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Recovery of Iron Values from Iron Ore Slimes using Reagents

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Abstract

Mining wastes include waste generated during the extraction, beneficiation or processing of minerals like iron ore fines, slimes and tailings. Approximately 10–20% of the raw material is discarded as slimes in to slime ponds/tailing dams. Recovery of iron values from slimes result in economic benefit by utilization of waste as a resource and minimizes the threat to the environment. The iron ore slime is generally considered as waste due to its ultrafine nature and its processing limitations. The chemical analysis of the present iron ore slime is 34.75% Fe with 21.94% SiO_2, and 14.4% Al_2O_3. This research work presents the route to size enlargement of slime using reagents and enrichment of iron values by various beneficiation techniques for the effective utilization of iron ore slime. Screen analysis revealed that 90% of the particles are smaller than 80.15 µm. In the present investigation, the recovery of lost iron values from iron ore slime waste was attempted using different beneficiation techniques like Enhanced gravity separation (EGS) Falcon concentrator, Gravity separation by Mineral separator and Vanner, and selective flocculation after treatment with various reagents. Mineral separator generated a concentrate assaying 46.3% Fe with 56.2% recovery using polyacrylamide flocculant at 90 sec collection time and similarly Vanner produced a concentrate assaying 54.5% Fe with a recovery of 41.6% using CMC. From selective flocculation studies, a concentrate assaying 45.3% Fe with 42.1 yield % was obtained using modified corn starch at a settling time of 5 min, as starch facilitates the selective adsorption on iron particles, which in turn leads to enhancement in selectivity and recovery.

Keywords: *Iron ore slime, Tailing dams, Gravity, Enhanced gravity separator, Reagent*

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INTRODUCTION

India is bestowed with large and rich sources of iron ore in terms of quantity and quality. India occupies sixth position in iron ore resource base and ranks fourth with respect to world iron ore production [1]. The existing reserves of hematite (averaging around 63% Fe) are the only source of iron ore and as such, these reserves may not last beyond 25–30 years at the present rate of consumption. Hence, to meet the future and projected requirement, additional domestic resources like slimes and fines dumped elsewhere in mines have to be utilized, which are available in abundance [2]. In India most of the washing plants located in mines generates lumps as well as fines. During this process, a large quantity about 18–25% of ROM of slime is generated containing around 48–60% Fe content, which are discarded as tailings [3]. According to the latest guidelines issued by IBM, the cutoff grade for tailings is 45% [4]. This huge accumulation of slimes poses environmental problems particularly during rainy season when these fines get washed away and affect the agricultural fields, water bodies, removal of vegetation cover, deforestation, land slope changes, increased risk of erosion, water pollution and contamination of agricultural goods and risk of human health [5, 6]. The major iron ore mines are located in the states of Jharkhand, Orissa, Goa, Karnataka and Chhattisgarh [7]. Statistics showed that the increase in 1% Fe in the concentrate increases productivity of the hot metal by 2% and reduced thereby coke and limestone requirements by 1.8 and 0.9 %, respectively [8]. As fines and slime forms considerable part of iron ore resources, value addition to the iron ore fines through beneficiation is the need of the hour [9]. Generally, iron ore tailings contain substantial quantities of ultrafine particles and these ultrafine particles cannot be treated effectively using conventional beneficiation methods because of limitations of particles size being treated by these processes. Selective flocculation process could be one of the promising physicochemical methods for treating these ultrafine particles. The present work is focused on utilization of waste slime by enlargement of size using reagents and recovering the iron values using beneficiation techniques. The source of iron ore slime used in this investigation is generated in laboratory from scrubbing/ washing of low grade iron ore from eastern part of India.

METHODOLOGY

Materials

A composite sample was generated after de-sliming the -150 µm material by hydro cyclone. The cyclone overflow sample for the present study was dried, thoroughly mixed and homogenized, and a representative sample was drawn for chemical analysis. The results indicated that the dried sample contained 34.75% total iron, 14.4% alumina, and 21.94% silica, with an LOI of 11.1%. Tables 1 and 2 present the reagents that were used in the size enlargement of iron ore slime and the optimum dosage conditions of dispersant and flocculants used in the experiments.

Table 1: *Various Reagents used in the Experimental Studies.*

Reagents	Source
Hydrochloric Acid, GR	Merck Laboratories
Sodium Hydroxide Pellets G R	Merck Laboratories
Starch (Soluble) LR	Qualigens
Starch (Corn) LR	Qualigens
Sodium Hexa Meta Phosphate (SHMP), AR	Himedia Laboratories
Guargum, LR	Merck Laboratories and s d fine Chem.
Carboxy Methyl Cellulose Sodium Salt, CMC (MW=average M_w~90000)	CDH Laboratories
Polyacrylamide (PAM) (MW=50,00,000)	Himedia Laboratories
Polyacrylamide Co Acrylic Acid (PACAA) (MW=1,50,000)	Aldrich Product
Sodium Oleate	Himedia Laboratories
Sodium Silicate	Himedia Laboratories

Table 2: *Optimum Dosage of Reagents used in Various Experiments.*

Reagents	Optimum dosages used, ppm
Sodium Hexametaphosphate (SHMP) as dispersant	50
Corn Starch (Corn Starch + NaOH pellets (2:1)) as flocculants	20
Guargum as flocculant	20
Carboxy Methyl Cellulose (CMC) as flocculant	30
Poly Acrylamide (PAM) as flocculant	1
Poly Acrylamide Co Acrylic Acid as flocculant	1
Sodium Oleate as flocculant	20

Experimental Methods

Size Analysis

Particle size analysis of the iron ore slime sample was carried out by wet sieving using standard sieves and also using warman cyclosizer.

Zeta Potential Measurement

Zeta potential of the sample was measured by using Delsa Nano C particle analyzer of Beckman Coulter make at different pH values of 3, 5, 7, 9 and 11. For the zeta potential measurement the sample size should be of less than 10 µm. The mineral suspension of 0.05% was prepared and conditioned for 15 min at room temperature (30°C). For this purpose, 50 mg of sample was taken in a beaker and made up to 100 ml with distilled water. The solution was agitated for 5 to 10 min using magnetic stirrer and ultrasonicated for 15 sec. Then the sample was transferred immediately to the flow cell, which was fitted in the machine at appropriate position, and then with the help of selected SOP (Standard Operating Procedure) the zeta potential of the sample was determined. The zeta potential of the sample was determined using a dispersant Sodium hexametaphosphate, and with flocculants such as Corn Starch, Guargum, CMC, PACAA. The zeta potential value is expressed in mV and the graph between pH Vs zeta potential is being plotted automatically by the software.

Flocculation Experiments

For the flocculation experiments, a representative sample was ground in a vibrating cup mill to -37 µm size. Flocculation experiments were carried out in a 100 ml measuring stopper cylinder with a pulp density of 1%. The sample was conditioned in a beaker prior to flocculation. pH was adjusted and then a desired quantity of the dispersant was added. Ultrasonic treatment of iron ore slime sample was carried out using an Ultrasonic bath. The slurry was conditioned for 10 min and then transferred to a 100 ml measuring cylinder and then flocculant was added. The measuring cylinder containing the pulp was inverted upside down for ten times and allowed to settle for a given length of time. Two-thirds of the volume of material was siphoned out after the desired settling time. The supernatant liquid that was siphoned was used for turbidity measurements after ultrasonication for 10 min using standard Systronics make Nephelo turbidity meter model 132. The supernatant and settled portions were dried separately, weighed and analyzed for Fe content. The flocculation studies were carried out in presence and absence of reagents at optimum dosages and pH values with the variation in the settling time.

Size Enlargement

Size enlargement was carried out by treating different reagents like Corn starch, Guargum, CMC, PAM, PACAA and Sodium Oleate at a constant pulp density to make the ultrafine particles to agglomerate and ultimate size enlargement takes place on account of aggregation.

Falcon Concentrator

Enhanced gravity separation is used in mineral processing to separate minerals based on differences in specific gravity. The most common and successful type of gravity separator used for fine particle sizes is a centrifugal gravity concentrator. Falcon concentrator is an enhanced gravity separator where centrifugal field is applied on the particles to enhance the gravity. In falcon concentrator, rotating bowl is spinned at a very high speed and the materials experience a very high centrifugal force (G). The feed streams particles are subjected to gravitational force up to 300 G's and are segregated according to effective specific gravity along the smooth spinning rotor wall. The heaviest layers pass over the concentrate bed retained in the riffles at the top of the rotor bowl. Falcon experiments were carried out using 100 g sample taken in a 1000 ml beaker at a pulp density of 10%. The sample was prepared and it was passed through falcon concentrator under different conditions by varying the different parameters like bowl rotation frequency (Hz), back water pressure (Psi) and with presence and absence of different reagents to find the optimum condition for the maximum recovery and grade of iron.

Enrichment using Mozley's Mineral Separator and Bartley Mozley Vanner

Treated iron ore slime sample was subjected to Mozley's mineral separator and Bartley Mozley Vanner know the response of the treated material.

Mozley's mineral separator: Mozley's mineral separator works on the principle of the gravity separation used in mineral processing to separate minerals based on the differences in specific gravity. Mineral separator consists of two trays, one is the flat type tray and the other one is V-Profile tray. The trays are mounted on a rectangular frame. The channel pipes are attached on the tray to supply the water to the sample while separation. A constant pulp with a constant pulp density of 40% was prepared and the slurry was homogenized using a mixer with reagents of proper dosages. The slurry was then passed through the mineral separator tray under the wash water pipe at the starting of the tray. The sample was studied for the two type of trays in presence and absence of different type of reagents like modified corn starch, Guargum, CMC, PAM, PACAA and Sodium oleate and the sample was collected at different time intervals like for every 5 sec on V-profile tray and for every 15 sec in flat tray by keeping the other parameters like water flow rate of 2.5 lit/min, table speed, amplitude and longitudinal slope constant. The slurry was gradually poured from a beaker on to the table; a spray bottle was used to remove all the material from the beaker. Water is supplied to the tray by the wash water pipe (copper tube) and the irrigation pipes (two white plastic tubes with water outlets at regular intervals). The sample was collected at the end of the table moving longitudinally down the trough in the direction of water flow. The tailings (low-density materials) separates first leaving the concentrate (high-density material) as the sample should be collected at regular intervals of time like 5, 10, 15, 20, 25, 30 sec, respectively. The material with higher iron values was collected in the last fractions. From this, the optimum condition for the maximum recovery of iron values was determined.

Bartley-Mozley Vanner: Vanner is a gravity concentrator and a high capacity concentrator designed to recover fine particles. Other than the specific gravity, size, shape and weight of the particles affect the relative movement and hence separation. Vanner consists of a long belt where the particles will move or separate, with eccentric weights mounted on a rectangular frame. A slurry of constant pulp density of 40% was prepared and reagents were added at the desired level and mixed thoroughly. Slowly the slurry was passed on to the deck, wash water is supplied continuously on to the deck through the channels. Due to the orbital or vanning motion by the eccentric weights the lighter particles will move down along with wash water and are collected at the downside of the deck, considered as tailings and the heavier particles will move to the upside of the deck that are considered as middling's. These middling's are again treated; from this the recovery of the concentrate takes place. Various operating parameters that are considered while operating the equipment are orbital speed, eccentric weight, amplitude, deck slope, pulp feed rate, wash water feed rate and using different reagents. From this, the optimum condition for the maximum recovery of the iron values was determined.

RESULT AND DISCUSSION

Chemical and Physical Characteristic of Iron Ore Slime

The chemical composition of the iron ore slime sample was found to contain iron 34.75%, alumina 14.4%, silica 21.94% and LOI 11.10%. The specific gravity of the given slime sample was measured and found to be 3.089. Table 3 presents the results of particle size analysis along with total Fe % of different size fractions of iron ore slime sample. Figure 1 indicated that ~75 wt% of particles are below 25 µm and the corresponding grade of the sample is 30.57% Fe with d_{90} is around 81.0 µm.

Table 3: *Size and Size wise Chemical Analysis of Iron Ore Slime Sample.*

Size (µm)	Wt % Retained	Fe (total, %)
-150+105	4.2	52.73
-105+75	7.3	46.76
-75+63	2.9	42.47
-63+45	2.5	52.06
-45+37	2.9	51.11
-37+25	6.2	51.17
-25	74.0	29.57
Feed (Calc.)	100.0	34.69

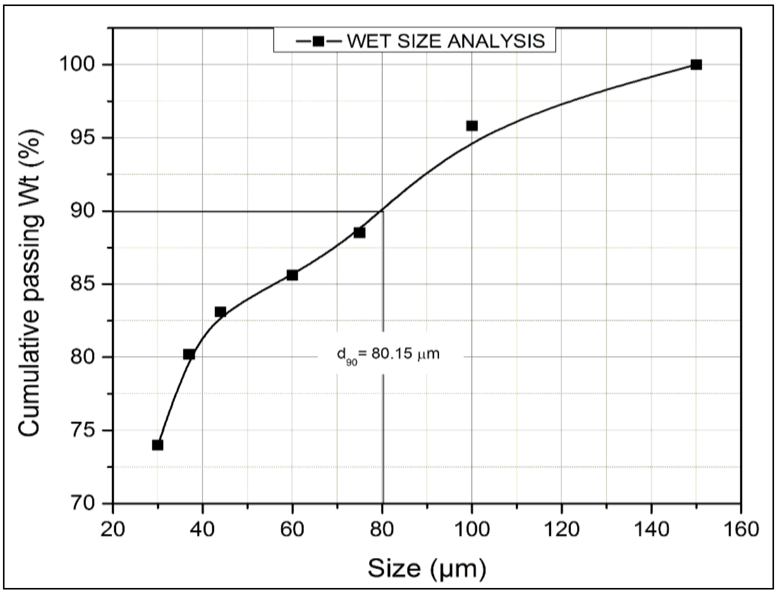

Fig. 1: *Graph showing Particle Size Distribution of Iron Ore Slime.*

Results on Enhanced Gravity Separator
Falcon concentrator is an enhanced gravity separator and was used to beneficiate the ultrafine particles, various sets of experiments were performed by changing the parameters like back water flow rate (psi) and bowl rotation frequency (Hz) at constant pulp density of 10%. Experiments were performed in presence and absence of reagents like dispersants and flocculants and the obtained results are depicted in Figure 2. The effect of bowl rotation frequency on Fe recovery % at different back water pressure in presence and absence of reagents are presented in Figures 2–4.

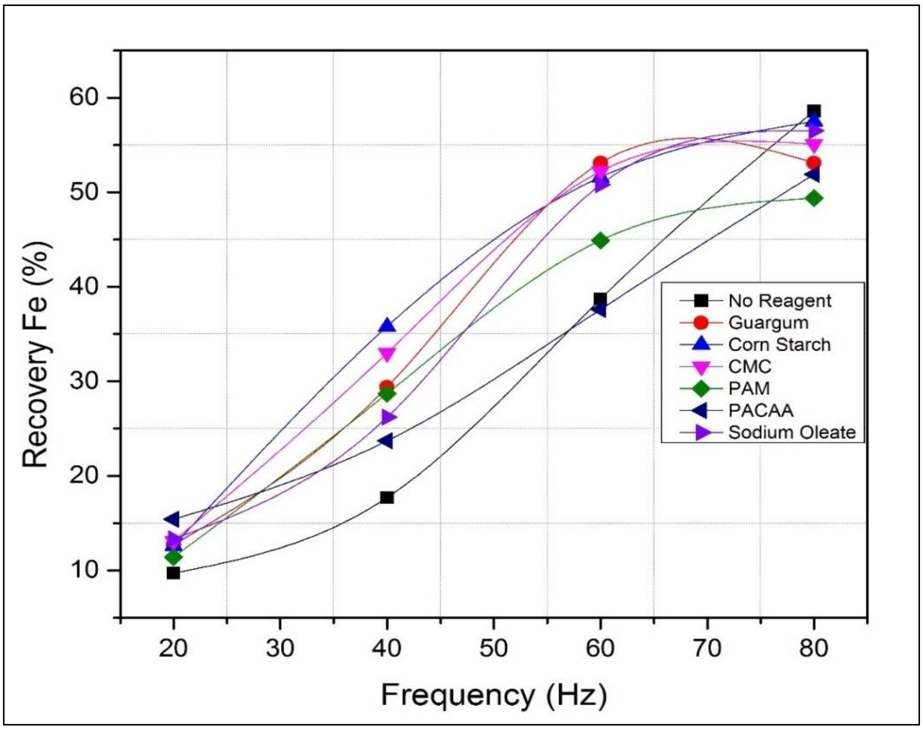

***Fig. 2:** The Effect of Bowl Frequency on Fe Recovery% of Underflow Product at Constant Back Water Pressure of 5 psi.*

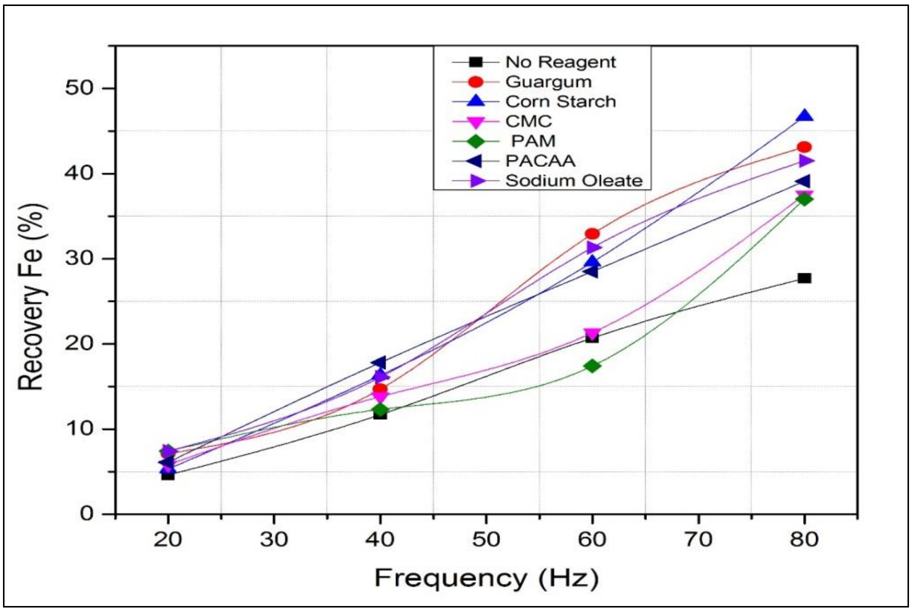

***Fig. 3:** The Effect of Bowl Rotation Frequency on Fe Recovery% of Underflow Product at Constant Back WATER pressure of 10 psi.*

The frequency of the bowl of falcon concentrator was changed from 20 to 80 Hz by keeping the other parameters like pulp density (P.D.):10%, back water pressure of 10 psi constant. The results that are obtained by the effect of frequency of the bowl, i.e., bowl speed at constant back water pressure of 10 psi on Fe grade and recovery on the underflow products of falcon concentrator.

In Figure 3, with an increase in the frequency from 20 to 80 Hz, there is an increase in recovery nearly by 50 units from 9.6 to 58.7% without use of any reagent. But with the use of reagents, Fe recovery increased only by 30 to 40 units with frequency. Maximum recovery of Fe 62.2% was obtained with Carboxy methyl cellulose (CMC) at 80 Hz frequency at 10 psi back water pressure; it is due to the effect of flocculant CMC in efficient separation of gangue from iron particles. It is to be noted that with the increase in the frequency from 20 to 80 Hz there is a decrease in the Fe grade nearly by 4 units from 56.72 to 52.26% in the case of blank condition. But with the reagents, we can observe that there is increase in Fe grade with some reagents like corn starch and sodium oleate and with other reagents there is a decrease of Fe grade by 1 to 3 units. Maximum Fe grade of 57.46% was obtained with PACAA (poly acrylamide co acrylic acid) at 20 Hz frequency. Here PACAA acted as a selective flocculant in separation of iron values from gangue.

From Figure 4, it was observed that with an increase in the frequency from 20 to 80 Hz, there is an increase in the Fe recovery by 23 units, i.e., from 4.6 to 27.7% and there is an increase in Fe grade by 2 units from 20 to 60 Hz and there is a slight decrease at 80 Hz frequency in case of blank condition. In case of reagent addition, it can be observed from the Figure 4 that with an increase in frequency there is an increase in Fe recovery by 30 to 40 units, whereas there is a decrease in Fe grade by 2 to 3 units with increase in the bowl speed. Maximum Fe recovery of 46.7% was obtained with corn starch at 80 Hz bowl speed and grade of 57.25% was obtained with Guargum flocculant at 20 Hz bowl speed. Both corn starch and Guargum act as selective flocculants in separation of iron values from the waste slime.

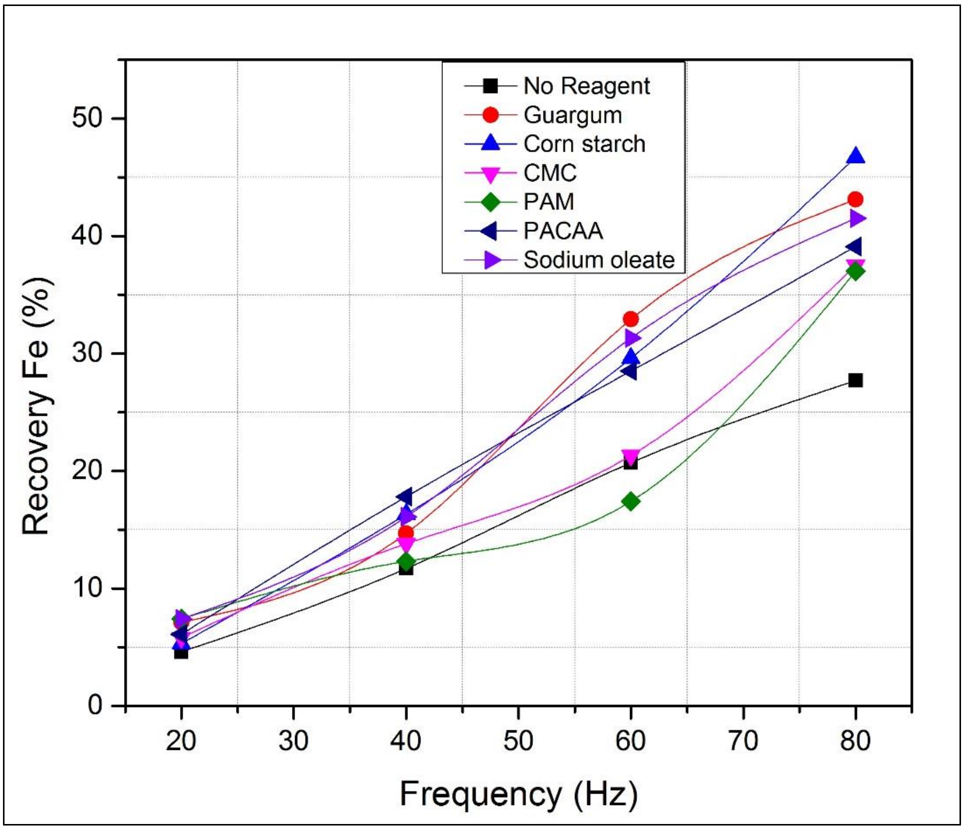

Fig. 4: *The Effect of Bowl Rotation Frequency on Fe Recovery% of Underflow Product at Constant Back Water Pressure of 15 psi.*

By comparing the Fe recovery at all the back water pressures of 5, 10 and 15 psi for all the frequencies, maximum recovery of 62.2% was obtained with reagent CMC at 80 Hz bowl speed at back water pressure of 10 psi and Fe grade of 57.46% was obtained at 20 Hz bowl speed with PACAA flocculant at a back water pressure of 10 psi. Interestingly, both maximum grade and recovery was obtained at 10 psi back water pressure. So it was considered as optimum condition for recovery and grade.

Results on Mozley's Mineral Separator

Mineral separator is a gravity separator used to treat ultrafine particles (slimes) at which the separation depends on the differences in specific gravity of the particles. Different sets of experiments were performed by changing the parameters like collection time and mineral separator (Flat and V-profile) trays at constant pulp density (P.D) of 40%, table speed, amplitude and wash water flow rate of 2.5 lit/min.

Experiments were performed in presence and absence of reagents by changing the parameters to know the optimum condition for the maximum recovery and grade.

Mineral Separator with Flat Tray

The effect of collection time on Fe grade and recovery Fe % in presence and absence of reagents are shown in Figures 5 and 6.

It was observed from the Figures 5 and 6 that with the increase of the collection time of the material from 0 to 90 sec, there is an increase of Fe recovery by 6 units from 23.1 to 29.4% and maximum Fe grade of 52.4% was obtained at a collection time of 90 sec at no reagent condition. Whereas in the case of reagent condition, Fe recovery increased by 40 to 50 units.

Maximum Fe recovery from 7.5 to 56.2% was obtained with PAM (poly acrylamide) flocculant and maximum Fe grade of 51.5% was obtained with Guargum flocculant at 90 sec collection time. It was also observed from the results that Fe recovery is better with the addition of flocculants when compared to no reagent condition and finally it can be said that maximum recovery of Fe is possible with PAM flocculant.

Mineral Separator with V-Profile Tray

The effect of collection time on Fe grade and Fe recovery % in presence and absence of reagents are represented in Figures 7 and 8.

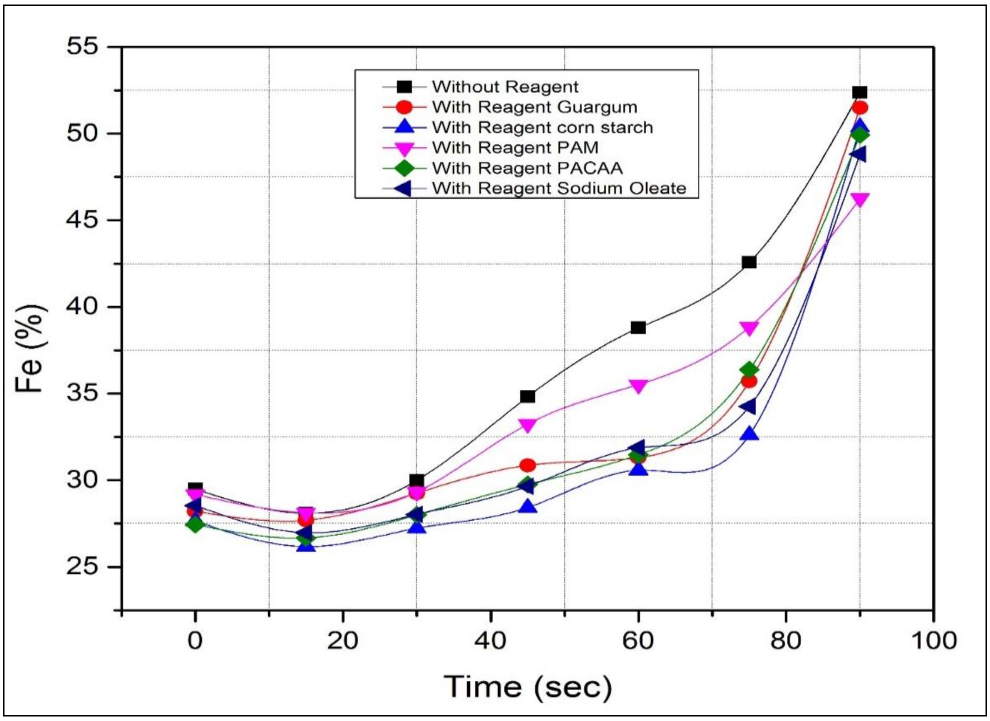

Fig. 5: *The Effect of Collection Time on Fe% in Presence and Absence of Reagents.*

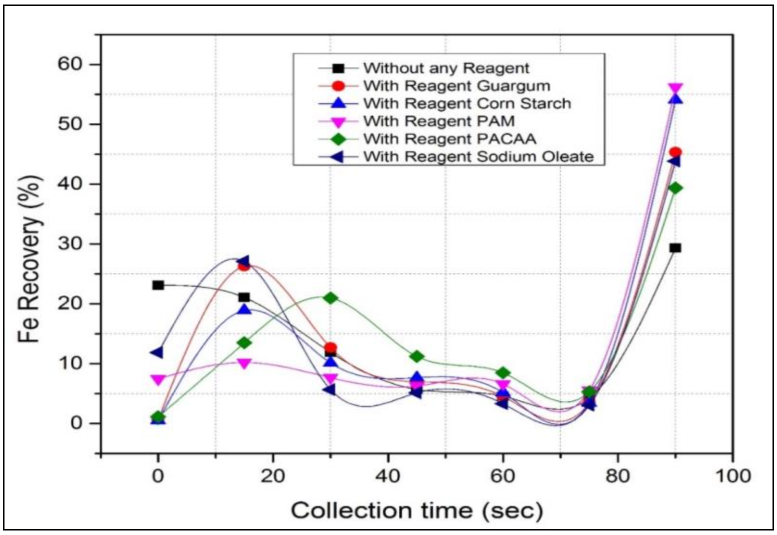

Fig. 6: *The Effect of Collection Time on Fe Recovery% in Presence and Absence of Reagents.*

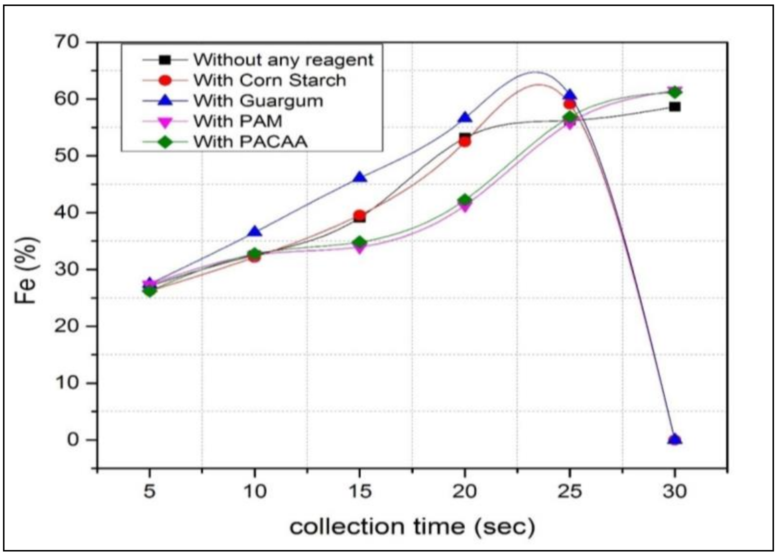

Fig. 7: *The Effect of Collection Time on Fe% in Presence and Absence of Reagents.*

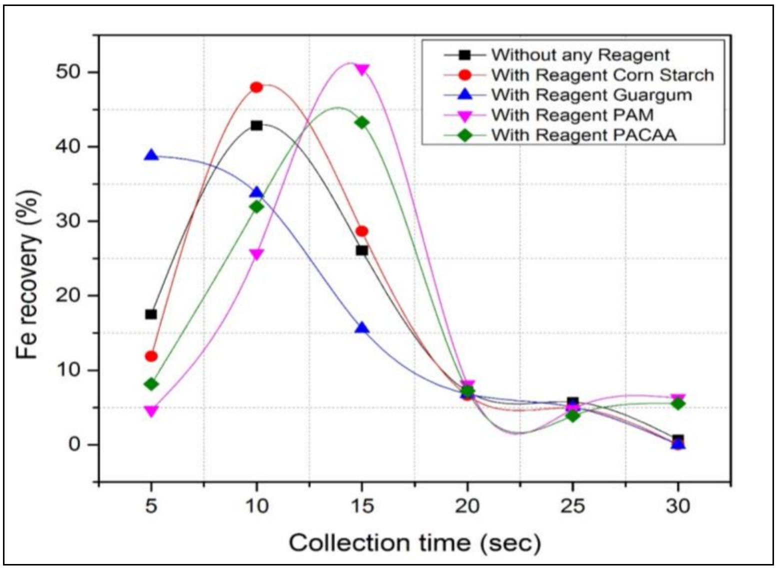

Fig. 8: *The Effect of Collection Time on Fe Recovery% in Presence and Absence of Reagents.*

It was observed from the Figure 8 that with the increase of the collection time from 0 to 30 sec, there is a decrease in the Fe recovery from 17.5 to 0.7% with Fe grade of 58.6%. Maximum Fe recovery of 50.5% was obtained with PAM flocculant at 15 sec of collection time whereas maximum Fe grade of 60.6% was obtained with Guargum at 25 sec collection time.

In the case of mineral separator during the experiment, when the slurry poured on the tray with the help of a beaker the lighter particles generally flow towards the collection end along with the wash water, whereas the heavier particles will be collected lastly and some amount remains on the tray itself. With the addition of flocculants to the slurry, it forms flocs and acts as heavier particles with some gangue and were collected as last fraction giving better recovery and grade.

Bartles Mozley's Laboratory Vanner

Vanner is a high capacity concentrator designed to recover fine particles. Vanner works on the principle of gravity separation. Particle separation depends on the specific gravity. Using this equipment four sets of experiments were done by varying the reagents (flocculants and dispersant) and one without using any reagent at constant parameters like belt slope of 2°, pulp density (P.D): 20% and belt speed of 20 rpm. The results both in presence and absence of reagents are graphically represented in Figures 9 and 10.

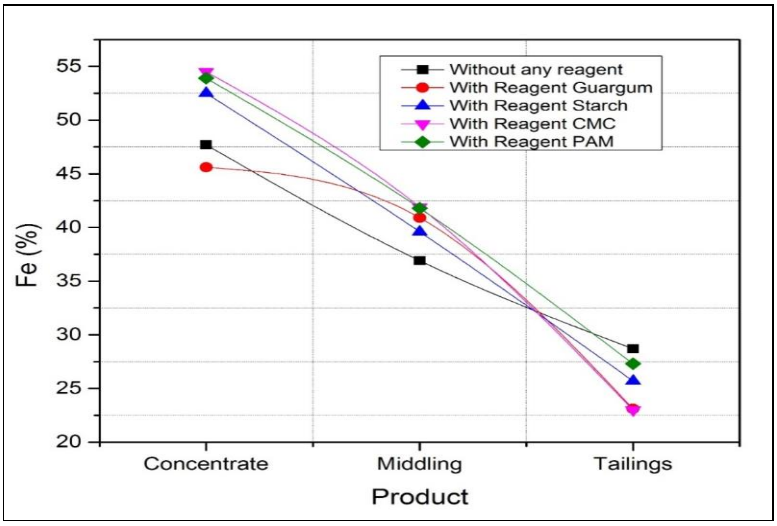

Fig. 9: *The Effect of Reagents on the Fe% of the Products.*

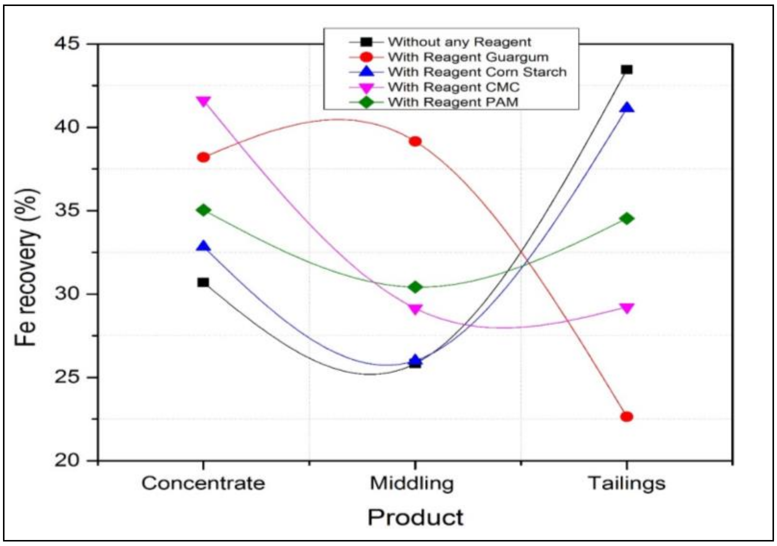

Fig. 10: *The Effect of Reagents on Fe Recovery % of the Products.*

It was observed from the Figures 9 and 10 that with use of reagents there is an increase in the Fe recovery % by 11 units from 30.7% without reagent to 41.6%. With reagent CMC, there is an increase in Fe grade from 47.7 to 54.5% Fe. CMC acts as a flocculant in acquiring better Fe recovery and grade. Similar effect is also observed on the middling product of Vanner. With the addition of reagent PAM, there is an increase in Fe recovery by 5 units from 25.8% without reagent to 30.4% and increase in Fe grade with the addition reagents from 36.9% without addition of reagent to 41.9% with reagent CMC and PAM. Hence, both PAM and CMC are considered as better flocculating agents for the improvement of recovery and grade.

Zeta Potential Measurement

The zeta potential values of iron ore slime in presence and absence of reagents at different pH values are shown in Figure 11.

From the Figure 11, it is evident that with an increase in pH from 3 to 11, there is a decrease in zeta potential value from positive to negative, thereafter increases negatively. The isoelectric point (IEP) was found to be 4.06. Corn starch, Guargum decreased the isoelectric point to 3.73 and 3.49, respectively. At high pH values, the zeta potential of Guargum, CMC and PACAA were decreased indicating the flocculating behavior of these reagents onto the iron ore slime. However, sodium hexametaphosphate (SHMP) show a little increase of negative zeta potential value compared to all other reagents, which shows its dispersing behavior. SHMP is observed to be a dispersing agent in alkaline pH. However, all the other flocculants such as corn starch, Guargum, CMC and PACAA show flocculating behavior at higher pH values.

Results of Flocculation

The settling behavior of iron ore slime sample was studied by varying the settling time. The effect of time interval on flocculation of iron ore slime was observed with respect to settled weight % and Fe grade. Flocculation and dispersion studies were carried by using different dispersant and flocculants with optimum dosages and at optimum pH values, where the recovery grade is maximum. Optimum settling time was observed to get better grade of Fe. The effect of settling time on yield % (settled wt %) and Fe% in presence and absence of reagents are represented in Figures 12 and 13.

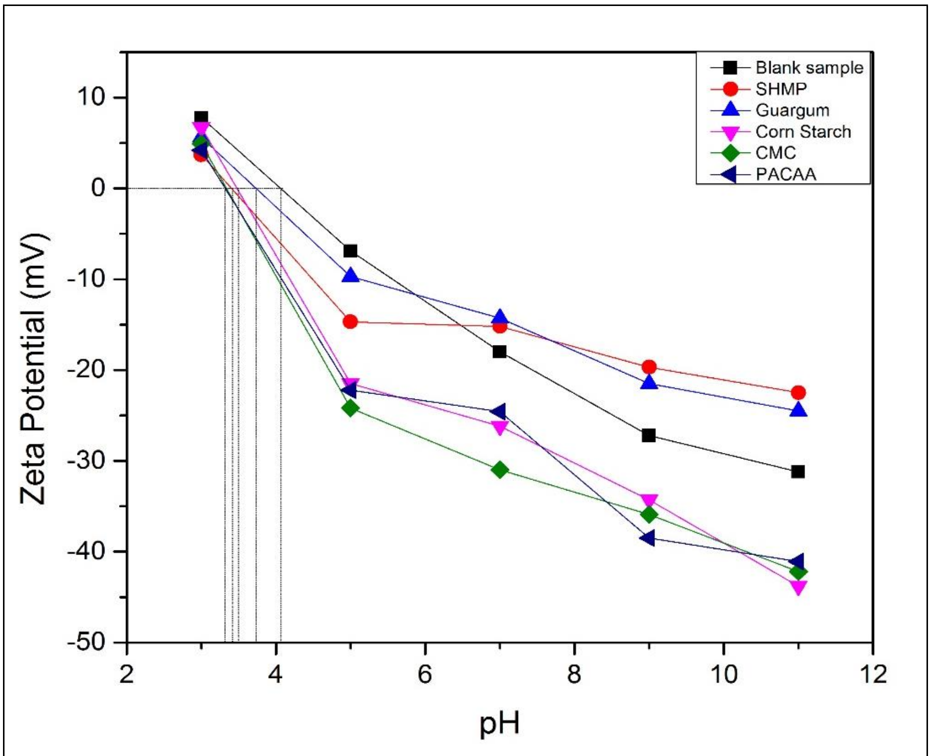

Fig. 11: *The Effect of Reagents on the Zeta Potential of Iron Ore Slime.*

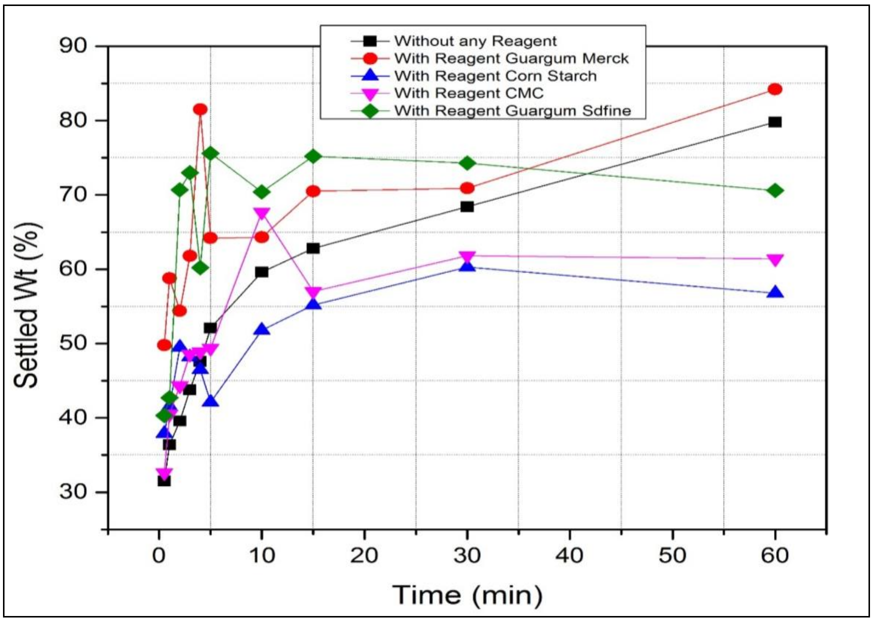

Fig. 12: *The Effect of Settling Time on Settled wt% in Presence and Absence of Reagents.*

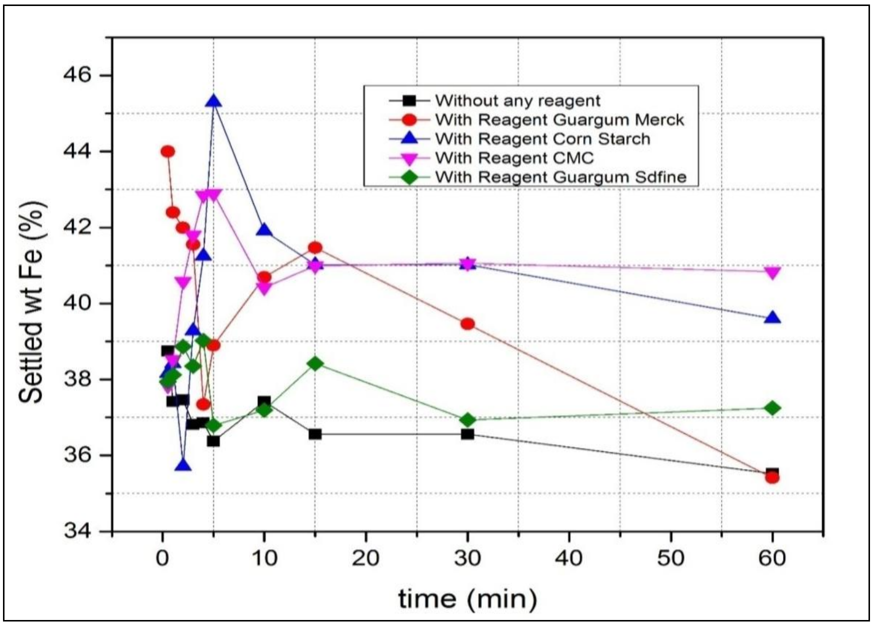

Fig. 13: *The Effect of Settling Time on Fe% in Presence and Absence of Reagents.*

Various sets of flocculation experiments were performed by keeping the parameters like pulp density (P.D):1% constant, at natural pH and without the addition of any reagent. It was observed from the Figures 12 and 13 that with the increase of the settling time from 0.5 to 60 min there is an increase in the settled wt% from 31.5 to 79.8% and decrease in Fe% from 38.75 to 35.52%. The decrease in Fe% is due to the settling of gangue along with iron particles. Experiments were done by the addition of reagents with optimum dosages and at optimum pH values at constant pulp density of 1%. From the results, it was observed that with the increase of settling time from 0.5 to 60 min there is an increase in the yield % from 49.8 to 84.2% with reagent Guargum with a dosage of 20 ppm at pH 10.5, it is due to better formation of flocs. Maximum grade of 45.3% Fe was obtained at 5 min settling time with reagent corn starch (20 ppm at pH 11.5), as corn starch acts as a selective flocculant. Starch facilitates the selective adsorption on iron particles, which in turn leads to enhancement in selectivity and recovery of iron mineral in iron ore slime.

CONCLUSIONS

Based on the test results, the following conclusions are drawn:

1. The iron ore slime contains 90% of the particles are finer than 80 µm, and assayed 34.75% Fe.
2. The optimum condition for the up gradation of iron ore slime using Falcon concentrator, to obtain maximum recovery was at 10 psi back water pressure, 80 Hz frequency (bowl speed) in presence of reagent carboxy methyl cellulose. The underflow product of the falcon concentrator was upgraded to 48.72% Fe with 62.2% recovery. But the maximum grade of 57.46% Fe was obtained with PACAA flocculant at 20 Hz frequency and 10 psi back water pressure.
3. The optimum condition for the upgradation of iron ore slime using mineral separator, to obtain maximum recovery was with last fraction of the flat tray with collection time of 90 sec and at 2.5 lit/min wash water flow rate in presence of reagent PAM. The product was upgraded to 46.3% Fe with 56.2% recovery. Whereas with V-profile tray the product was upgraded to 60.6% Fe with Guargum.
4. The optimum condition for the upgradation of iron ore slime using laboratory Vanner, to obtain the maximum recovery was with belt slope of 2°, belt speed of 20 rpm and with the use of reagent. The concentrate product of the laboratory Vanner upgraded to 54.5% Fe with 41.6% recovery with reagent carboxy methyl cellulose. Whereas the middling product of the Vanner upgrade to 41.8% Fe with 30.4% recovery with reagent PAM (polyacrylamide).
5. Selective flocculation and dispersion studies were performed using different reagents at different settling times. Maximum up gradation of 45.3% Fe was obtained using 20 ppm modified corn starch flocculant, 1 ppm polyvinyl pyrrolidone dispersant at pH 11.5 and settling time of 5 min.
6. Zeta potential studies revealed that at higher pH flocculation usually occur. Also, sodium hexametaphosphate was showing high dispersing property.

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