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Coagulation and Flocculation Study of Iron Ore Fines

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ABSTRACT

A comparative study of the flocculation and coagulation response of an iron ore fines suspension has been carried out, and the extent of flocculation has been assessed by measurement of electrophoretic mobility, supernatant clarity, and settling rate. Of the several commercial flocculants and polyelectrolyte studied, the combination of medium molecular weight anionic flocculants Magnafloc 1011 and Rishfloc 258 (1:1), and coagulant aluminum nitrate was most effective in terms of enhancing settling rate and supernatant clarity.

Key Words. Flocculant; Coagulant; Iron ore; Vacuum filtration

INTRODUCTION

Polymeric flocculants are widely used in mineral and mining industries to enhance particle sedimentation in the application of thickening processes to slurry dewatering. Flocculation is a process of agglomeration of fine particles suspended in liquid and most commonly used as an aid for solid–liquid separations. For systems difficult to process, a coagulant is also sometimes used in conjunction with the flocculant. The use of coagulants and flocculants for thickening slurries and aiding filtration is therefore practiced where a very specific quality of effluent is present. In addition, they are used in acid

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drainage from mines, reuse stream management, and control of runoff from settling ponds. In polymeric flocculation, floc properties are of paramount importance. The floc properties required for different methods of separation are unique (1). For example, sedimentation requires dense and large flocs with regularity in shape (perfectly spherical); filtration requires porous, strong, dense, large flocs; and in flotation low density, strong flocs with a narrow size distribution are desired (2). The characteristics of the flocs formed depend on several factors such as surface chemistry, size and size distribution, shape, density, etc., of the solid component; viscosity, dielectric constant, etc., of the suspension; and the chemical nature of the backbone and side chains, molecular weight, molecular weight distribution, charge, and charge density of the flocculant used (3, 4). However, the mechanism of flocculation is assumed to be one of the major factors responsible for floc structure. Optimization of solid/liquid separation processes based on sedimentation and flocculated suspensions requires careful control of floc size and structure (3).

The adsorption of high molecular weight polymer on a particle's surface may be due to the electrostatic, hydrogen/van der Waals, and covalent bonding (5). Some of the mechanisms responsible for floc formation are 1) bridging, 2) charge neutralization, 3) polymer complex formation or network flocculation, and 4) depletion flocculation. The bridging mechanism is related to adsorption of very high molecular weight polymers which form links between the particles when the electrostatic charge is overcome (6). Flocs formed by the bridging mechanism are usually stronger than those formed by charge neutralization or the electrostatic patch mechanism.

Washing of iron are is a mandatory step in 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; classified fines (-10 mm $+$ 150 μm), which are fed to the sintering plant with or without beneficiation; and the fines and slimes (-100 mesh), which are presently discarded as waste. The iron ore wash from washing plants mainly consists of iron in suspension contaminated with alumina and quartz sand. The alumina-containing phases in the slimes are characterized as 2-μm grains of kaolinite, illite, and montmorillonite (7). Slurries containing appreciable quantities of such materials in a dispersed state are difficult to sediment or filter. The suspension is flocculated in thickeners. This is done in order to produce a low turbidity overflow which can be recycled to the washing plant. After the thickened pulp is dewatered, it can be used as sinter feed.

Flocculation of fine particles also leads to a substantial improvement in filtration rate, producing higher cake yields and lower cake moisture, and this allow for greater throughputs and better performance by the existing equipment. Current practice favors the use of synthetic polymers in comparison to inorganic electrolytes because they are more effective than the latter and only a small amount of synthetic polymers is required in comparison to inorganic electrolytes, which makes their use cheaper and cost-effective overall.

This paper is concerned with establishing the type of flocculant required to obtain dense and large flocs of regular shape and high floc strength needed for subsequent dewatering studies under moderate pressure, and comparing the performance of flocculants in the treatment of iron ore fines. The overall objective is to obtain baseline laboratory-scale data for the combination of flocculant and coagulant, because flocculant alone is unable to produce the required level of supernatant clarity.

EXPERIMENTAL

Materials

Substrate

The iron ore sludge used in this study was obtained from one of the iron ore washing plants in Bihar, India. The characteristics of the sample are summarized in Table 1.

Flocculant

Polyacrylamide-based flocculants of varying ionic character and molecular weight were obtained from Allied Colloid, UK, and Rishflocs, Mumbai. Some

TABLE 1
Material Characteristics

Properties	
Analysis:	
Al_2O_3	7.3%
SiO_2	3.7%
Fe	58.9%
Particle size:	
Less than 45 μm	60.0%
Less than 30 μm	38.0%
Less than 20 μm	25.0%
Less than 10 μm	20.0%
pH of slurry	5.2
pH of the zero point charge	5.2
Specific surface area of the particles	1.4 m^2/cm^3

TABLE 2
Characteristics of Flocculants

Flocculants	Ionic charge	Molecular weight[a]	Nature	Solubility
Magnafloc 592	Strongly cationic	2.5–5.0	Powder	Water
Magnafloc 1011	Anionic	5.0	"	"
Magnafloc 351	Nonionic	2.5–5.0	"	"
Rishfloc 440MV	Strongly anionic	"	Crystalline	"
Rishfloc 258	Weakly anionic	"	"	"
Rishfloc 1226	Nonionic	"	"	"

[a] Molecular weight range is based on a scale of 1–5, where 1 represents molecular weights of 0.5–1.0 million and 5 represents molecular weights of 15–20 million.

of the important properties of the flocculants used are shown in Table 2. All reagents were water soluble and were prepared as per the method suggested by the manufacturer.

METHODS

Batch Settling Test

The iron ore fines slurry and an appropriate amount of water (6% solid by weight) were stirred at 600 rpm in a 1000-mL beaker for proper dispersion. The flocculant (0.01% solution in distilled water) was added dropwise to the well-dispersed slurry while the stirring was continued. The contents were carefully transferred into a 1000-mL graduated cylinder. The cylinder was stoppered, inverted five times for proper mixing, and allowed to settle. A clear interface, called the "mudline height," with supernatant above and a dense suspension below, developed. The mudline height was recorded at regular time intervals as it descended. The settling rate was estimated from the straight line portion of a plot of the mudline height against the settling time.

Electrophoretic Mobility

Electrophoretic mobility was measured with a Rank Brothers Mark-II microelectrophoresis apparatus utilizing the two electrode mode and a flat quartz cell. A detailed description of this apparatus and the procedure for test suspensions have appeared elsewhere (8).

Adsorption Measurement

The amount of flocculant adsorbed on the ore surface was determined by measuring the residual flocculant concentration in the supernatant liquid by complexing with a cationic dye and measuring the optical density using a UV/Vis spectrophotometer.

RESULTS AND DISCUSSION

Surface Charge

The sample shows a zero point charge at pH 5.2. Normally the ZPC of pure hematite in distilled water is 4.2 (9). Figure 1 shows the result of zeta potential measurements in the presence of flocculants Magnafloc 1011 and Rishfloc 258 (1:1) at an optimum dosage of 43 g/t. It was observed that the presence of polymer decreases the electrophoretic mobility and shifts the ZPC toward lower pH values. This may be due to electrostatic interaction of the anionic polymer with iron ore in the acidic pH.

Adsorption Studies

The results of adsorption studies at different pH values are shown in Fig. 2. The shape of the adsorption isotherms shows an increasing content of adsorbed polymers up to a plateau with increasing initial concentrations of polymer. In general, it was found that decreasing the pH value increases the adsorption plateau value. This result supports the assumption that electrostatic forces influence the adsorption process in lower pH ranges. In the acidic pH range there is a predominant presence of positively charged surface sites which interact electrostatically with negatively charged anionic flocculants,

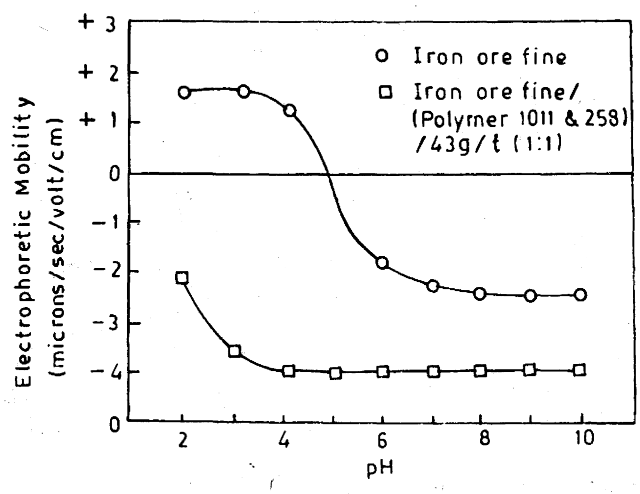

FIG. 1 Effect of pH and flocculant addition on the electrophoretic mobility of iron ore fines.

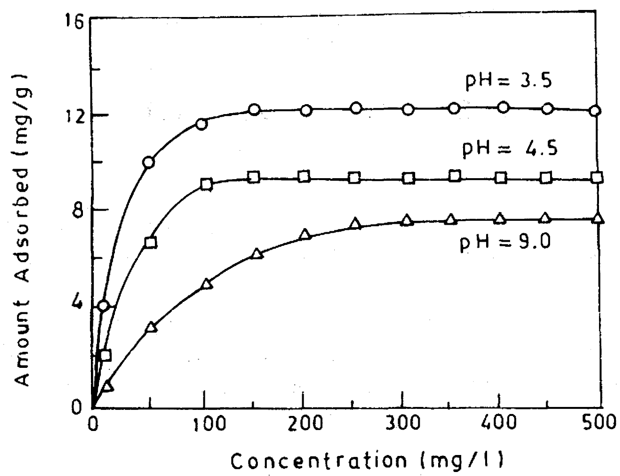

FIG. 2 Adsorption isotherms of polymeric flocculants Magnafloc 1011 and Rishfloc 258 on iron ore fines as a function of the equilibrium concentration and the pH value.

resulting in high adsorption. However, in the alkaline pH range, negatively charged surface sites repel the negatively charged flocculants, so there is comparatively a lower adsorption of flocculants, and the adsorption of flocculants is not due to electrostatic forces but because of other forces which preferably become the effective adsorption mechanism in an alkaline medium.

Settling Behavior

Figure 3 shows typical settling results at different pH values, coagulant concentrations, and solid contents. The flocs initially settle comparatively faster and gradually level off with time. This is due to the force of gravity acting on individual flocs which settle freely when the flocs are at large distances from each other. As settling proceeds, the interfloc distances are reduced and the difficulty of flocs to settle freely increases. At a high solids concentration free settling is completely hindered as a result of increased pulp density, which is a function of solid concentration and the collision of flocs. Better settling was obtained between 5 and 8% solids. Hence subsequent experiment was carried out only at a pulp density of 6% w/v.

Figure 4 shows the interface level as a function of settling time for all six flocculants and the unflocculated slurry at pH 5.2 (4). In general, two flocculants (Magnafloc 1011 and Rishfloc 258), which are anionic in nature, showed the best settling rate (2.50 cm/s) at a dose of 0.025 and 0.018 kg/t, respectively. However, the floc strengths were quite poor and therefore it was de-

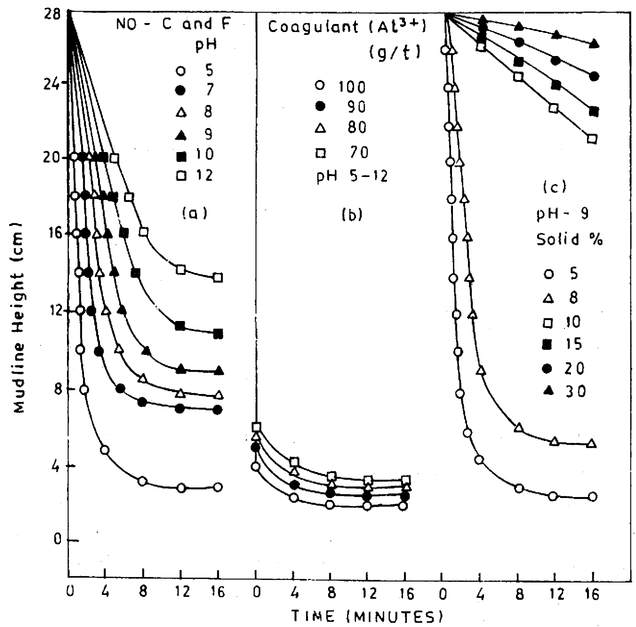

FIG. 3 Mudline height of iron ore fines suspension (a) at different pH values, (b) with $Al_2(NO_3)_3{\cdot}9H_2O$, and (c) at different solid concentrations (C = coagulant, F = flocculant).

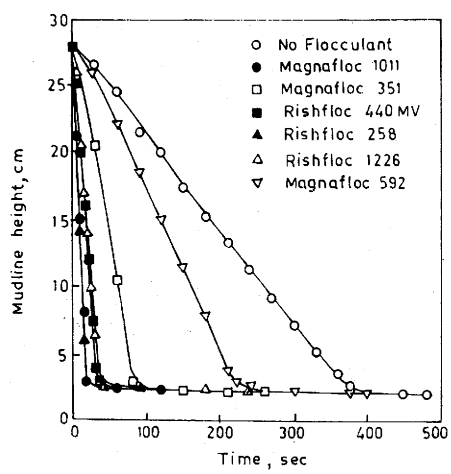

FIG. 4 Settling characteristics with different polymeric flocculants (12 g/t), pH 5.2 (4).

cided to see if a combination of the two flocculants would improve the floc strength. Among all the combinations, Mangnafoc 1011 and Rishfloc 258 were found to be best not only in terms of floc strength but also in the settling rate which increased significantly (5 cm/s) at a dosage of 0.043 kg/t (1:1). Most synergism effects in flocculation are derived from the combined use of polymers with different charge, but in the present study the synergistic effect is due to a combination of anionic flocculants.

While the settling rate with a single flocculant was satisfactory, the floc strength was very poor for filtration dewatering. Therefore, a combination of the two best flocculants was tried in an effort to improve the floc strength. It may be that polymer adsorption on surfaces of like charge occurs when the repulsion due to electrostatic forces is not strong enough to prevent polymer adsorption due to the following mechanism of interaction (10): 1) van der Waals interaction, 2) hydrogen bonding, 3) local electrostatic attraction, and 4) development of linkage between a similarly charged polymer of the particle surface with a divalent or trivalent ion of the opposite sign. Flocs formed by a bridging mechanism are usually stronger than those formed by charge neutralization or the electrostatic patch mechanism. This laboratory has undertaken a systematic investigation in this area, and further experimental results will be reported in the near future.

Figure 5 illustrates the response of the hindered settling of an iron ore fines suspension to flocculant addition at pH 5.2 (4). The curves exhibit the characteristic optimum value of flocculant dosage beyond which further addition

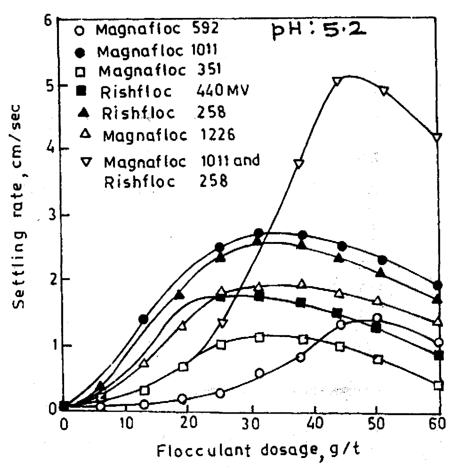

FIG. 5 Effect of flocculant dosage on settling rate (4).

TABLE 3
Summary of Flocculants Combined Coagulant Results

Treatment	Flocculant/coagulant (g/t)	Turbidity (NTU)	Color	Iron (mg/L)
Supernatant after natural settling	0	350	Red-grey	120
Supernatant after coagulant treatment	100	300	Red-grey	100
Supernatant after Magnafloc 1011	18	250	Grey	75
Supernatant after Rishfloc 258	25	240	Grey	82
Supernatant after Magnafloc 1011 and Rishfloc 258 treatment	18:25	130	Very light grey	45
Supernatant after Magnafloc 1011 and Rishfloc 258 and coagulant treatment	43 (1:1)/100	15	Colorless	10

leads to an adverse effect on the settling rate. The optimum flocculant dosage is the lowest dose at which the filtrate is clean and colorless, and the settling rate is maximum. In general, it was observed that the settling rate decreases at higher dosages of flocculants. This may be due to either restabilization caused by a higher concentration of flocculant on the particle surface or the floc size being very small due to a larger number of polymer molecules interacting with the same number of particles (11). After obtaining a better settling rate and sufficient floc strength, the problem of poor clarity of the supernatant liquid (turbidity 250 NTU) remains. Therefore, we tested a combination of the best flocculants with a trivalent coagulant [$Al_2(NO_3)_3 \cdot 9H_2O$]. The summary of test results with combinations of flocculants and coagulant are shown in Table 3. A significant improvement in supernatant clarity was observed (15 NTU).

Filtration Characteristics

Figure 6 shows the effect of flocculant dosage on the settled volume of slurry and filter cake moisture. At low and very high dosages of flocculant, a substantial reduction in settled volume is obtained. However, this is detrimental with respect to cake moisture content as it is evident from the figure. There exists flocculant dosage at which the lowest cake moisture as well as the highest settled volume are obtained.

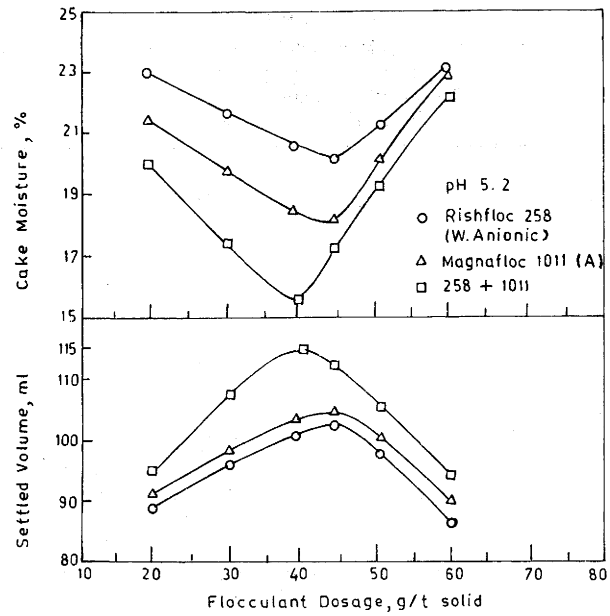

FIG. 6 Effect of flocculant on slurry settled volume and residual cake moisture.

SOME GENERAL OBSERVATIONS

The ranking of flocculants used in this investigation, as measured by their effectiveness in enhancing settling rate and resistance to filtration, indicated that Magnafloc 1011 and Rishfloc 258 were equally effective, but their combination gave the best separation. Good settling is associated with larger flocs, which necessarily have more capillaries and hence a higher water-holding capacity. The combination of above two flocculants along with an electrolyte was observed to be best for both supernatant clarity and settling rate. Ideally, the metal ion should neutralize the surface charge on the particles or compress the diffuse double layer around the particles, creating conditions under which the particles should show reduced mobility and thus flocculate more readily (12). Floc strength is of paramount importance in subsequent filtration and dewatering experiments. The settling data are consistent with the current views on polymeric adsorption, according to which the initial increase in sedimentation rate is attributed to a mechanism in which bridges are formed by the simultaneous adsorption of polymer on groups of primary particles. The decrease in settling rate observed beyond the optimum flocculants dosage is generally thought to arise from restabilization phenomena involving steric repulsion between particles carrying adsorbed polymer molecules (13).

CONCLUSIONS

1. The settling rate of iron ore fines can be greatly improved by using a combination of anionic polymers.
2. Flocs formed by a combination of two anionic polymeric flocculants of high molecular weight are found to be suitable for vacuum filtration because the floc strength is improved.
3. The combination of flocculants and coagulant significantly improves supernatant clarity in comparison to individual agents.
4. A direct correlation exists between ZPC and moisture reduction in filter cakes.

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