separation (HGMS) are inadequate to address the problems associated with fines and interlocked particles. Flotation is the most suitable technique for the selective separation of iron ore fines from gangue minerals. Direct flotation of iron ore using oleic acid is relatively expensive in terms of high reagent consumption and entrapment of gangue minerals in the froth. Therefore, beneficiation of iron ore fines by reverse flotation using cationic collectors is gaining wider application in the mining industry, as a final cleaner process, to obtain high grade concentrate, as value addition technique to improve the grade of the pre-concentrate obtained by physical beneficiation. When used as cleaner process, this has the advantage of relatively less reagent consumption in the process due to less quantity of gangue that has to be separated.

The State of Goa in India is endowed with large reserves of proven hematite iron ores. These are being beneficiated by washing, scrubbing, hydrocycloning and magnetic separation. Alumina was found to exist as fine clay and in some cases both silica and alumina were interlocked with iron ore particles and their removal by conventional methods was found to be difficult. M/s. Sociedade De Fomento Industrial Limited, Goa, India are able to produce concentrates assaying 64 - 65 % Fe with 5 % alumina plus silica, from their magnetic circuit after installing High Gradient Magnetic Separator at their Greater Ferromet Beneficiation Plant. The schematic flowsheet of the plant is shown in Fig. 1. Different products generated in their beneficiation circuit are given in Table 1. In order to further improve their concentrate quality, pilot scale studies were carried out using Conventional Flotation Cell, Flotation Column designed and developed by National Metallurgical Laboratory Madras Centre, India and Dual Extraction Column (DEC); and their comparative performance was evaluated.

2 Experimental

2.1 Pilot Scale Flotation Column

The flotation column 0.5 m dia and 8 m height designed and developed by NML Madras Centre is shown schematically in Fig. 2. The feed is introduced into the column at a point from a height of about one-third from the top. Slurry/froth interface is maintained using differential pressure transmitter (DPT)

T.V. Vijaya Kumar, D.S. Rao, S. Subba Rao, S. Prabhakar* and G. Bhaskar Raju, National Metallurgical Laboratory Madras Centre, CSIR Madras Complex, Taramani, Chennai - 600 113, India
Y.S. Reddy and P. More, Sociedade De Fomento Industrial Limited, Margao - 403 601, Goa, India
*Corresponding author
Tel.: +91-44-22542077; Fax: +91-44-22541027
e-mail: swaprabha@yahoo.com
Details about the authors on page 126

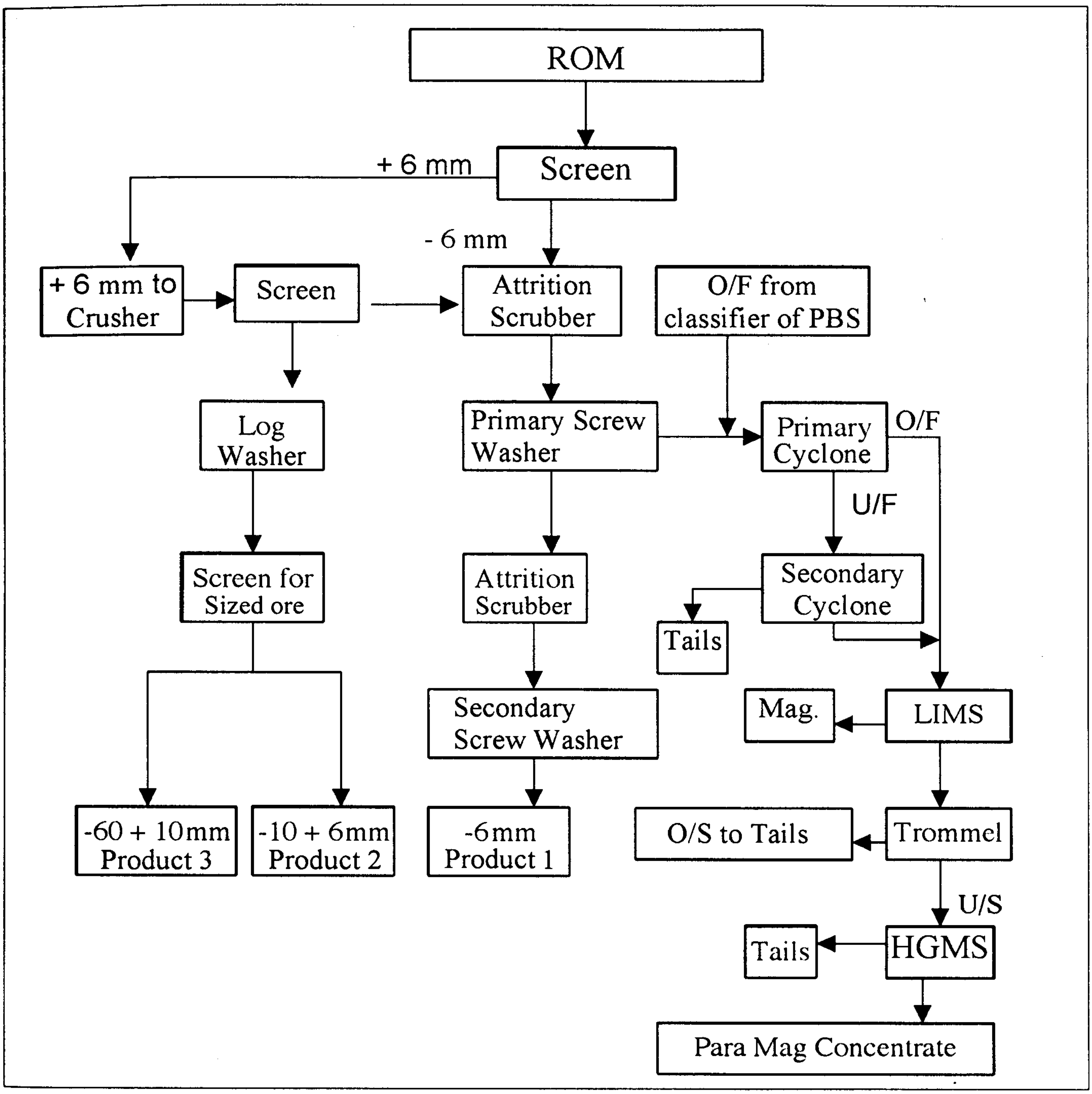


Fig. 1: Schematic Flow Sheet of Primary Beneficiation Plant of Fomento

mounted to column shell. The output signal of the DPT is looped to an Electro-pneumatic valve through a programmable process controller. Changing the set point of the process controller could alter the froth depth. Indigenously designed spargers and wash water spray arrangement were used in the present study. Air flow rate, slurry flow rate and wash water flow rates were measured using suitable flow meters. Gas hold-up of 11 - 13 % is maintained in the column during the studies and the bubble size is estimated to be 0.101 cm. Froth depth of 50 cm was maintained throughout the test work.

2.2 Dual Extraction Column

This column has two chambers, one upper and the other lower, each of 0.275 m^3 (Fig. 3). The feed is introduced into a feed well,

Tab. 1: Different commercial grade iron ore products and tailings generated at Greater Ferromet Beneficiation Plant

Description	[illegible] % Rec	Fe	Al_2O_3	[illegible]
Concentrate	46.0	62.3	1.84	3.57
-60 + 10 mm	11.8	61.5	1.88	2.80
-10 + 6 mm	2.1	60.3	2.19	3.28
Para magnetics 30 to 10 mm	12.2	65.3	1.34	2.85
sized lumps	3.2	59.0	2.50	3.20
Tailing	24.7	46.4	6.24	11.68
[illegible]	[illegible]	[illegible]	[illegible]	[illegible]

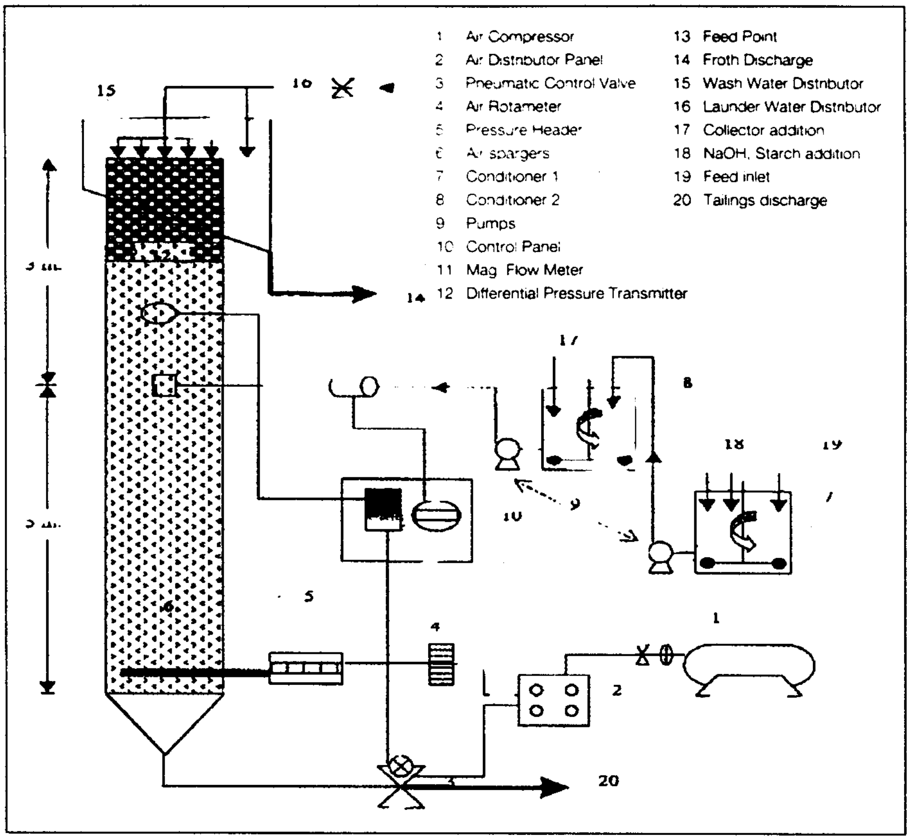


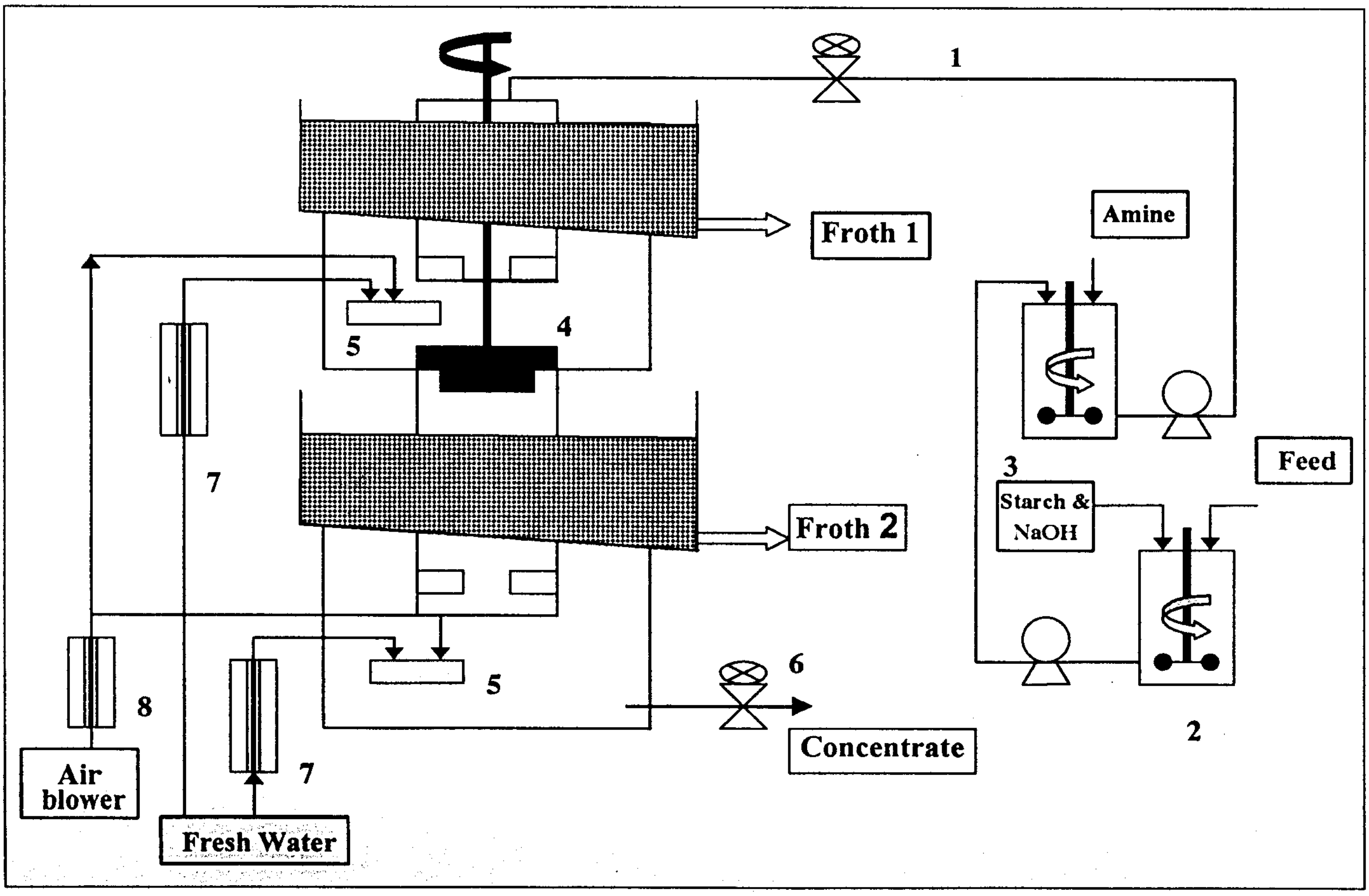
Fig. 2: Schematic assembly and layout of flotation column set-up

which in turn is equally distributed through a bottom plate. Air bubbles are created by introducing fresh water at 2.11 kg/cm^2 into two header pipes with hose attachments leading into multi-infusers specially designed to create a high volume of finely divided air bubbles. A blower to combine in the infusers similar to turbo system provides low-pressure air at 0.35 kg/cm^2. As the rising air bubbles move upward in the chamber, separation of values from the gangue takes place and tailings reports to a launder for removal.

The concentrate exits the upper chamber via a discharge throat at a controlled rate by means of a dart or plugs valve positioned in the opening and connected to a movable shaft capable of up or down movement through a thread bar and adjusting wheel arrangement. The non-separated gangue still present in the concentrate now has sufficient residence time in the lower chamber where an identical separation as described for the upper chamber takes place.

The tailings from this lower chamber overflow into the launder and combines with tailings from the upper chamber for further dewatering. The concentrate exits the lower chamber via a concentrate discharge pipe. A froth height of 7.5 - 10.0 cm is maintained.

Fig. 3: Schematic diagram of Dual Extraction Column; **1** Feed Control Valve, **2** Conditioner 1, **3** Conditioner 2, **4** Dart Valve, **5** Air water infuser, **6** Control Valve, **7** Water flow meter, **8** Air flow meter



2.3 Conventional Flotation

A conventional flotation cell (Outokumpu make) of 1 m^3 volume is used and a froth height of 5.0 cm is maintained during the investigation.

2.4 Materials

M/s. Ideal Speciality Chemicals, Mumbai, India, supplied commercial grade cationic amine based collectors Chem-540F and Chem-550F used in the investigation. These are basically fatty amines incorporating acetate groups for better water solubility. Sodium hydroxide and starch of commercial grade were used as pH regulator and depressant for iron ore respectively.

Concentrate from HGMS is tapped to the first conditioner in which sodium hydroxide and starch are added. This slurry is further pumped to the second conditioner where it is conditioned with amine collector. Minimum residence time of 180 seconds is maintained in both the conditioners. The conditioned slurry of approximately 20 % solids is pumped through flow meter and the feed distributor box attached to the respective flotation cells. Initially, the flotation cell is allowed to run for a minimum of 3 - 4 residence times or till the percent solids discharged through the valve is constant. Samples are drawn only after reaching the steady state conditions.

3 Results and Discussion

3.1 Characterization

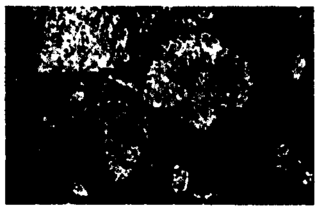
Color of lump iron ore samples collected from the mines was found to vary from reddish brown to yellow. They were found to be associated with argillaceous/arenaceous material. This extraneous material was found to contribute extra silica and alumina to the finer fractions of ore during processing. Microscopic examination of the iron ore samples revealed the presence of hematite, goethite, martite, magnetite and limonite as iron minerals while quartz and clay constituted the silicates. Hematite / martite and magnetite were found to be granular while goethite showed oolitic, pisolitic and colloform texture. The size of hematite / martite grains ranged from 9 to 180 µm while that of silicate from 5 to 80 µm. Silicate grains were present as inclusions within goethite and hematite and vice-versa (Figs. 4 and 5). Sieve and chemical analyses of feed to column and concentrate are shown in Table 2. It is evident that gangue particles are interlocked with iron minerals above 150 µm fraction. Though this fraction is relatively very low by weight, contribution of gangue is very high in this fraction. Most of the free gangue is in finer fraction i.e., below 45 µm.

3.2 Preliminary Tests

Preliminary flotation tests were conducted in Denver Sala D12 laboratory flotation cell to test the selectivity of collectors viz., Chem-540F and Chem-550F and the results are shown in Tables 3 and 4 respectively. From the results it is observed that collector Chem-550F is selective towards Al_2O_3 whereas collector Chem-540F is suitable for the elimination of both SiO_2 and Al_2O_3. It is also observed that reagent dosage of 0.2 kg/t is sufficient to reduce SiO_2 and Al_2O_3 to the desired levels and by increasing the collector dosage above 0.2 kg/t, further reduction in gangue is achieved but at the cost of recoveries. Similarly

Fig. 4: (lef) Hematite (H) grains within a matrix of clay and goethite; Patches of clay are seen as black; Reflected light x150

Fig. 5: (right) Silicate matrix within goethite (G). Fine inclusions of hematite (white grains) Within the silicates (S); Reflected light x150



Tab. 2: Sieve and Chemical analysis of column feed and concentrate

[illegible]	Sieve [illegible]	Column Feed				Column Concentrate			
		[illegible]	% Fe	Al_2O_3	[illegible]	Wt %	[illegible]	Al_2O_3	SiO_2
1	-300+150	00.36	59.6	NA	NA	00.17	58.7	2.64	5.39
2	-150+75	21.96	66.5	1.07	1.45	20.08	67.3	0.60	1.10
3	-75+45	28.61	67.0	0.90	1.45	35.74	68.0	0.74	1.00
4	-45	49.07	64.7	1.47	2.83	44.01	66.3	1.10	1.44
[illegible]	[illegible]	[illegible]	[illegible]	1.21	[illegible]	100.00	[illegible]	0.87	1.22
[illegible]	[illegible]	[illegible]	[illegible]	1.22	[illegible]	100.00	[illegible]	[illegible]	[illegible]

Tab. 3: Lab scale Conventional flotation test results using Chem-540F as collector

Process Parameter	Slurry pH:	8.50
	Starch:	1.0 kg/t
	NaOH:	0.05 kg/t

Feed: Fe: 64.8 %, Al_2O_3: 1.57 %, SiO_2: 2.76 %

[illegible]	[illegible]	[illegible]		Concentrate Assay (%)			Tails Assay (%)			Fe % Rec
		Conc	Tails	Fe	Al_2O_3	SiO_2	Fe	Al_2O_3	SiO_2	
1	0.20	88.0	12.0	66.7	0.85	0.67	56.5	4.87	7.87	90.3
2	0.30	77.9	22.1	66.9	0.99	0.64	59.7	3.04	6.49	80.2
3	0.40	70.5	29.5	67.1	0.79	0.62	61.6	2.57	5.04	72.4
4	0.50	60.7	39.3	67.3	1.00	0.57	62.7	2.02	4.02	72.4

Tab. 4: Lab scale conventional flotation test results using Chem-550F as collector

Process Parameter		Slurry pH:	8.50							
		Starch:	1.0 kg/t							
		NaOH:	0.05 kg/t							
Feed: Fe: 64.8 %, Al_2O_3: 1.57 %, SiO_2: 2.76 %										
Sl No	[illegible]	Wt (%)		Concentrate Assay (%)			[illegible]			[illegible]
		Conc	Tails	Fe	Al_2O_3	SiO_2	Fe	Al_2O_3	SiO_2	
1	0.25	87.2	12.8	66.6	1.01	1.38	56.7	3.95	9.16	88.9
2	0.40	78.3	21.7	67.1	0.87	1.11	58.6	2.40	7.83	80.6
3	0.50	74.6	25.4	67.3	0.82	0.91	60.0	2.68	6.33	76.6
4	0.75	69.3	30.7	67.4	0.79	0.83	60.7	2.17	5.68	71.5

other process parameters such as pH of the slurry and starch dosage were studied and optimized. The optimum results are achieved by maintaining the slurry pH between 8.5 - 9.0, which is in conformity with the solution chemistry of amines and surface properties of iron minerals, SiO_2 and Al_2O_3. It is well known that amines are positively charged molecules up to pH 10.0. By adjusting the slurry pH to 8.5 to 9.0, all particles (silica, alumina and iron oxides) will acquire negative charge and dispersed due to electrostatic repulsion. When starch is added subsequently, it adsorbs on surface sites of iron oxide particles and reduces the negative character of the iron oxide particles. At this stage when amine is added, it could adsorb specifically on silica and alumina particles making them hydrophobic. Thus, after establishing the process parameters of reverse flotation of iron ore fines in the conventional laboratory flotation cell, a series of tests were conducted in the 7.5 cm diameter laboratory size flotation column to optimize the column parameters. The overall optimum conditions obtained for target grade of 66 to 67 % Fe is given in Table 5.

3.3 Pilot Scale Tests

At optimum conditions, continuous tests were conducted using 0.5 m diameter flotation column, 0.275 m^3 dual extraction column and 1 m^3 conventional flotation cell, on the concentrates produced in the plant by HGMS and the results are presented in Table 6. The results clearly suggest that the flotation column is superior over dual extraction column and conventional flotation, for the beneficiation of iron ore fines. However, in all the three systems the recoveries are comparable. Though, the dual extraction column produces concentrate of the grade on par with the flotation column, the quality of concentrate is inferior. The amounts of SiO_2 and Al_2O_3 in the concentrates are much lower in the case of flotation column.

Tab. 5: Optimized column flotation parameters

Parameter	Value
(a) Process parameters	
Chem-540 (Collector)	0.2 kg/t
Slurry pH	8.5 - 9.0
Starch	0.5 kg/t
NaOH	0.07 kg/t
(b) Operating parameters	
Superficial velocity of air	1.02 cm/s
Superficial velocity of wash water	0.06 cm/s
Superficial velocity of slurry feed	1.1 cm/s
Froth depth	75 - 100 cm
Percent solids	20
Pulp density of feed	1.19

Thus, it is imperative that flotation column would be a better option over dual extraction column and conventional flotation under the operating conditions mentioned. Better quality of concentrate by flotation column could be attributed to relatively higher froth bed and wash water addition at a positive bias that result in qualitative change in the froth of flotation column. The wash water addition improves the froth rheology and product discharge. It also replaces the draining inter-film liquid, thus promoting froth stability. Furthermore, secondary cleaning resulting from the displacement of less hydrophobic particles (iron ore) by more hydrophobic particles (alumina and silica), is typical for the upper portion of the froth with high gas hold up and insufficient bubble surface area.

4 Conclusions

Microscopic examination of iron ore samples of Goan origin in India inidicated the presence of hematite, goethite, martite and limonite as iron minerals and quartz and clay as silicate gangue. Presence of silicate within goethite and hematite and vice versa was also observed. Among the two commercially available cationic collectors tested, Chem-540F was found suitable for reduction of both silica and alumina. Process and operating parameters have been optimized in a conventional flotation cell as well as on a 7.5 cm flotation column. Pilot scale studies on iron ore samples drawn from different mines using 0.5 m diameter flotation column showed that a concentrate of 67 % Fe with alumina and silica of around 2 % and with Fe recoveries of 85 - 90 % could be achieved in a single stage operation from a feed of 64 - 65 % Fe and 4 - 5 % alumina plus silica.

The performance of Dual Extraction Column was found to be at par with that of flotation column. However, if the reduction of the gangue is the main criterion, then it appears flotation column has superior advantage. This could be attributed to greater froth depth, which could be manipulated and maintained easily in flotation column and wash water addition that aids in cleaner and stable froths.

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Tab. 6: Results of continuous test runs

(a) Flotation Column

S. No.	Feed Assay (%)			Conc. Assay (%)			Tails % Fe	Fe % Rec.	Al_2O_3 + SiO_2
	Fe	Al_2O_3	SiO_2	Fe	Al_2O_3	SiO_2			
1	64.8	1.51	2.82	66.7	1.15	0.62	59.5	75.8	1.77
2	66.7	1.36	1.99	67.4	1.04	0.90	56.7	94.4	1.94
3	65.0	1.14	3.18	68.1	0.86	0.59	61.2	84.5	1.45
4	66.0	1.24	2.21	67.2	1.09	0.86	58.6	87.6	1.95
5	65.7	0.93	2.73	67.6	0.77	0.91	58.6	81.1	1.68
6	65.4	1.28	2.67	67.2	1.10	0.71	55.7	94.2	1.81

(b) Dual Extraction Column

S. No.	Feed Assay (%)			Conc. Assay (%)			Tails % Fe	Fe % Rec.	Al_2O_3 + SiO_2
	Fe	Al_2O_3	SiO_2	Fe	Al_2O_3	SiO_2			
1	64.5	1.06	4.14	66.3	0.76	2.15	47.1	93.2	2.91
2	64.4	1.31	4.54	66.4	1.20	1.72	44.0	93.9	2.92
3	64.8	1.75	4.03	66.4	1.20	2.17	45.0	94.8	3.37
4	64.3	1.44	3.91	66.1	1.17	1.86	42.9	94.8	3.03
5	65.2	1.17	2.64	65.9	1.03	1.75	53.0	95.6	2.78
6	64.8	1.46	2.67	66.2	1.20	1.55	53.0	91.3	2.75

(c) Conventional Flotation Cell

S. No.	Feed Assay (%)			Conc. Assay (%)			Tails % Fe	Fe % Rec.	Al_2O_3 + SiO_2
	Fe	Al_2O_3	SiO_2	Fe	Al_2O_3	SiO_2			
1	64.9	1.24	2.95	65.8	1.19	2.20	39.1	98.0	3.39
2	65.3	1.18	2.20	66.2	1.03	2.11	58.1	90.1	3.14
3	65.6	1.10	2.37	66.2	0.94	1.74	52.4	96.5	2.68
4	65.7	1.07	1.45	66.3	0.95	1.12	59.3	92.3	2.17
5	64.9	1.31	2.47	65.9	1.02	1.61	60.6	82.4	2.63
6	65.7	1.07	2.07	66.4	1.03	1.79	57.3	93.3	2.82

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