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Recovery of Iron Values from Waste Iron Ore Slime

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Abstract
Iron ore slime is a waste material generated after beneficiation of iron ores. Due to limited iron ore resources and fast depleting of high grade iron ore, iron ore producers are emphasizing on recovery of iron values from iron ore slime. Different beneficiation techniques such as Hydrocyclone, Wet High Intensity Magnetic Separator (WHIMS), Dispersion-flocculation, and Flotation are some options to recover the lost iron value from the waste. In the present work an attempt has been made, to beneficiate iron ore slime using Hydrocyclone, WHIMS and dispersion-flocculation. Iron ore slime (-150 μm) was obtained after scrubbing and wet screening of a low grade iron ore assayed 42.7% Fe with 12.5% Al_2O_3 and 13.68% SiO_2. Size analysis revealed that 90% of the iron ore slime was having size less than 72 μm. The isoelectric point of the iron ore slime was found to be at pH 4.21.The optimum condition was applied for Hydrocyclone to obtain maximum recovery i.e., 89.2%. The cyclone underflow was enriched to 54.8% Fe with 45% recovery using WHIMS at current intensity of 2 Amp. Dispersion-flocculation could upgrade up to 47% Fe only. The nonmagnetic fraction of WHIMS was upgraded from 37 to ~39% Fe using dispersion-flocculation under the optimized condition.

Keywords: *Iron ore slime, recovery, Hydrocyclone, WHIMS and dispersion-flocculation*

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INTRODUCTION

World steel production is increasing steadily over the years leading to increase in the requirement of iron ore. According to information published in the Indian Mineral Yearbook, 2011–2012, the total iron ore resource of the country is estimated to be around 28.52 billion tonnes. This excludes Banded Hematite Jasper (BHJ) and Banded Hematite Quartzite (BHQ) deposits [1]. The main iron ore deposits in India occur in the eastern, central, and southern parts, for example, Jharkhand, Orissa, Karnataka, Chhattisgarh, and Goa. Generation of iron ore slimes in India is estimated to be 10–25% by weight of the total iron ore mined [1–4].

The iron ore values are lost to the tune of 15–20 million tones every year. These slimes are readily available in finer size typically assaying 55–60% Fe [3]. These slimes are dumped in the tailing ponds in the mine area and cause environmental concern. Iron ore slime is a waste generated after beneficiation of iron ores. Due to limited iron ore resources and depleting high grade iron ore, iron ore producers are emphasizing on recovery of iron values from iron ore slimes. Generally, physical beneficiation techniques such as gravity, magnetic, flotation and selective-flocculation for enriching the iron values from slimes are employed.

Rath et al. studied dispersion-flocculation experiments on individual samples of hematite, quartz and kaolinite as a function of different process parameters such as pH, flocculation time and dosages of reagents. They could find out a condition for selective separation of hematite from quartz and kaolinite and applied to ternary synthetic mineral system [5].

Thella et al. conducted beneficiation on iron ore slime using desliming and flotation. The slime assayed 51% Fe. Classification of slimes with two stage Hydrocyclone gives a concentrate containing 61.99% Fe with Fe recovery of 54.55% in Stage-I. Concentrate from Stage-II Hydrocyclone contains 62.19% Fe with Fe recovery of 47.45% with respect to initial feed [6]. Manna et al. conducted dispersion study on ultra fines of iron ore collected from two locations, Joda and Noamundi slime ponds in the states of Orissa and Jharkhand (India), respectively using sodium hexametaphosphate as a dispersing agent. NaOH was used as pH regulator. Under a given settling condition, a high grade concentrate containing 67.9 wt.% Fe, 1.2 wt.% Al_2O_3 and 1.25 wt.% SiO_2 with 58% iron recovery is achievable using off grade iron ultra fines containing 57.8 wt.% Fe, 7.55 wt.% Al_2O_3 and 7.15 wt.% SiO_2 [7].

Panda et al. investigated the recovery of fine hematite particles from banded hematite quartzite (BHQ) ore with potato starch using selective flocculation. The starch molecules show good selectivity for hematite particles in the pH range of 8.5–9.5 and in the reagent concentration range of 20–40 mg/l. However, under optimum conditions of pH and reagent concentration, the BHQ ore could be upgraded from an initial grade of 38.9% Fe to 57.2% Fe with 71% recovery [8].

Abro et al. studied selective flocculation of Dilband iron ore and observed an upgradation of the ore from 52% hematite (i.e., 39% Fe) to 60% hematite (i.e., 45% Fe) with an average hematite recovery of 15% [9]. In the present work an attempt has been made, to beneficiate iron ore slime using Hydrocyclone, WHIMS and dispersion-flocculation.

METHODOLOGY

Materials

The material under study was obtained after scrubbing and wet screening of a low grade iron ore sample at 150 μm (Figure 1).

Fig. 1: *The Raw Material (iron ore slime) for the Study.*

Sample for Dispersion-flocculation Studies

The sample for dispersion-flocculation studies was drawn from main sample through coning, quartering and reduced in size to pass 325 mesh i.e., 45 microns. The sample was crushed and ground to 45 μm using vibrating cup mill to conduct dispersion-flocculation studies.

Reagents

The chemicals that were used in dispersion-flocculation studies are given in Table 1**.**

Table 1: *List of Chemicals used in Dispersion-flocculation Studies.*

CHEMICALS	SOURCE
Sodium Hexa Meta Phosphate A.R	Himedia Laboratories
Sodium Hydroxide Pellets G.R	Merck
Starch (Soluble)	Qualigens
Carboxymethyl Cellulose Sodium Salt	CDH Laboratory
EDTA Disodium Salt	Qualigens
Dodecylamine Hydrochloride-99%	Acros Organics
Conc. Hydrochloric acid	Merck Laboratories

METHODS

The material was taken and its detailed characterization studies have been carried out in terms of its size distribution and chemical analysis. Physical characterisation of the material was done by wet sieve analysis.

2 kg of iron ore slime reject was passed through Mozley's Hydrocyclone under different conditions. Different parameters such as apex diameter, vortex finder diameter and pressure were changed to find optimum condition for maximum recovery of iron particles. The underflow and overflow are collected separately and transferred to trays and dried in oven and weighed. After obtaining the optimum condition for maximum recovery, several batches of 2 kg of sample was run under the optimum condition to obtain a sizeable amount for further experimentation.

Wet High Intensity Magnetic Separator (WHIMS) was carried out on underflow product of hydrocyclone using ALMINERAL make, Gaustec model equipment. Experiments were conducted by varying the current intensity 2, 5 and 8 Ampere, respectively to obtain the optimum condition of maximum Fe grade upgradation.

Zeta potential of the material with and without reagents was measured using Delsa Nano C particle analyser of Beckman Coulter at different pH Value.

The dispersion-flocculation experiments were carried out by addition of varied dosages of reagents at different time period and pH conditions. 1 g of sample was taken in 100 ml stoppered measuring cylinder and pH was maintained and the chemicals are added sequentially. It is then inverted upside down 10 times for proper mixing and allowed to settle under gravity for 60 sec. The supernatant solution is pipetted out up to the mud line 30 cm and ultrasonicated for 10 min. Then turbidity is measured using a Systronics Digital Nephlo Turbidity Meter 132.

The supernatant and settled material was then dried separately, weighed and analyzed for Fe content. The dispersion-flocculation experiments were carried out in absence and presence of various reagents as a function of time, pH, and reagent concentration. The optimum conditions and dosage of reagents for maximum up-gradation was found, and under these conditions an attempt was made to upgrade Fe % of nonmagnetic fraction of WHIMS.

RESULTS AND DISCUSSION

The size analysis of as received iron ore slime reject sample was carried out by wet sieving techniques to know the distribution of Fe at various size fractions. The iron ore slime reject thus obtained were subjected to chemical analysis to ascertain the different quantitative elemental composition of the sample. The chemical analysis of slime reject is shown in Table 2 and size wise wet chemical analysis of the same is shown in Table 3.

Table 2: *Chemical Analysis of the Iron Ore Slime Reject.*

Fe%	SiO_2%	Al_2O_3%	LOI %
42.7	13.68	12.5	12.53

Table 3: *Size Wise Chemical Analysis of the Iron Ore Slime Reject.*

Size (µm)	Weight %	Fe%
100	5.5	51.3
75	4.8	47.4
60	3.1	49.8
44	2.5	54.3
37	7.4	50.9
30	4.7	50.3
-30	72.1	39.7

90% of the particles are passing through 72 µm. The Table 3 depicts that most of the particles are in the range below 30 micron size. Also more than 72% of the material is of grade ~40 % Fe.

CONCENTRATION THROUGH HYDROCYCLONE

Beneficiation studies on hydrocyclone shows that maximum recovery is obtained with apex diameter 9.4 mm, vortex finder diameter 11 mm and pressure 10 psi. The Fe % and Fe recovery obtained under optimum condition are tabulated in the Table 4.

Table 4: *The % Fe and % wt. Results at Optimum Condition by Hydrocyclone.*

Condition	Wt. %	Fe%	Fe recovery%
Underflow	86	44.37	89.2
Overflow	14	32.7	10.8

CONCENTRATION OF CYCLONE UNDERFLOW BY WHIMS

With increase in current intensity, the grade decreased from 54.88 to 51% but Fe recovery increased from 45 to 56%. Maximum grade was obtained at 2 Ampere current intensity i.e., 54.88% Fe and maximum Fe recovery was obtained at 8 Ampere current intensity i.e., 56%. The nonmagnetics fraction at a current of 2 Ampere contains 37.0% Fe values.

ZETA POTENTIAL MEASUREMENT

The zeta potential values of iron ore slime with and without reagents at different pH are shown in Figure 2. The isoelectric point of the iron ore slime sample was found to be at pH 4.21.

In presence of Sodium hexa meta phosphate, starch and sodium oleate, the iep is lowered to acidic side due to chemical interaction of these reagents with the sample. Zeta potential below pH 7 could not be determined in presence of critical micelle concentration (CMC) and Dodecylamine as the sample was highly flocculated in the above pH range. However, above pH 7 the zeta potential values seem to be very close to the sample.

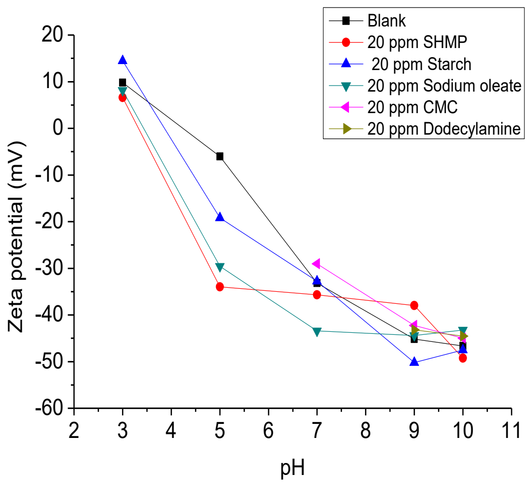

Fig. 2: *Zeta Potential of Sample with and without Reagents.*

DISPERSION-FLOCCULATION STUDIES

Selective dispersion-flocculation studies were conducted with iron ore slime with and without reagents. Preliminary experiments on behavior of different dispersants such as sodium hexa meta phosphate, EDTA, Sodium silicate with iron ore slime indicated that Sodium hexa meta phosphate acted as a good dispersant.

Hence, sodium hexa meta phosphate with 2.4 kg/t was used as dispersant for rest of the experiments. The behavior of iron ore slime with different reagents of dosage 20 ppm and without any reagent as a function of pH is given in Figure 3. Due to the combined effect of the dispersant and flocculant, it was observed that the wt.% settled in Figure 3 is lowered. The pH trend indicates the % settled decreases with increase in pH for both with and without reagents.

The pH was maintained at 9 for flocculants such as starch, dodecylamine and sodium oleate whereas for CMC it was pH 7. After selection of pH for different flocculants, further studies were carried out to find the effect of the dosage of flocculants and the results are presented in Figures 4(a) and (b).

In this case, the settled wt.% was chemically analysed for its Fe content and the same is presented in Figure 4(b). As the dosage increased, % Fe of dodecylamine and CMC decreased. For sodium oleate and starch upto 30 ppm % Fe increases but beyond that % Fe decreases. Correspondingly the supernatant turbidity is minimum for starch and sodium oleate at dosage 30 ppm [Figure 4(a)].

However, dodecylamine and CMC have low turbidity at dosage 10 ppm [Figure 4(a)]. The effect of time period on flocculation of iron ore slime was studied by varying time period and keeping other conditions constant such as pH and flocculant dosage.

The result of effect of time period on iron ore slime flocculation is presented on Figures 5(a) and (b). As expected the increase in time increased the % of settled fraction [Figure 5(a)]. Within time period 1 to 2 min maximum iron was upgraded. In the supernatant the turbidity also shows a lower value [Figure 5(b)]. Summary of optimum conditions of dispersion-flocculation studies for different flocculants is given in Table 5.

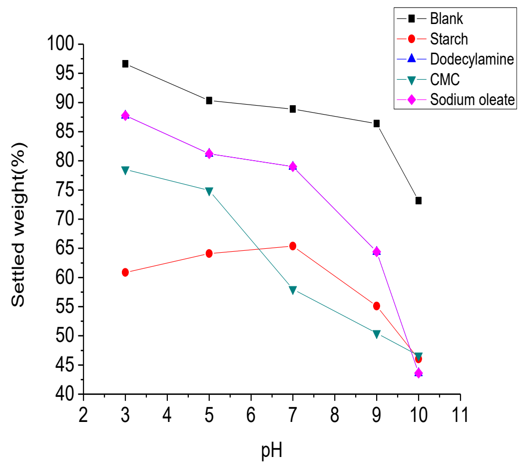

Fig. 3: *Effect of pH on Settled Weight %.*

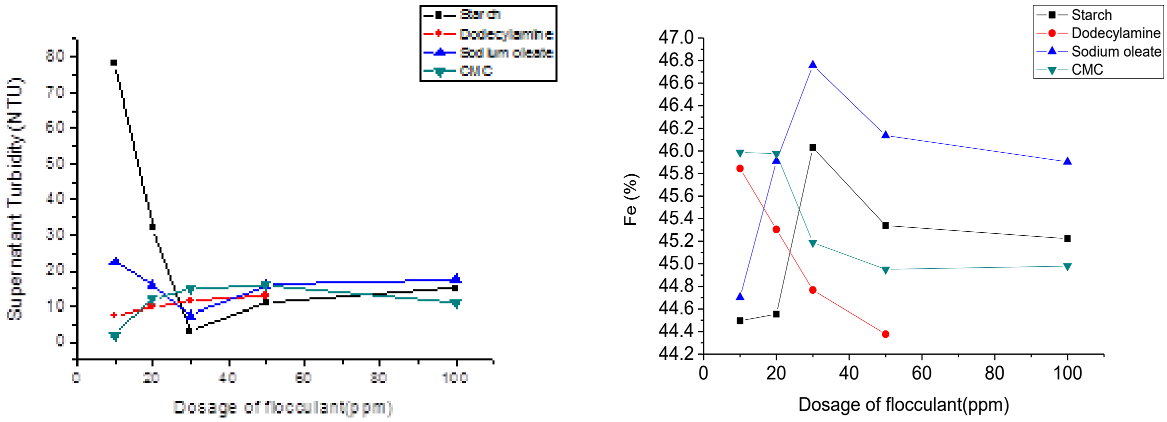

Fig. 4: *Effect of Flocculant Dosage on (a) Supernatant Turbidity and (b) Fe Content of Settled wt%.*

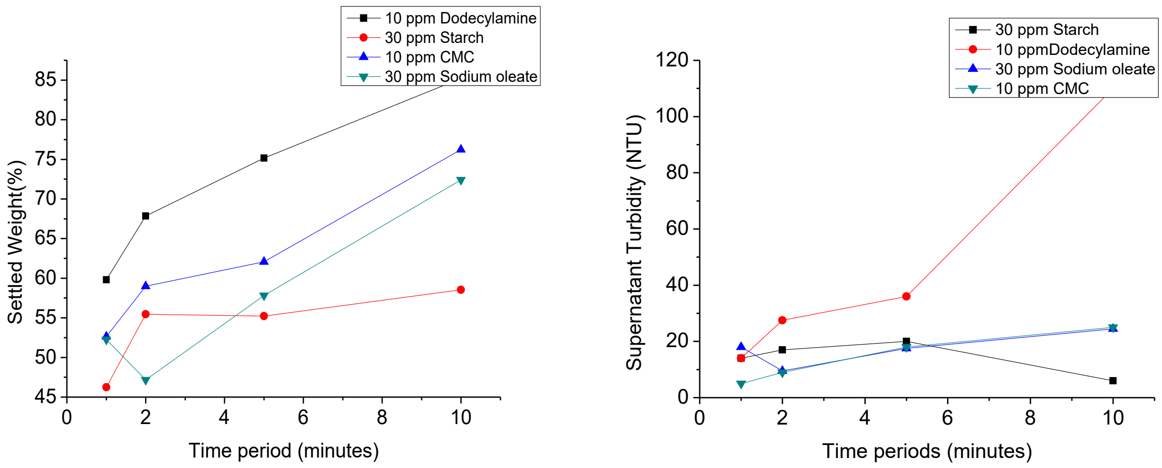

Fig. 5: *Effect of Time Period on (a) Settled Weight % and (b) Supernatant Turbidity.*

Table 5: *Summary of Optimum Conditions of Dispersion-flocculation Studies.*

Sl. No.	**Flocculant Type**	**Conc. (ppm)**	**Dispersant, SHMP (Kg/ton)**	**pH**	**Fe%**	**Turbidity NTU**	**Weight%**	**Time minute**
1	Sodium oleate	30	2.4	9	47.1	9	47.16	2
2	Soluble starch	30	2.4	9	46.2	17	57.5	1
3	CMC	20	2.4	7	46.3	5	52.5	1
4	Dodecylamine	10	2.4	9	45.84	14	59.79	1

It is indicative from the table that sodium oleate could able to improve the Fe % to the maximum when compared to the other flocculant.

Beneficiation of Nonmagnetic fraction of WHIMS using dispersion-flocculation

The dosage of flocculants and dispersant was optimized as 30 ppm and 2.4 kg/ton, respectively. pH 9 and settling time 1 min was maintained. The nonmagnetic fraction obtained after concentration by WHIMS at 2 Ampere was subjected to dispersion-flocculation under above mentioned condition. The non mag fraction assayed 37.0 Fe %. The dosage of flocculant was slightly varied. The effect of flocculant dosage on weight % and Fe % of settled fraction was studied. The results are presented in Figure 6. The increase in sodium oleate concentration no doubt increased the settled weight % but the grade was diluted. Better grade was observed at dosage 20 ppm. Increase in dosage more than 30 ppm doesn't have much effect on grade.

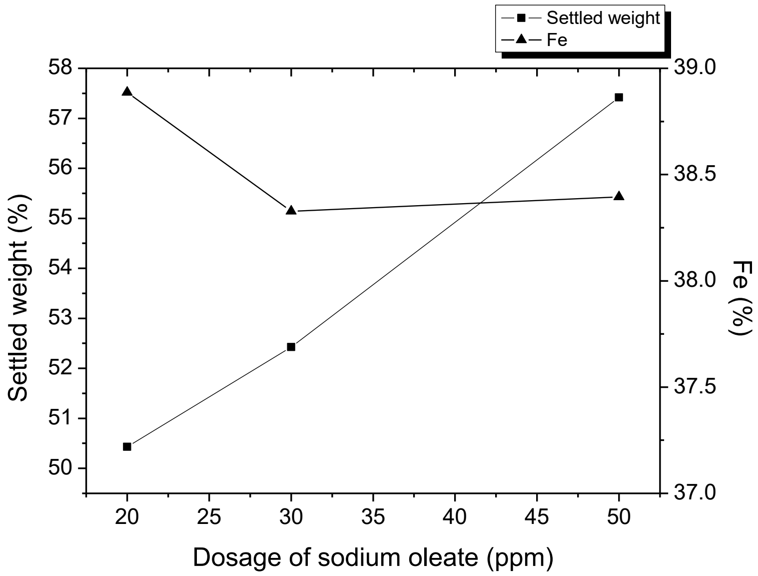

Fig. 6: *The Effect of Sodium Oleate Dosage on Weight % and Fe % of Settled Fraction.*

CONCLUSION

From the above studies few important conclusions can be drawn:

1. The iron ore slime contains 90% of particles less than 72 µm and assayed 42.7 % Fe.
2. The optimum condition for desliming using Hydrocyclone to obtain maximum recovery was at apex diameter 9.4 mm, vortex finder diameter 11 mm and pressure 10 psi. The underflow was upgraded to 44.37 Fe % with 89.2% recovery.
3. The cyclone underflow was upgraded to 54.8% Fe with 45% recovery using WHIMS at current intensity 2 Ampere.
4. Dispersion-flocculation studies performed using different reagents by varying time and pH. Maximum up-gradation of 47.1% Fe was obtained while using 30 ppm sodium oleate and 2.4 kg/ ton of sodium hexa meta phosphate at pH 9 and settling time of 2 min.
5. Zeta potential studies indicated that iso electric point of the slime is at pH 4.21. The iep is lowered in acidic side as a result of interaction of flocculants with the slime sample.

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