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## **Investigation on the Effect of Ultrasonic Pretreatment on Selective Separation of Iron Values from Iron Ore Tailings by Flocculation**

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### **ABSTRACT**

Selective flocculation studies were carried out on Barsuan iron ore tailings having 50.5% iron, 7.2% alumina, and 7.8% silica in the absence and presence of ultrasonication at different experimental conditions using sodium hexametaphosphate as a dispersant and starch as a flocculant. The imposition of ultrasonication resulted in a marked improvement in grade as well as recovery. The results indicate that ultrasonication is able to mitigate the adverse effects of gangue materials. A concentrate assaying 59% iron with 75% recovery could be obtained without ultrasonication, while the application of ultrasonication led to an improvement in concentrate grade to 65% with a corresponding increase in recovery to 91%. The improvement is attributed to the disruption caused at a solid/liquid interface which results in favorable modification of the mineral surface, facilitating selective adsorption of flocculant on the iron ore fines. This, in turn, leads to enhancement in selectivity and recovery. In addition, ultrasonication also leads to effective dispersion, causing enhanced activity.

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## INTRODUCTION

Iron is the second most common metal in the earth's crust. The principal minerals in iron ores are hematite, magnetite, limonite, and siderite. The major iron-ore-producing countries in the world are the former USSR, Brazil, India, the United States, and Australia. Run-of-mine iron ores are normally not suitable for economic smelting, and hence require beneficiation. The beneficiation processes are basically classified as (a) mineralogical beneficiation (for the enrichment of iron content and the decrease of impurities like alumina, silica, phosphate) and (b) sizing of the ore and agglomeration of the ore fines (1). As mentioned above, India is one of the leading producers of iron ores in the world. The total iron ore reserves of India amount to 19.3 billion tonnes (13.1 billion tonnes hematite and the rest magnetite). Although these ores are rich in iron content, they also contain large amounts of alumina. An adverse alumina/silica ratio (ideally it should be less than 1, with an alumina/iron ratio below 0.05) is detrimental to blast furnace chemistry as well as to sinter plant operations. A large amount of alumina in iron ore and sinter leads to a highly viscous slag in the blast furnace, resulting in a high coke rate, so washing is a mandatory step for preparing ore for a blast furnace. Washing of iron ore leads to three products: coarse ore lumps ( $-10$  mm size) which are directly charged to the blast furnace; classifier fines ( $-10$  mm +  $150$   $\mu\text{m}$ ) which are fed to a sintering plant with or without beneficiation (essentially gravity separation); and slimes ( $-150$   $\mu\text{m}$ ) which are presently discarded as waste. The losses of iron in slimes (assaying 50–60% Fe) are estimated to be of the order of 10–25% by weight of the iron ore mined. This operation, coupled with the increased mechanization of mines, has resulted in the substantial production of inferior quality fines and slimes. Besides the loss of iron, enormous environmental hazards result. There therefore exists an urgent need to find ways of utilizing these slimes.

A considerable amount of work has been carried out in the past by various workers to process such iron ore fines by selective flocculation (2–4). The majority of data reported are on synthetic mineral mixtures (hematite, clay, quartz, etc.) (5–8) and a few are on natural ore slimes (2, 3, 9). Ultrasonication has attracted a lot of attention in recent years due to its ability to enhance the selectivity of mineral suspension in the fine particle size range (10–13). The beneficial effects of ultrasonication in the selective flocculation of iron ore fines have been reported by various workers (14, 15). The present investigation was undertaken to study the effect of ultrasonic pretreatment on the flocculation selectivity of iron ore from Barsua, India.

## EXPERIMENTAL

### Materials

#### *Iron Ore Sample*

The iron ore sample used in this study was obtained from Barsuan iron ore plant of the Steel Authority of India Limited. In the coarse size range up to 45  $\mu\text{m}$ , the particle size distribution of tailings was carried out by wet sieving using standard Tyler sieves, while Fritsch microsieves were used below 45  $\mu\text{m}$ . Chemical analysis of the head sample and particle size analysis with chemical composition of the different fractions of tailing samples are given in Tables 1 and 2, respectively. As noted, 70% of the particles were below 45  $\mu\text{m}$ . The alumina-containing phases in the said slime were earlier characterized as 2  $\mu\text{m}$  grains of kaolinite, illite, and montmorillonite (2).

#### *Reagents*

Laboratory-grade reagents like corn starch and sodium hexametaphosphate were used as the flocculant and dispersant, respectively. AR grade NaOH was used for the preparation of starch solution.

### Methods

#### *Preparation of Starch Solution*

A 1% slurry of the starch was heated to 85°C in the presence of 0.5% sodium hydroxide and rapidly cooled to room temperature. The heating and cooling cycles were kept below 5 minutes (16). A fresh solution was prepared and used every day.

TABLE 1  
Chemical Analysis of Barsuan Iron  
Ore Tailing

Constituents	Assay (%)
Fe	50.5
Al <sub>2</sub> O <sub>3</sub>	7.2
SiO <sub>2</sub>	7.5
CaO	0.1
MgO	Traces

TABLE 2  
Particle Size Analysis with Chemical Composition of Different Size Fractions of Barsuan  
Iron Ore Tailing

Size ( $\mu\text{m}$ )	Wt%	Fe (%), total	$\text{SiO}_2$ (%)	$\text{Al}_2\text{O}_3$ (%)
+ 210	7.4	60.58	2.70	4.82
- 210 + 150	6.0	61.48	1.82	4.40
- 150 + 105	8.3	61.57	1.85	4.30
- 105 + 75	5.2	61.24	2.32	4.50
- 75 + 45	3.3	61.90	2.45	3.70
- 45 + 30	17.1	58.02	4.58	5.38
- 30 + 20	10.5	55.05	7.50	6.90
- 20	42.2	47.00	13.02	10.52
Bulk	100.0	52.50	7.82	7.40

### **Flocculation Experiments**

For the flocculation experiments a representative sample was ground in a planetary mill using an agate bowl and balls to produce material all passing 400 mesh ( $37 \mu\text{m}$ ) size. Flocculation experiments were carried out in a 500-mL measuring cylinder at a pulp density of 5%. The sample was conditioned in a 500-mL beaker prior to flocculation. At the end of 10 minutes the desired quantity of flocculant was added and at the same time the stirrer speed was reduced to create a low shear condition. The stirring was continued for 2 minutes following addition of flocculant. The entire suspension was then transferred to a 500-mL measuring cylinder, and the suspension was allowed to settle after the cylinder had been inverted twice. Two-thirds of the volume of material was siphoned out after 1 minute of settling. The flocs were cleaned by diluting the sample with water to the original volume, conditioning the sample for 1 minute, and then siphoning out two-thirds of the supernatant from the cylinder. The final product (flocs) and the rejects were dried, weighed, and analyzed.

Ultrasonic treatment of iron ore tailing was carried out using an Ultrasonic Bath Model 555 (made in Germany). The device operated at a frequency of 22 kHz. The power output was approximately 250 W. The slurry was treated for 10 minutes and then transferred to a 500-mL measuring cylinder. The rest of the procedure was repeated as above, i.e., without ultrasonication.

## RESULTS AND DISCUSSION

The chemical composition of the iron ore slime sample is shown in Table 1. It can be seen from these data that the sample contained a low amount of iron and a high amount of alumina and silica. Table 2 presents a particle size analysis and the chemical composition of different size fractions of the tailing. It is interesting to note that the finer fractions (e.g., below 20/30  $\mu\text{m}$ ) are preferentially enriched with alumina and silica. The results of flocculation with corn starch are shown in Fig. 1. The

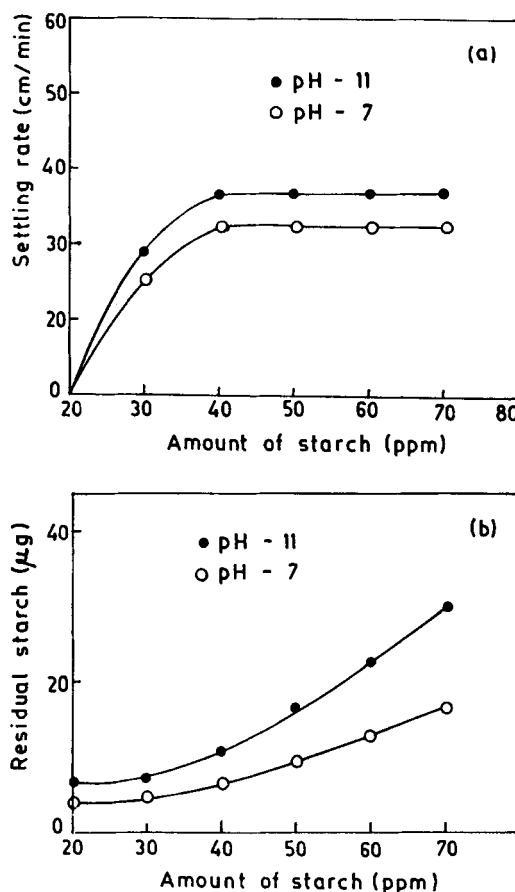


FIG. 1 Flocculation test results on iron ore fines as a function of corn starch addition: (a) settling rate, (b) residual starch concentration in solution.

settling rates increased sharply with starch addition up to 40 ppm, reaching limiting values thereafter. The settling rate at pH 11 was found to be marginally higher than at pH 7. The residual starch concentrations increased substantially beyond 40 ppm, attesting to the fact that excessive additions of starch beyond the optimum do not contribute to an increase in the settling rate.

Figure 2 shows the effect of corn starch addition on grade and recovery. It clearly reveals that at the threshold concentration 40 ppm corn starch gives the optimum grade (53%) as well as recovery (80%), but at higher concentrations the grade marginally deteriorates. This may be due to either restabilization caused by a higher concentration of polymer on the surfaces of particles or the floc size is very small due to a higher number of polymer molecules interacting on the same number of particles.

The effect of dispersant (NaHMP) is shown in Table 3. There is little improvement in the grade (59%), but the formation of flocs tends to decrease with an increase in the concentration. A very high dosage of dispersant was not favorable. It may, however, prevent adsorption of flocculant on particle surfaces. This observation corroborates other research (2).

Figure 3 shows the results of zeta potential as a function of starch concentrations at pH 7 and 11. The zeta potential remains negative throughout the range studied and a plateau region is observed. Major changes in zeta potential values occur at low starch concentrations (20–40 ppm). The zeta potential at the plateau region may correspond to adsorption density at saturation coverage. Adsorption of negatively charged polymer cannot

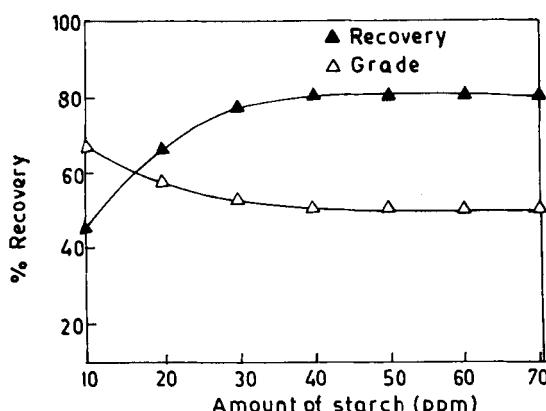


FIG. 2 Effect of corn starch on the recovery and grade of iron ore fines, pH 11.

TABLE 3  
Effect of Dispersant on Concentration (pH 11; corn starch, 40 ppm)

Concentration of dispersant (%)	Fe (%)	Recovery (%)
0.1	55.4	90
0.2	56.2	85
0.5	59.0	75
0.8	57.0	87
1.0	54.0	92

normally be expected to make a mineral particle less negative within the constant ionic strength conditions of a system.

Flocculation results as a function of ultrasonication time and the sequence of starch addition are shown in Fig. 4. It was found that the amount of starch adsorbed increased dramatically when the starch was added prior to ultrasonic treatment, which corroborates the results of Arol and Iwasaki (14). However, when starch was added following ultrasonic treatment, no significant change was observed in the adsorption of starch.

Figure 5 shows the results of selective flocculation in an ultrasonic field at pH 11. Ultrasonication for 20 seconds resulted in 58% grade with a recovery of 31%. Ultrasonication for 40 seconds further improved the

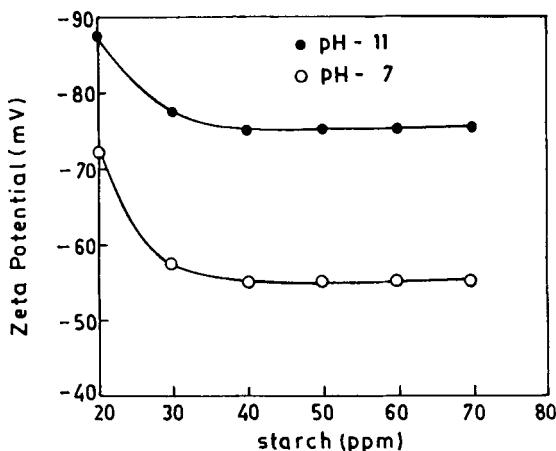


FIG. 3 Zeta potential of iron ore fines as a function of starch concentration at two different pH values.

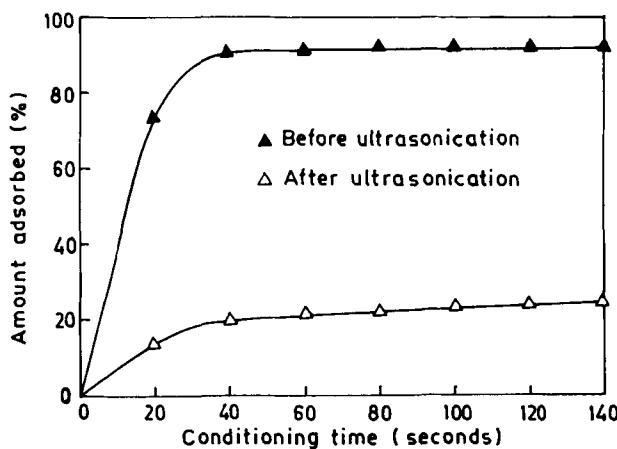


FIG. 4 Effect of ultrasonication on the adsorption of starch.

grade 60% with a recovery of 41%. Similarly for 80 and 160 seconds the grade was increased to 61 and 65% and the recovery to 51 and 91%, respectively. After an additional 200 seconds the grade had decreased to 60%. Thus it is concluded that at the optimum time of 160 seconds a grade of 65% iron with a recovery of 91% can be achieved. The final product was also analyzed for alumina and silica, and it was shown that the propor-

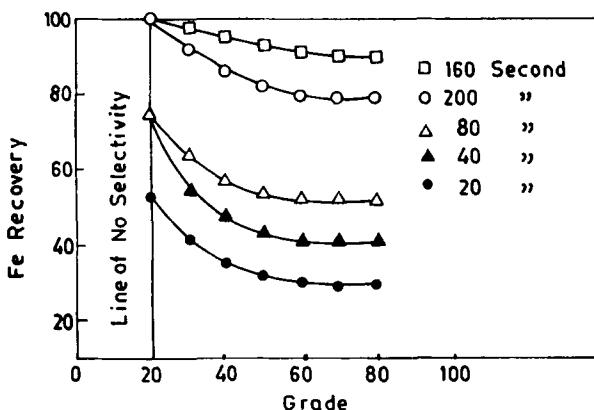


FIG. 5 Selective flocculation of iron ore fines in an ultrasonic field.

tions of alumina and silica were also reduced significantly (1.6% alumina and 1.2% silica).

## CONCLUSIONS

The investigation carried out to improve flocculation selectivity for the separation of iron values from iron ore tailings by ultrasonic pretreatment revealed the following facts.

1. A final product with 65% grade may be obtained by recovering 91% of the iron values present in the ore after imposing ultrasound for 160 seconds and cleaning three times. Alumina and silica content were reduced to 1.6 and 1.2%, respectively.
2. It is interesting to note that the imposition of ultrasound after corn starch addition had a beneficial effect but that ultrasound before corn starch addition did not. A possible explanation for this is as follows: The presence of clay materials is invariably problematic in selective flocculation. It is presumed that by applying ultrasound, clay materials are temporarily removed from the iron ore particles and starch is preferably adsorbed. Ultrasonication before starch addition was not found to be suitable because clay materials are further deposited on the iron ore surface and prevent starch adsorption.

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