International Journal of Engineering Research
Volume No.4, Issue No.11, pp : 604-608
ISSN:2319-6890)(online),2347-5013(print)
01 Nov. 2015

Evaluation of Flotation Collectors in Developing Zero Waste Technology for Processing Iron Ore Tailings

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Abstract: Reverse cationic column flotation technology is gaining traction in iron ore processing industry for recovering iron values from iron ore slimes/tailings. Development and selection of appropriate collector suitable to the material to be processed plays vital role in the development of the process/technology. Three cationic collectors which are vegetable oil based, biodegradable and environmental friendly were tested for their efficacy in reducing alumina and silica and improve iron content in the tailings of an operating iron ore washing plant. Selectivity index as defined by Douglas was used as response parameter to measure the efficiency of separation while comparing the performance of these collectors during flotation tests. Sokem 522C proved to be better in comparison to Sokem 701 and Sokem 702 with the highest selectivity index of 20.87. The concentrate generated analyzed 63.30% Fe, 2.20% SiO_2 and 2.52% Al_2O_3 with 69.00% weight recovery and 79.21% Fe recovery from the tailings containing 55.14% Fe, 17.34% SiO_2 and 4.04% Al_2O_3. The concentrate generated is suitable for pellet making due to its attractive granulometry and acceptable levels of alumina content (≤ 2.50%) in it. The tailings generated in this process could be utilised for cement manufacturing and tiles or pavement blocks making.The process developed is 'zero waste' while mitigating the environmental issues related to managing tailings in iron ore processing plants.

Keywords: Iron ore, ultra fines, cationic collector, tailings pond, column flotation

I. Introduction

Ninety eight percent of iron ore mined is used in iron and steel making. High grade iron ore deposits exhausted rapidly due to increased demand and consumption of steel. Optimal utilization of different products generated during processing of iron ore is the need of the hour. Most of the iron ore mines in India have washing plants to produce lumps as well as fines. Around 8-10 million tonnes of slimes containing around 48-60% of Fe are discarded every year [1]. These slimes cannot be used in iron making as they contain higher amount of gangue [2]. Several beneficiation techniques have been tried from time to time to reduce the gangue so that the beneficiated products could be effectively used for iron making [1, 3-4]. Efforts have been made to reduce alumina in the slime by using classification followed by separation in a hydrocyclone wherein it was possible to obtain a product containing 64% Fe, 1.4% silica and 3.5% alumina from a feed assaying 57% Fe, 4% silica, and 8.3% alumina [5]. Several researchers worked on the reduction of alumina from iron ores, focusing on flocculation techniques that resulted in success of varying degrees [6-8].

The beneficiation of iron ore slimes produced from washing plants and tailing ponds of Kiriburu mines was studied using wet high-intensity magnetic separators (WHIMS) followed by classification in hydrocyclone whereby a concentrate assaying 63% Fe and 3.3% alumina was produced with an overall iron recovery of 56% [9]. Though multi-gravity separation is a useful technique for treating iron ore slimes in general and for reducing alumina in particular, it is not very successful commercially due to its low capacity [10]. Separation of Barsua, Bolani, and Kiriburu iron ore slimes was studied using classification by hydrocyclone followed by high-intensity magnetic separation [3]. Their results show that it is possible to obtain a concentrate assaying 60-65% Fe with 60-80% recovery. Another study used classification in a hydrocyclone followed by spiral concentration for iron ore slimes obtained from washing plants and tailing ponds of Kiriburu mines [11]. The experimental results show that it is possible to increase the iron content up to 64.17% at a yield of 37.3% with simultaneous decrease in the alumina content, down to 1.17%. Few researches also tried separating the gangue (viz. quartz and kaolinite from iron-bearing minerals, mostly hematite and goethite), to produce a suitable concentrate for downstream processing [12]. Earlier studies indicated that silica and alumina could be reduced by reverse cationic column flotation of a pre-concentrate as a value addition step and also from screw classifier overflow obtained from two different operating beneficiation plants [13-15]. In another study involving flotation column and reverse flotation process, high depressant dosage was suggested to achieve high grade concentrates with low impurity content [16]. They could achieve approximately 60% mass recovery and 80% metal recovery in the flotation stage.

M/s JSW Steel Limited, Karnataka, one of the leading producers of steel in India operates two beneficiation plants with the objective of reducing alumina and silica from the iron ore fines. The unit operations comprise of wet screening, scrubbing, spirals, magnetic separation, and classification by screw classifiers and hydrocyclones. A flotation plant has been in operation to process the tailings from both the beneficiation

ISSN:2319-6890)(online),2347-5013(print)
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plants that were accumulated at Sultanpura, Ballari district, Karnataka.

Finding the right collector for flotation and its suitability to the ore under investigation needs careful selection, experimental planning and evaluation of the results. Selection of an appropriate cationic collector has a vital role to play in the reverse flotation of iron ore slimes for the reduction of alumina and silica in general and alumina in particular. It depends on various factors such as the chemical composition of the reagent, its technical performance, price and availability. However, the dosage and efficiency of the reagent (collector) in terms of its selectivity in separation process are of utmost importance. The process of reagent selection and optimization in flotation systems as practiced today is rather informal and reductionist on the part of both reagent manufacturers and plant metallurgists. For example, when a plant experiences a recovery problem, an immediate temptation might be to seek an alternative collector. Thus, a testing program either in the laboratory or in the plant, most likely the former, may be initiated to screen several alternative collectors [17]. With the reluctance of reagent manufacturers to part with the information related to chemical composition and properties of the reagents, the alternative route to screen out the best reagent among the available ones for the task at hand is to resort to statistical analysis of the data pertaining to flotation tests conducted under standard and identical test conditions using these reagents or by measuring the efficiency of separation using these reagents by selectivity index. The present paper addresses such an issue of selecting the best collector amongst the three during one of its elaborative test work programme conducted related to reduction of alumina and silica in the tailings.

II. Materials and methods

Materials

Mineralogical analysis of tailings indicated that it is mainly composed of hematite, goethite and limonite as the iron bearing minerals whereas quartz and kaolinite (clay) formed the gangue. Microscopic studies reveal that iron bearing minerals are in intimate association with each other as well as with gangue in the size range below 5 microns. A comprehensive study on the mineralogical, geochemical and separation characteristics of the iron ore fines of this region with special reference to their implications on beneficiation in general and flotation in particular can be found elsewhere [18-19]. Cationic amine collectors Sokem 522C, Sokem 701 and Sokem 702 which are generically same but compositionally different from each other were developed by M/s Somu Organo-Chem Pvt. Ltd., Bengaluru, India in collaboration with CSIR-National Metallurgical Laboratory - Madras Centre (NML - MC). The causticised maize starch supplied by M/s Riddhi Siddhi Gluco Biols Ltd., Ahmedabad, India was used as depressant for iron bearing minerals. Commercial grade sodium hydroxide was used as pH regulator.

Chemical analysis

A representative tailings sample (feed to flotation) was drawn for chemical analysis and the chemical analysis results are as shown in Table 1.

Table 1. Chemical analysis of iron ore tailings.

Sample	Fe, %	SiO_2, %	Al_2O_3, %
Tailings	55.14	17.34	4.04

Size analysis

The particle size distribution of the tailings was carried out using laser particle size analyzer (Cilas 1180) and the particle size distribution is shown in Figure 1. The d80 of this iron ore tailings sample was found to be 28µm.

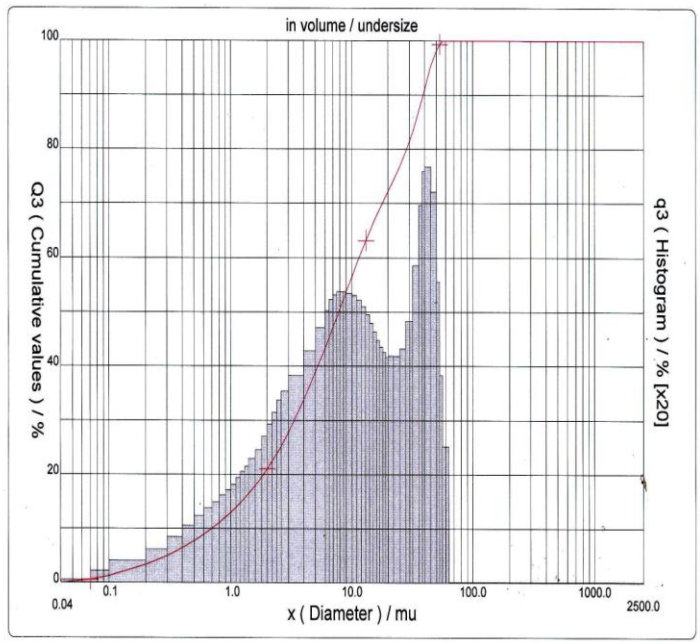

Fig. 1. Particle size distribution of iron ore tailings

Flotation tests

The laboratory scale experiments are conducted using Denver flotation machine, D12 with a cell volume of 3.0 L. The pH of the slurry is maintained at 10.0. The iron ore tailings slurry (50% solids by weight) is conditioned with sodium hydroxide as pH regulator, causticised starch (0.75 kg/t) as depressant for iron bearing minerals and the reagent as collector for gangue for three minutes each. The solids are kept in suspension by the impeller rotating at a speed of 1200 rpm. Flotation is conducted at 30% solids for 10 minutes or till the flotation ceases. The concentrate and tailings are collected separately. They are analyzed for total iron, silica and alumina.

III. Results and discussion

Based on the nature of iron ore, either iron minerals or gangue minerals can be floated using suitable reagents/collectors. In the case of low grade ore, iron minerals are floated directly using oleic acid or other collectors (Direct flotation). On the other hand, if the gangue (Al_2O_3 & SiO_2) content is relatively low, then they are floated using fatty amines as collectors (Reverse flotation). Selection of direct or reverse flotation is mainly defined by the overall economics of the process. In this particular case, since gangue content is relatively less, reverse flotation using cationic collector is adopted. Mostly, fatty amines with long chain hydrocarbons with chain length of 12-18 carbon atoms are used as collectors for the flotation of silica and alumina bearing minerals. In order to enhance the solubility of amines, they are usually converted to their chloride salts and/or acetates. Fatty amines with better solubility and surface activity are ideally suitable.

It is well known that amines are positively charged molecules up to pH 10.0.

$$R\text{-}NH_2 + H^+ \rightleftharpoons R\text{-}NH_3^+ \quad\text{———}\quad (1)$$

Positively charged molecules are easily attracted towards negatively charged particles by electrostatic interaction. Thus, negatively charged particles are made hydrophobic by adsorbing collector molecules. Since the iso electric point of silica is around 2.5, silica particles are negatively charged above pH 2.5.

$$SiOH_2^+ \xrightarrow{-H^+} SiOH \xrightarrow{-H^+} SiO^- \quad\text{———}\quad (2)$$

Though pH 3.0 to 10.0 is an ideal range for adsorption of amine molecules on silica particles, alumina and iron minerals are positively charged up to pH 7.5 - 9.0. In order to achieve better selectivity, the particles are thoroughly dispersed by adjusting the pH to above 9.0 where iron minerals, silica and alumina bearing minerals are negatively charged. At this stage, surface of the iron minerals is masked to avoid amine adsorption by conditioning with starch. Hence iron minerals could be depressed and, silica and alumina bearing minerals could be selectively floated using cationic collectors.

Cationic collectors are increasingly being used for the flotation of silica away from iron ores and phosphate ores [20-21]. Many plants in the world are using these reagents in the processing of low grade hematite and magnetite ores [22-23]. One of the advantages is the rapid flotation with sharp selectivity. Deshpande et al made a study on the selection of cationic collector for reduction of silica in reverse flotation of Kudremukh iron ore, India [24].

The reverse flotation studies were carried out using three reagents namely, Sokem 522C, Sokem 701 and Sokem 702 that are bio-degradable and eco-friendly. The reagents were added at different dosages in each of the experiments. The results were shown in Tables 2, 3 and 4. They were evaluated in terms of 'Selectivity Index' (SI) for the efficacy of the collector in the separation process.

Table 2. Effect of variation in dosage of Sokem 522C on reverse flotation of iron ore tailings

Product	Sokem 522C, kg/t	Weight %	Assay, %			Distribution, %		
			Fe	SiO_2	Al_2O_3	Fe	SiO_2	Al_2O_3
Tails.	0.2	10.78	34.47	28.09	9.40	6.74	25.32	29.01
Conc.		89.22	57.63	10.01	2.78	93.26	74.68	70.99
Tails.	0.3	21.55	34.47	39.63	7.52	13.61	74.68	43.90
Conc.		78.45	60.08	3.69	2.64	86.39	25.32	56.10
Tails.	0.4	27.06	37.60	36.97	6.76	18.35	86.01	48.81
Conc.		72.94	62.08	2.23	2.63	81.65	13.99	51.19
Tails.	0.5	31.00	36.98	51.04	5.92	20.79	91.25	51.35
Conc.		69.00	63.30	2.20	2.52	79.21	8.75	48.65
Head (assay)			55.14	17.34	4.04			

Tails.: Tailings & Conc.: Concentrate

Table 3. Effect of variation in dosage of Sokem 701on reverse flotation of iron ore tailings

Product	Sokem 701, kg/t	Weight %	Assay, %			Distribution, %		
			Fe	SiO_2	Al_2O_3	Fe	SiO_2	Al_2O_3
Tails.	0.2	5.19	40.60	21.47	10.27	3.80	9.53	15.83
Conc.		94.81	56.29	11.15	2.99	96.20	90.47	84.17
Tails.	0.3	8.24	37.59	25.33	10.60	5.63	17.23	24.91
Conc.		91.76	56.59	10.93	2.87	94.37	82.77	75.09
Tails.	0.4	12.53	36.59	29.67	9.20	8.28	31.76	32.72
Conc.		87.47	58.07	9.13	2.71	91.72	68.24	67.28
Tails.	0.5	16.49	33.11	37.74	8.21	9.98	52.66	37.69
Conc.		83.51	58.94	6.70	2.68	90.02	47.34	62.31
Head (assay)			55.14	17.34	4.04			

Table 4. Effect of variation in dosage of Sokem 702 on reverse flotation of iron ore tailings

Product	Sokem 702, kg/t	Weight %	Assay, %			Distribution, %		
			Fe	SiO_2	Al_2O_3	Fe	SiO_2	Al_2O_3
Tails.	0.2	6.47	39.81	21.75	11.22	4.69	11.70	21.00
Conc.		93.53	56.00	11.35	2.92	95.31	88.30	79.00
Tails.	0.3	11.57	39.38	25.47	10.10	8.16	23.50	32.98
Conc.		89.43	57.18	10.70	2.76	91.84	76.50	67.02
Tails.	0.4	14.16	36.03	32.90	8.96	9.22	38.50	35.55
Conc.		85.84	58.52	8.67	2.68	90.78	61.50	64.45
Tails.	0.5	16.63	34.27	40.06	7.96	10.30	54.96	37.29
Conc.		83.37	59.52	6.79	2.67	89.70	45.04	62.71
Head (assay)			55.14	17.34	4.04			

As the dosage of the collector increased, there was substantial improvement in the quality of the concentrate produced in all the three cases. But better results were obtained

ISSN:2319-6890)(online),2347-5013(print)
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in case of collector Sokem 522C. In this case, iron percentage increased from 55.14% (feed) to 63.30% while its recovery was 77.50% at 0.5 kg/t. At equivalent dosage of Sokem 701 and Sokem 702, the grade of concentrate was 58.94% Fe with 90.02% Fe recovery and 59.52% Fe with 89.70% Fe recovery respectively. Moreover, alumina in the concentrate, using Sokem 522C as collector during flotation, was 2.52% which was in acceptable limits ($\leq$ 2.50%) for pellet making compared to that of 2.68% using Sokem 701 and 2.67% using Sokem 702 as collectors. The corresponding silica levels were 2.20%, 6.70% and 6.79% in the concentrates using the above mentioned three collectors/reagents respectively.

Selectivity index gives an accurate scientific measure of the effectiveness of the separation. It seems, in fact, to be the most accurate measure that could be devised. The performance of the collectors was evaluated based on modified version of Selectivity Index (SI) derived by E. Douglas [25]. 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. It serves as a useful measure of the efficacy of the separation process on a number of different samples as well. It is given by

$$SI = [(R-C)*(c-f)*100] / [(100-C)*(c_{max.}- f)] \quad -- (3)$$

where C is % weight of the concentrate; R is % recovery of iron in the concentrate; c is % iron of the concentrate; cmax is the maximum (theoretical) iron in the concentrate and f is % iron of the feed. Using the above shown formula, the SI for the three collectors at different dosages were calculated and shown in Figure 2.

The selectivity indices for Sokem 701 and Sokem 702 varied from 1.56 to 12.05 as the dosage of these two was varied from 0.2 to 0.5 kg/t. But in the same dosage regime, the selectivity index for Sokem 522C varied from 6.11 to 20.87. Better performance of Sokem 522C could be attributed to its ability to impart greater hydrophobicity to gangue so as to enable them to be recovered more efficiently into the tailings. This clearly indicates that Sokem 522C is preferable in comparison to Sokem 701 and Sokem 702 for reduction of alumina and silica in tailings.

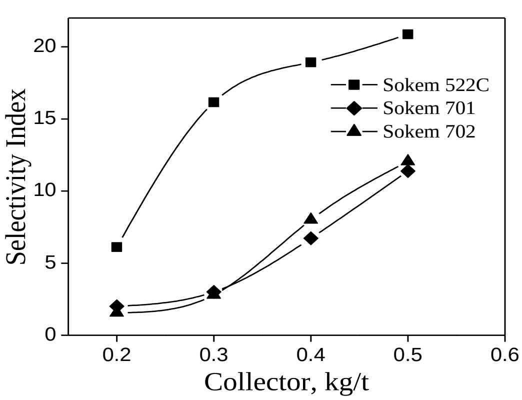

Fig. 2. Comparison of selectivity indices of collectors

IV. Conclusions

Iron ore processing industry eagerly waits for suitable and viable technology for recovering ultrafine iron values from slimes/tailings from the old and existing tailings dam. Reverse cationic column flotation technology offers the best viable solution. Selection of right and appropriate collector is crucial for the success of the process. Three commercially available collectors namely Sokem 522C, Sokem 701 and Sokem 702 were evaluated for their performance in reducing alumina and silica in tailings and thereby increase iron value in the concentrate. 'Selectivity Index' was chosen as response parameter during their evaluation. Variation of collector dosage has greater influence in improving the concentrate grade. Sokem 522C was found to be the best amongst the three collectors tested with selectivity index of 20.87. The concentrate produced, using this collector, analyzed 63.30% Fe, 2.20% SiO_2 and 2.52% Al_2O_3 with 69.00% weight recovery and 79.21% Fe recovery from feed containing 55.14% Fe, 17.34% SiO_2 and 4.04% Al_2O_3. It is suitable for pellet making due to its attractive granulometry and acceptable levels of alumina content in it. The final tailings generated after flotation could be used for cement manufacturing and tiles/pavement blocks making. Based on the process and cationic collector developed for alumina reduction in the tailings, new facilities involving dredging and flotation based processing plant were set up at the tailings dam of M/s JSW Steel Limited, India.

V. Acknowledgments

The authors are grateful to the Director of CSIR-National Metallurgical Laboratory, Jamshedpur for his valuable guidance and permission to publish the work and M/s Somu Organo Chem Pvt. Ltd., Bengaluru for their logistical support.

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