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Recovery of Iron Values from Iron Ore Slimes by Selective Magnetic Coating

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ABSTRACT

Effective separation of iron values from iron ore slimes using a wet magnetic separator and selective magnetic coating is reported. The selectivity of coating for enhancing the magnetic response of iron particles was achieved by controlling the surface properties of the particles, i.e., electrokinetics potential, through pH adjustment and use of sodium hexa-metaphosphates as the dispersant. Experiments were conducted with synthetic mixtures of iron, alumina, and silica using oleate colloidal magnetite at different fields of magnetic intensity. After the iron values were separated from the synthetic mixtures, the technique was applied to iron ore slimes of Joda area of India. Iron concentrate containing ~65.9% Fe, 1.0% SiO_2, and 1.56% Al_2O_3 was obtained from a feed containing ~59.0% Fe, 3.98% SiO_2, and 6.5% Al_2O_3. The effects of pH, magnetic intensity, and addition of sodium oleate were recorded. The X-ray diffraction (XRD) studies brought out the significant enrichment of iron in the concentrate relative to the feed, and alumina–silica enhancement in the tailing products. The scanning electron microscopy (SEM) study highlighted the coagulation of hematite and magnetite in the presence of sodium oleate, thereby facilitating the effective separation.

Key Words. Selective magnetic coating; Iron ore slimes; Magnetic separation

INTRODUCTION

Most of the iron ore mines in India that produce hematite ores have washing plants to produce lumps and fines and also to remove a part of fine gangue materials present in the product. During the washing process, around 8–10 million tons of slimes (210 μm size) containing ~48–60% Fe is discarded every year. These slimes cannot be used in iron and steel making because they contain a high accumulation of gangue materials. (1, 2). The slimes thrown into the tailing ponds also pose enormous environmental hazards. Thus, the safe disposal or utilization of such vast mineral wealth remains as a challenging task for the mineral engineers of the country. Conventional mineral processing techniques attempted previously to recover the iron values have had limited success (3–7). The removal of alumina in any of the processes was not quite satisfactory. However, the productivity of the sinter plant is linked to the lower alumina content in the feed. Thus, a critical analysis of the earlier work has led us to study different mineral processing techniques which can successfully convert the beneficiate slime into a product acceptable for iron and steel production without affecting productivity.

Selective magnetic coating as a method of fine particle separation has received some attention in recent years. The process involves selective adsorption of fine magnetite or colloidal magnetite onto the desired mineral surface, which renders the coated grains amenable for recovery by magnetic separation techniques. The factors that influence the addition of the magnetite include the electrical charge of the minerals, concentration of magnetite, pH, presence of adsorbed surfactant, and electrolytic concentration of pulp (8). The control of these parameters has reportedly led to the separation of many minerals, such as chalcocite from silica, sphalerite from other gangue minerals, coal from ash, and calcite from apatite and dolomite (9, 10). This paper describes the beneficiation studies conducted on a synthetic mixture and natural iron ore slime samples from Joda, India, with a view to upgrade the level of iron and reduce the silica–alumina content to an acceptable limit. The effects of different variables such as pH, magnetic intensity, and concentration of sodium oleate and oleate colloidal magnetite are discussed.

EXPERIMENTAL METHODS

Selective magnetic coating studies were carried out on a synthetic mixture of iron oxide, quartz, alumina, and kaolinite, as well as on the natural iron ore slime samples obtained from Joda Iron ore washing plant, Orissa.

For the study of synthetic feed, a hand-picked sample of pure hematite and crystalline pure quartz (99.9% SiO_2) was obtained from Orissa Mining Corporation and Saroj Mining Corporation, Karnataka, respectively. The samples of the desired size were crushed and ground in a stainless ball mill at 40% solids concentration in deionized water. The quartz sample was washed several times with dilute HCl followed by distilled water to remove any contaminated iron and super fines. Laboratory grade pure alumina (gibbsite) and kaolinite were used in the synthetic mixture. Synthetic feed materials were prepared by mixing hematite, quartz, alumina, and kaolinite in desired proportions. The purity of these materials was checked by X-ray diffraction (XRD) study and chemical analysis.

The iron ore slime sample from Joda iron ore washing plant was thoroughly mixed before a representative sample was drawn from it. The sample was than dried at 105°C, powdered just to break the agglomerated particles, and used in the investigation. A portion of the sample was subjected to wet sieving using ISS standard sieves. Each of these size fractions thus produced was analyzed for Fe, SiO_2, Al_2O_3, and loss on ignition (LOI).

Colloidal magnetite was prepared by the combination of the ferrous and ferric ammonium sulfate in 1:1 ratio in distilled water at 70°C (10). NaOH was added into the resulting solution with constant stirring. The precipitated magnetite thus obtained was washed thoroughly to remove excess NaOH and unreacted salts. The oleate colloidal magnetite was prepared by adding 10^{-2} M solution of sodium oleate at pH 11 and boiling the sample until the magnetite was completely dispersed. The volume of the suspension was made up to 100 mL. The concentration of colloidal oleate magnetite was found to be 0.024 g/mL.

The magnetic separation studies were carried out with a wet high-intensity magnetic separator supplied by Rapid Box Mag separator, U.K. The magnetic intensity of the instrument was varied by grid gaps and applied current. Prior to magnetic separation, all conditioning on coating tests of both synthetic mixture and iron slimes were carried out separately in a 1-L beaker at a solid–liquid ratio of 1:10. The sample (natural slimes or synthetic mixture) was first conditioned for 10 min at a desired pH. The pH of the solution was regulated by adding either NaOH or $HClO_4$ acid. Oleate colloidal magnetite or sodium oleate was added in desired quantity into the suspension. The dispersant sodium hexa-metaphosphate (NaHMP) was added while the sample was stirred in a digital stirrer. After proper conditioning, the slurry was then passed through the magnetic separator slowly. The magnetic and nonmagnetic fractions were collected separately, dried, and analyzed for Fe, Al_2O_3, and SiO_2 to determine the quality and recovery of the mineral values.

The electrophoretic mobility of the mineral particles was measured in the pH range of 2–10 using the Rank Brothers Mark II. The electrophoresis apparatus in the flat cell configuration and was converted to zeta-potential by means of Smoluchowski equation. All three products of Joda slimes (feed, magnetic, and nonmagnetic) were subjected to XRD and scanning electron microscope (SEM) analysis. Phillips (PW-1710) X-ray diffractometer with Cu $K\alpha$ target operating at 40 kV and 20 nA, and a JEOL SEM (JSM 35CF) attached with Kevex Sigma Gold EDS system were used for this purpose.

RESULTS AND DISCUSSION

Because H^+ and OH^- are the potential determining ions for oxide minerals (11), the zeta-potential is measured at various pH values and shown in Fig. 1. The isoelectric points of hematite, colloidal magnetite, and oleate colloidal magnetite occur at pH 7.1, 6.8, and 5.8, respectively. The zeta-potential of quartz and kaolinite was found to be negative throughout the pH range studied. The point of zero charge values of these minerals are comparable with the reported values. It appears that magnetic coating between hematite and oleate colloidal magnetite may take place due to electrostatic interaction or due to Vanderwaals forces. The maximum coating and separation of hematite takes place at pH 7.1, which has been verified by the experimental results reported in the following subsections. The selective coating of the oleate magnetite on hematite surface was brought about by controlling the pH of the suspension. The quartz and kaolinite particles, which are negative at the experimental pH, do not adsorb electrostatically with hematite or colloidal magnetite. In addi-

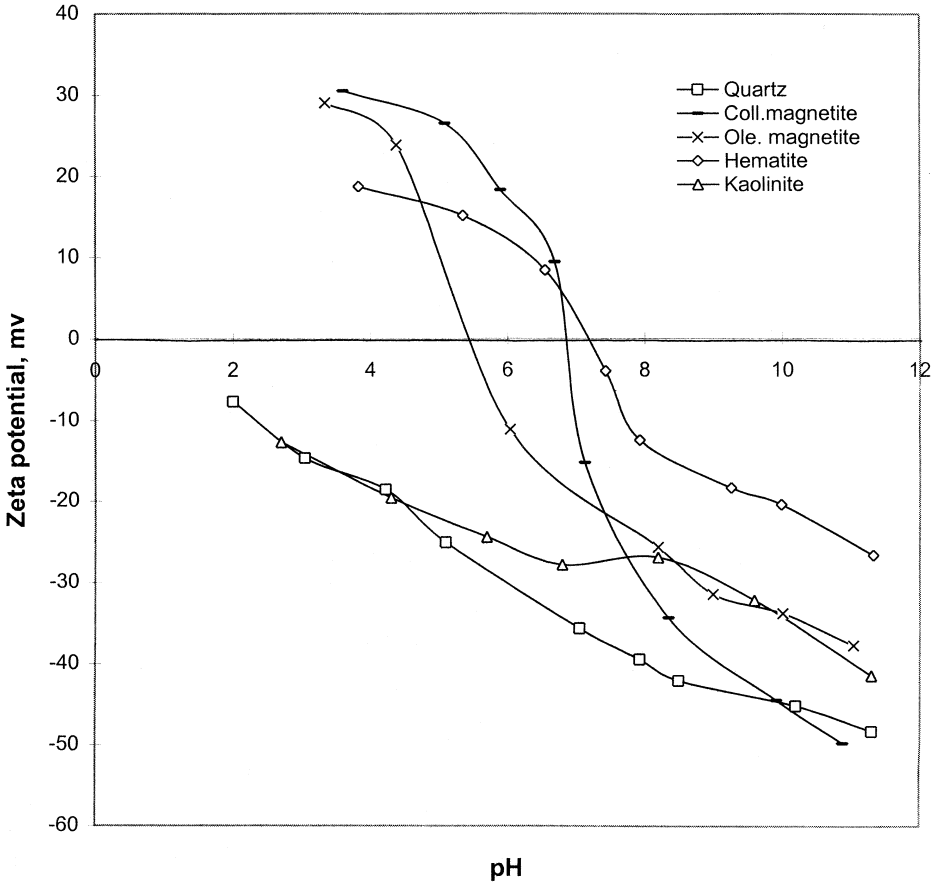

FIG. 1 Variation of zeta potential of hematite, colloidal magnetite, oleate magnetite, quartz, and kaolinite with pH (2×10^{-3} M, KNO_3).

tion, negatively charged oleate ions do not have any specific attachment for kaolinite or quartz, but have high adsorption affinity with hematite at pH 7.1–7.5 (12).

Studies of Synthetic Mixtures

The coating experiments carried out earlier (13) on the synthetic mixtures of hematite, quartz, and corundum indicated that the recovery and grade of hematite was better at pH 7.2, at which the zeta-potential of hematite is found to be zero. The experiments conducted in the pH range of 4.5–11 indicated that the maximum recovery of hematite (~98%) was obtained at the pH 7.2, but was not as remarkable at other pH values. The effect of the oleate colloidal magnetite concentration showed that only 0.25 mL of colloidal suspension per gram of hematite is sufficient to obtain a significant increase in iron grade and recovery. The studies also confirmed that the separation of hematite from corundum was achieved by using small dosages of NaHMP as the dispersant. Further experiments were carried out only at pH 7.2 using 0.25 mL of oleate colloidal magnetite (2.8 g/kg) and NaHMP as the dispersant.

Quartz, gibbsite, and clay (kaolinite and montmorillonite) are generally considered to be the gangue minerals in the Indian iron ore slimes (14). Therefore, the recovery of iron ore fines from synthetic mixture of hematite–quartz, hematite–gibbsite, and hematite–kaolinite by selective magnetic coating were carried out. The results of separation of hematite from these mixtures as a function of magnetic intensity is shown in Fig. 2. Note that in all cases the recovery increased with an increase in magnetic intensity. The recovery of hematite from hematite–quartz mixture was better than the other that of two mixtures. This is because the quartz particle is more negative than kaolinite, and alumina oxide at the experimental pH (7.2) does not adsorb colloidal oleate magnetite because of physical entrapments. The results of the study indicated that more than 90% of hematite recovery could be obtained at magnetic intensity of 7.8 kG. Even at 5.0 kG, the recovery of hematite in all cases was more than 50%. Because the applied magnetic intensity is much less than the required intensity for hematite separation in any wet magnetic separation techniques, it can be inferred that the response to magnetic separation is enhanced by the addition of a small amount of colloidal magnetite in conjunction with a surfactant. The addition of oleate magnetite further facilitated selective attachment with the hematite surface, similar to the effects of flotation. In all cases, however, a very small dosage of oleate colloidal magnetite is needed for effective separation of hematite from other gangue materials. Having established the efficacy of the selective magnetic coating on the separation of iron oxide from kaolinite, quartz, and gibbsite, further experiments were conducted on natural samples obtained from the Joda washing plant.

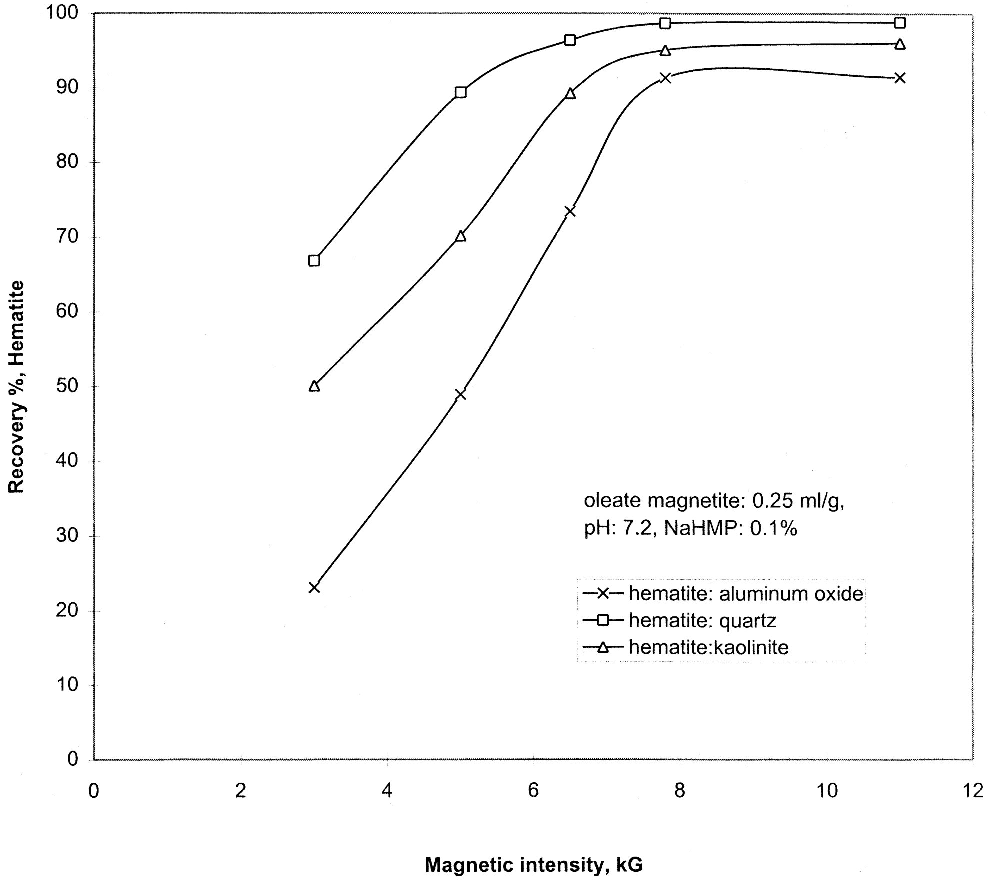

FIG. 2 Effect of magnetic intensity on the recovery of hematite from synthetic mixtures.

Studies on Joda Iron Ore Slimes

The chemical composition of the Joda ore slimes is shown in Table 1. The sample contained ~58.9% Fe, 3.98% SiO_2, and 6.5% Al_2O_3. The size analysis of the slime sample is shown in Table 2. The 80% passing size was around 35 μm. The results of the size analysis indicated that alumina and silica tend to

TABLE 1
Chemical Analysis of Joda Iron Ore Slimes

Constituents	Percent
Fe_2O_3	84.5
SiO_2	3.98
Al_2O_3	6.5
LOI	5.1

TABLE 2
Size and Chemical Analysis of Joda Slimes

Size (μm)	Weight (%)	Fe (%)	Al_2O_3 (%)	SiO_2 (%)	LOI (%)	Fe distribution (%)
+ 500	1.6	62.0	6.11	1.4	3.82	1.7
–500 + 250	4.5	62.83	5.60	1.0	3.47	4.8
–250 + 150	2.5	61.71	6.13	1.1	4.50	2.6
–150 + 45	9.1	59.78	8.17	1.1	5.23	9.2
–45 + 30	5.3	54.73	10.21	4.9	6.57	4.9
–30	77.0	58.64	6.10	4.8	5.16	76.8
Head	100.0	58.85	6.45	4.15	5.12	100.0

concentrate in the finer fractions. Similar size distributions were also reported in the other iron ore slimes of India (15). The iron ore sample from Joda was very fine and consisted of hematite (Fe_2O_3) and goethite (FeO-OH) as its major phases. The presence of magnetite (Fe_3O_4) in subordinate amount was recorded under reflected light microscope. The associated gangue minerals included gibbsite and very small amount of quartz. The XRD patterns revealing all of these phases are shown in Fig. 3F.

The bulk slime sample was subjected to different magnetic intensities and then mixed with 0.8 g/kg of dispersant, NaHMP. The hexametaphosphate ion plays a vital role in the dispersion of the minerals. The adsorption of phosphate ions makes the surface of alumina minerals more negative and thereby the particles remain in dispersed phase due to mutual repulsion of strongly negatively charged particles (16). The results of magnetic separation of Joda iron ore slimes with and without sodium hexametaphosphate is shown in Table 3. The results indicate that in the absence of the dispersant, the iron grade of 62–63% can be obtained with 50–60% iron recovery from a feed containing around 58.9% Fe. The grade of iron concentrate decreases with an increase in magnetic intensity. The maximum iron recovery of 61% was achieved by increasing the magnetic intensity to ~11.4 kG. Note also that both iron grade and recovery increased considerably when a small dosage of dispersant (NaHMP) was used. The alumina and silica levels decreased to 2.9 and 2.0%, respectively. The maximum iron concentrate around 65.3% Fe with 53.5% recovery was obtained by applying a magnetic intensity of ~7.8 kG. The recovery of iron increased to 72.7% by increasing the magnetic intensity to 11.4 kG, but at the same time the grade of iron decreased to 64.1% and the level of alumina increased.

The recovery of iron values from Joda iron ore slimes was carried out using different concentrations of oleate colloidal magnetite as the coating agent in the feed pulp. Because the slime sample contained some amount of magnetite,

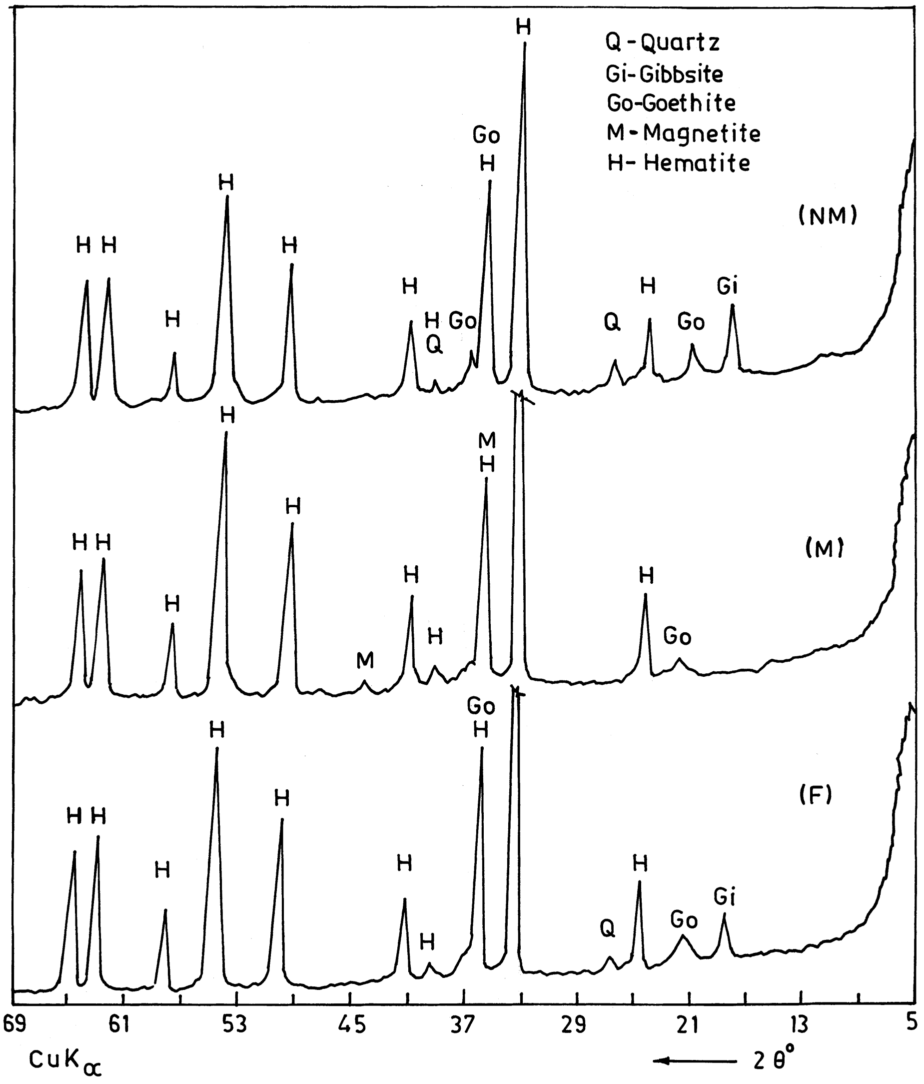

FIG. 3 XRD spectra of Joda iron ore slimes feed (F), magnetic (M), and nonmagnetic (NM).

TABLE 3
Magnetic Separation of Joda Iron Ore Slimes

Magnetic intensity (kg)	Weight (%)	Fe (%) without NaHMP	Fe (%) recovery	Weight (%)	Fe (%) with NaHMP	Fe (%) recovery
3.0	11.2	63.3	12.0	9.0	66.18	10.1
5.0	22.0	63.0	23.3	25.1	66.0	28.3
6.5	35.7	62.8	37.9	–	–	–
7.8	46.9	62.7	49.9	47.9	65.3	53.5
11.0	52.5	62.3	55.1	59.7	64.5	65.5
11.4	55.8[a]	62.0	61.0	66.6[b]	64.1	72.7

[a] Al_2O_3: 3.5%; SiO_2: 2.2%.

[b] Al_2O_3: 2.9%; SiO_2: 2.0%.

TABLE 4
Effect of Sodium Oleate and Oleate Colloidal Magnetite on the Magnetic Separation of Slimes (Magnetic Intensity: 7.8 kG, NaHMP: 0.8 g/kg, pH 7.2)

Sodium oleate, g/kg	Weight (%)	Fe (%)	Fe (%) recovery	Oleate magnetite mL/g	Weight (%)	Fe (%)	Fe (%) recovery
0.4	58.7	65.3	64.0	0.125	57.6	65.54	63.6
0.8	59.4	65.3	65.0	[b]0.25	57.6	66.18	63.9
1.0	[a]57.2	65.9	63.3	0.4	59.0	65.31	65.0
1.4	57.2	65.8	63.5	0.5	65.6	64.20	70.8
1.6	57.2	65.5	64.1				

[a] Al_2O_3: 1.56; SiO_2: 0.98.
[b] Al_2O_3: 1.48; SiO_2: 0.90.

it was thought worthwhile to take the advantage of such a phase. Experiments were carried out using different concentrations of sodium oleate. A constant dose of 0.8 g/kg of dispersant and a magnetic intensity of 7.8 kG were used in all the experiments. The effects of sodium oleate and oleate colloidal magnetite on the recovery of iron values from iron ore slimes are shown in Table 4. The results show that an iron concentrate containing around 65.9% Fe with 63% recovery can be obtained using 1.0 g/kg of sodium oleate. The SiO_2 and Al_2O_3 content was reduced to 0.98 and 1.56%, respectively. In addition, a sodium oleate concentration of 0.4 g/kg was sufficient to provide good grade iron concentrate. On the other hand, the results on colloidal magnetite indicated that a concentration of 0.25 mL/g (2.8 g/kg) gave rise to an iron concentrate of 65.54% with 63.6% recovery, and 0.9% silica and 1.48% alumina. The critical analysis of the experimental results suggests that because the recovery of good grade iron is achieved by the addition of sodium oleate, sodium oleate may be preferred over oleate magnetite because of the higher cost and higher energy requirements of the latter. Because fine magnetite grains present in the feed become coated with sodium oleate, they form the nucleus and coagulate the other iron-rich particles. Thus, the magnetic susceptibility and efficiency in magnetic separation are enhanced.

The effect of pH on the magnetic separation of Joda slime is shown in Table 5. A magnetic intensity of 7.8 kG, 1.0 g/kg of sodium oleate, and 0.8 g/kg of NaHMP were kept constant during the changes in pH values. A concentrate of 65.9% iron with 63.3% recovery was obtained at a pH value of 7.2. At all other pH values, the grade and recovery of iron content decreased, which attests to its selectivity. The enrichment and efficiency also show a maximum value at pH 7.2.

TABLE 5
Effect of pH on Magnetic Separation of Slimes (Magnetic Intensity: 7.8 kG, Sodium Oleate 1.0 g/kg, NaHMP: 1.6 g/kg)

pH	Weight (%)	Fe (%)	Fe (%) recovery	Enrichment (%)	Efficiency (%)
6.0	54.1	64.23	59.4	50.00	29.00
7.2	57.2	65.90	63.3	60.73	38.44
8.5	47.1	64.79	52.0	53.77	27.96
10.0	48.0	64.50	52.3	49.26	25.76

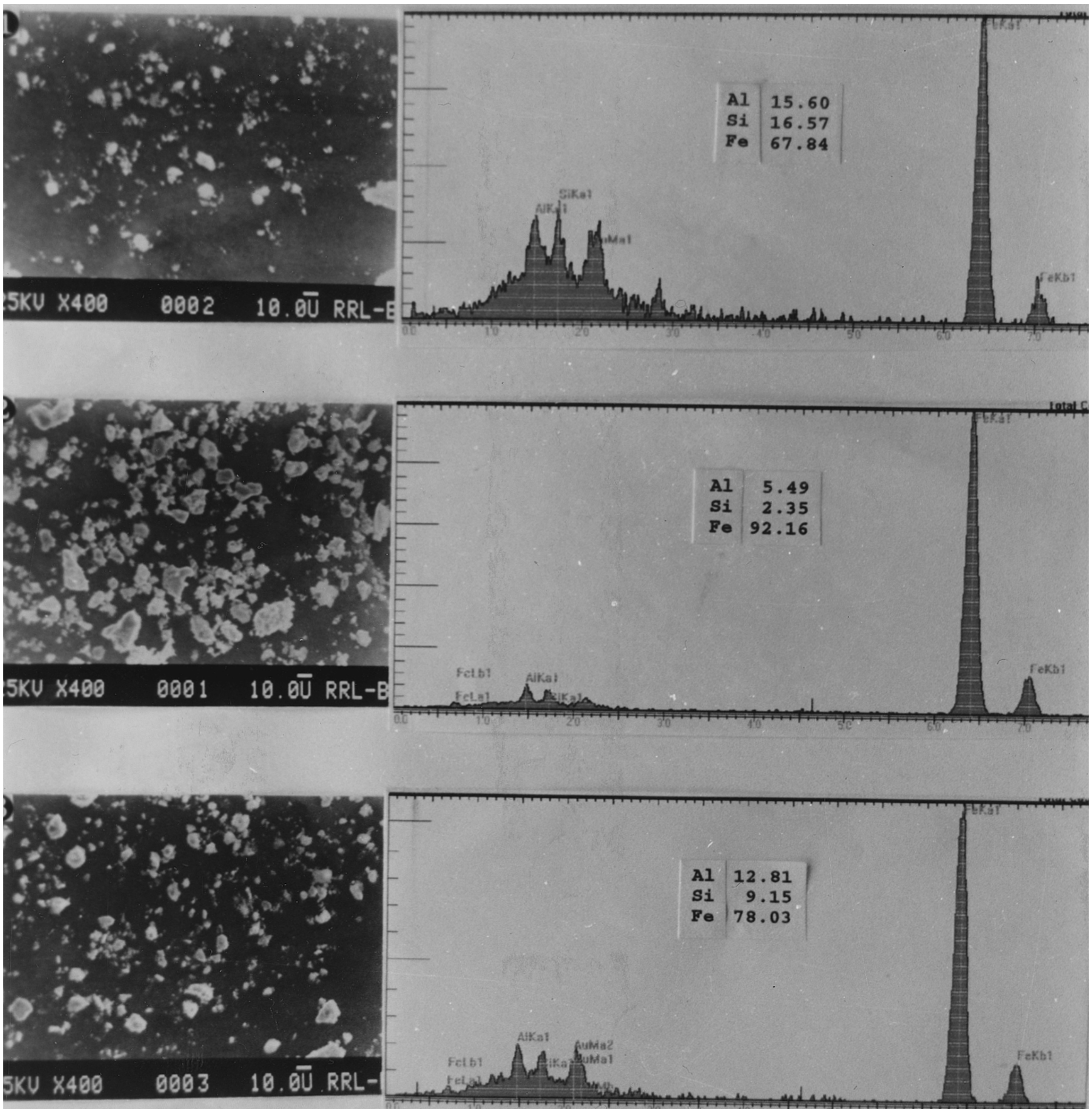

FIG. 4 Electron micrographs and EDS spectra of (1) feed; (2) magnetic, and (3) nonmagnetic.

The XRD patterns of feed, magnetic, and nonmagnetic samples on the coated slime sample are illustrated in Fig. 3. The X-ray characteristic peaks of hematite (2.69 Å) show higher intensity in magnetics than that of feed, indicating the larger content of this phase. Conversely, the peak intensities diagnostic of gibbsite and quartz show a relative increase in the nonmagnetic sample. The electron micrographs of all three products are shown in Fig. 4. The magnetic grains are relatively large, which may be due to coagulation of iron particles in the presence of sodium oleate. In contrast, the nonmagnetic particles are very fine and comparable to those in the feed. The energy-dispersive system (EDS) spectra for all three products exhibit the distinct difference in the height of the alumina and silica spectra. A marked difference in Fe, Al, and Si elemental distribution between these three products is also noted (Fig. 4).

CONCLUSIONS

Selective magnetic coating carried out on synthetic mixture and natural iron ore slimes indicated that it is technically possible to separate iron fines by rendering selective coating on hematite surfaces followed by magnetic separation. The separation is largely dependent on the charge of the surface. Hematite can be separated suitably by using the dispersant (NaHMP) at pH 7.2 and applying around 7.8 kG of magnetic intensity. Studies on natural ore slimes have confirmed that using only sodium oleate along with the dispersant can give rise to encouraging results. The natural magnetite grains present in the slime coagulate the hematite particle during selective coating and enhance the magnetic properties of the iron oxide minerals present in the slime. A concentrate containing ~65.9% Fe, 1% SiO_2, and 1.56% Al_2O_3 was obtained by this technique and can be suitably used in sinterfeed.

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