Surface Chemical and Settling Studies on Hematite, Quartz and Kaolinite in Presence of Organic Reagents

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Abstract

The industrial practice of beneficiation of iron ores produces substantial amount of slimes which causes loss of iron values and environmental pollution. Slimes consists of extremely fine grained iron bearing minerals, impurities and poses problem in processing by conventional beneficiation techniques. The present study aims to develop flocculation technique for selective separation of iron bearing mineral from slimes. Initial experiments were directed towards understanding surface chemical properties of constituent minerals viz. hematite, quartz and kaolinite with or without organic reagents such as tannic acid, starch and polyacrylamide-co-acrylic acid (m.w.-150 lakh). In this paper, the results of flocculation-dispersion experiments on individual samples of hematite, quartz and kaolinite are discussed as a function of different process parameters such as pH, flocculation time and dosages of reagents. Based on the studies, conditions were established for selective separation of hematite from quartz and kaolinite. The results of flocculation-dispersion of individual minerals were applied to ternary synthetic mineral system.

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1. Introduction

India is endowed with huge deposits of rich iron ores. However, there is a rapid decline of the high grade iron ore reserves due to increase in domestic production and also due to the export of the iron ores [1]. In India, except a few, not many iron ore companies follow the beneficiation of the iron ore, However, simple operations such as sizing and washing is widely practiced. The industrial practice of sizing, washing and beneficiation of iron ores produces substantial amount of slimes which causes loss of iron values and environmental pollution [2-3]. Slimes consists of extremely fine grained iron bearing minerals, impurities and poses problem in processing by conventional beneficiation techniques. Although, conventional processes like magnetic separation and flotation are used to recover the ultra fine iron values but are not fully effective to recover the iron bearing mineral particles due to size effect [4-8]. The present study aims to develop flocculation technique for selective separation of iron bearing mineral from slimes.

In this paper, surface chemical studies on the individual minerals viz. hematite, quartz and kaolinite were investigated. A detailed study has been made to understand the settling behaviour of different reagents on three different individual minerals. Experiments have been carried out at different reagent dosages, time, pH, and individual hematite ore, quartz and kaolinite to optimise the process parameters of separation.

2. Materials and Methods

The brief description of the different materials taken and the methods adopted is summarised in this section.

2.1 Materials

Three different individual pure mineral samples of hematite, quartz and kaolinite (USA Wards Corporation) were collected for the present study. The samples were ground to less than 37 micron and were used for flocculation – dispersion studies. The iso-electric point of the material under study was measured using the Delsa Nano C particle analyzer of M/s Beckman Coulter. The particle size analysis was carried out using Malvern particle size analyser model Master sizer 2000 with wet dispersion system Hydro 2000 MU.

AR grade organic reagent of poly acrylamide-co-acrylic acid (PACAA) of molecular weight 150 lakh from Aldrich, tannic acid powder pure from Merck and starch from Qualigen were used in this study.

2.2 Methods

Freshly prepared solution of PACAA, tannic acid and starch were used in flocculation-dispersion experiments. The individual mineral samples were taken and 1 % by weight of slurry was prepared. The experiments were carried out by addition of different dosage of reagents at different time period and at different pH conditions. Sample was taken in a 100 ml stoppered measuring cylinder, all the parameters were maintained and settling test was performed. After the settling time gets over, 10 ml of the supernatant was taken out in a turbidity cell, through a pipette and immediately the turbidity of the sample was determined using a Systronics make Digital Nephelo Turbidity Meter, model 132. Immediately the supernatant suspension was decanted. The settled material was dried and its weight was taken. The flocculation-dispersion experiments were carried out in absence and presence of various reagents as a function of time, pH, and reagent concentration. The detailed parameters are grouped in Table-1.

Table 1: Experimental conditions for flocculation-dispersion tests carried out on individual Samples of hematite, quartz and kaolinite

Parameters	PACAA	STARCH	TANNIC ACID
Reagent dosage (ppm)	0,1,10,25,50		
pH (±0.1)	5 9 11		
Time (min.)	1, 2 ,5,10,30,60,120		

3. Results and Discussions

The results with respect to the zeta-potential and flocculation-dispersion tests carried out are described in this section.

3.1 Size Distribution

Particle size distribution of the individual samples is presented in Table-2. It is seen from the size measurement that the samples are extremely fine in size. It is observed from the size distribution result that hematite is finer than kaolinite and quartz. Quartz is the coarsest among the three minerals with 90% of the particles are passing 25.5µm. The least size recorded for hematite, 10% of the particles are of size 0.4µm.

Table 2: Size distribution of the individual minerals

Mineral	D_{10}, %	D_{50}, %	D_{90}, %
Hematite, µm	0.4	3.5	19.0
Quartz, µm	1.6	7.0	25.5
Kaolinite, µm	1.0	6.6	31.2

3.2 Zeta Potential Measurement

The iso-electric point of hematite was determined to be at pH 5.4 and for quartz and kaolinite below pH 2 (Table-3). On addition of 50 ppm PACAA, the zeta potential values were lowered in all the pH points and the iso-electric point values have shifted to pH value closer to 2, which indicates that polyacrylamide-co-acrylic acid interacts chemically with hematitic surface showing a higher flocculating behaviour in comparision to other reagents.

Table 3: Iso-electric points of hematite, quartz and kaolinite samples

Minerals	Iso-electric point, iep (pH)
Hematite	5.4
Quartz	< 2
Kaolinite	< 2

Starch also results in good settling behavior. However, tannic acid acts as dispersant in both acidic and alkaline i.e. below and above the i.e.p. Starch shows a higher dispersing behaviour with quartz at higher pH.Starch and tannic acid shows good dispersion behaviour with kaolinite with increasing pH, while polyacrylamide-co-acrylic acid shows good flocculation with kaolinite.

3.3 Flocculation-dispersion Studies

The flocculation-dispersion test results are presented and discussed in this section for all the three different individual pure mineral samples.

3.3.1 Effect of time on settling behaviour

The results of settling study on the three individual minerals as a function of time are given in Fig 1. The supernatant turbidity as a function of time is also studied and the results are given in Fig 1b. When the time period increases the turbidity of all the minerals decreases as expected. The lowest turbidity for hematite recorded as 294 NTU, at a time duration of 120 minutes (Fig 1a). Amongst all the minerals, the supernatant turbidity for hematite is the least. However, the supernatant turbidity was higher for kaolinite and quartz before 30 minutes of settling time when compared to hematite. In case of quartz, turbidity was higher in comparison to the other two minerals. At the same time, as the time of settling increases the turbidity of kaolinite decreases steeply. The lowest turbidity of kaolinite recorded as 164 NTU.

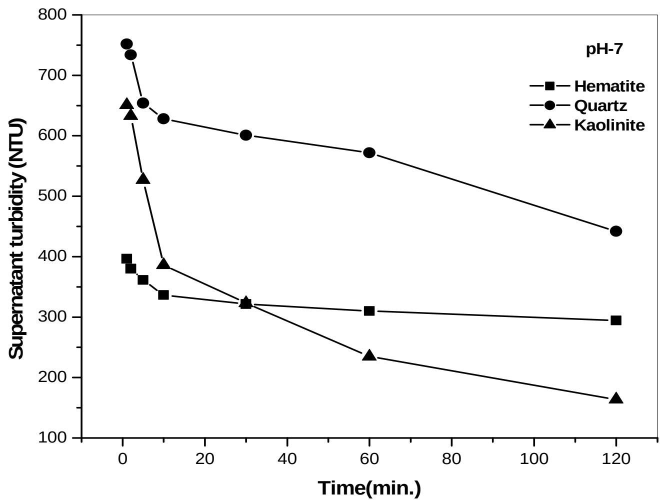

Fig 1a : Turbidity of three individual minerals as a function of time

The settling characteristics of the individual mineral samples are given in Fig 1b. In all the cases, as the time of settling increases, the % settled increases upto 10 minutes and beyond, a steady increase was observed upto 2 hours. The settled amount was more than 70% in less than 10 minutes for hematite which is comparatively on the lesser side with respect to settling of quartz and kaolinite.

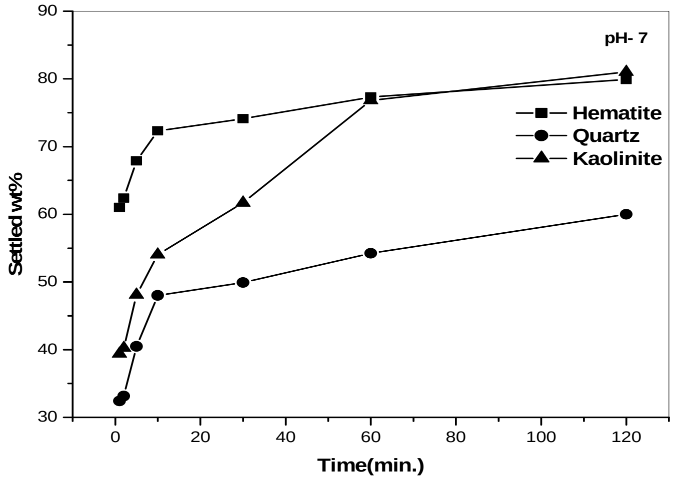

Fig 1b: Settled wt% of three individual minerals as a function of time

3.3.2 Effect of pH

To know the effect of pH, further study has been made and the results are shown in Fig-2. In these experiments, the settling time was kept at 10 minutes for all pH values studied. Settling tests were carried out at three different pH values and the results are given in Figs 2(a&b).

The turbidity of Kaolinite shows a fast increasing trend with increase in pH from 5 to 11. However, in other two cases also, there is a steady increase in supernatent turbidity with increase in pH from 5 to 11. Hematite shows minimum turbidity while quartz shows the highest turbidity at pH 11.

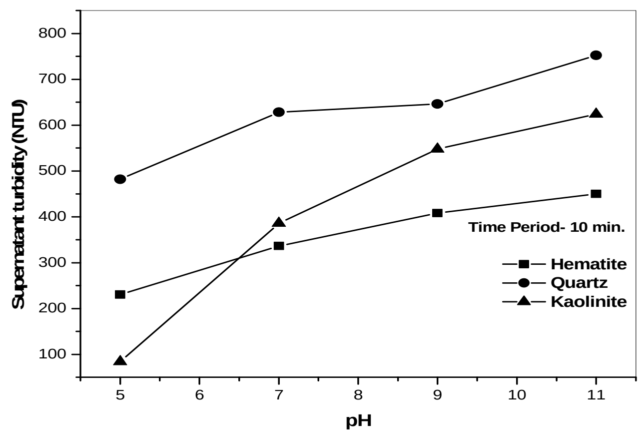

Fig 2a Effect of pH on the turbidity of three individual minerals at 10 min time

Quartz gets dispersed more and more as the pH moves to alkaline side, it shows a decreasing trend in its % material settled (Fig 2b). The supernatent turbidity value is nicely corroborating the % settled in all the three cases. The settling of hematite is maximum at pH 9. 8 i.e. % of hematite by weight gets settled within 10 minutes at pH 9.

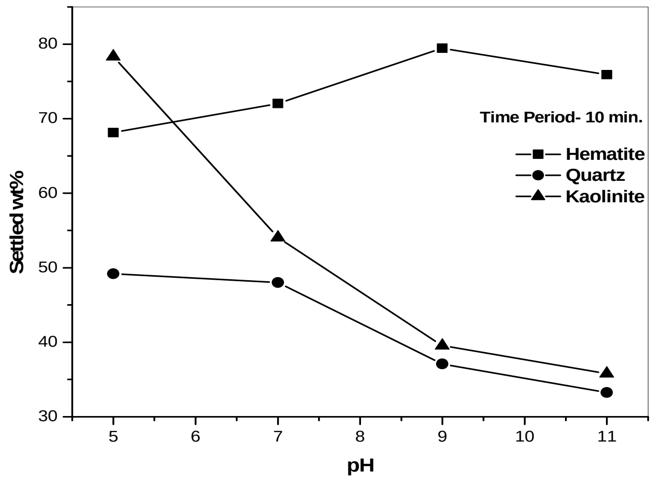

Fig 2b: Effect of pH on the settled wt% of three individual minerals at 10 min time

3.3.3 Effect of various reagents on settling behaviour

To know the effect of change in reagent concentration on all the three individual minerals, further study has been made with different reagents at constant time of 10 minutes with pH 9. The results are given in Fig 3.

Above Fig 3a clearly shows that polyacrylamide-co-acrylic acid works as a good flocculant for hematite and kaolinite, At the same time it is also observed that with an increase in concentration it acts as a dispersant for quartz, but its dispersing power is less.

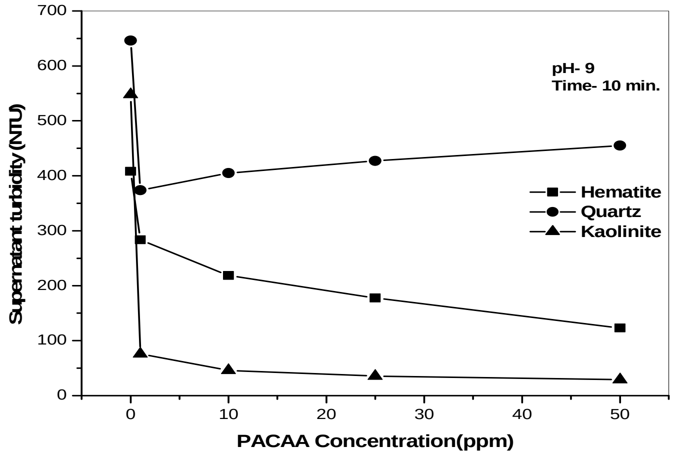

Fig 3a: Turbidity of three individual minerals with and without polyacrylamide-co-acrylic acid

Tannic acid works as a good dispersant for quartz and kaolinite, at a concentration of 1 ppm. At the same time it is observed that with an increase in concentration its dispersing power is decreasing (Fig 3b). In presence of starch, hematite shows low dispersion whereas kaolinite and quartz show high disppersion (Fig 3c).

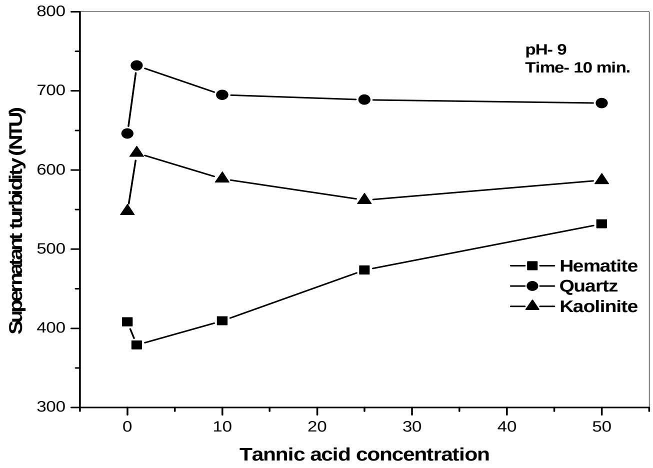

Fig 3b: Turbidity of three different individual minerals with and withoutTannic acid

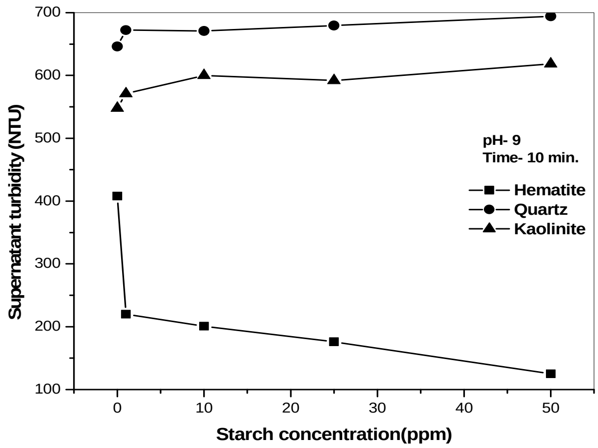

Fig 3c: Turbidity of three different individual minerals with and without starch

From the result of the threee different reagents viz. Tannic acid, starch and PACAA with hematite, quartz and kaolinite, 50 ppm of starch gave the best result wherein use of starch caused settling of hematite particles while quartz and kaolinite showed good dispersion at pH 9 with settling time of 10 minutes.

The best possible condition comes out from the present study has been applied to a ternary mixture of hematite, quartz and kaolinite taken in the ratio 2:1:1. Strach concentration was taken as 50 ppm. The result of the experiment is given below in the form of histogram. At pH-9±0.1, with the addition of starch, at a concentration of 50 ppm, 71 % of the mixture settled and the Fe content analyzed 44.2% (Fig 4).

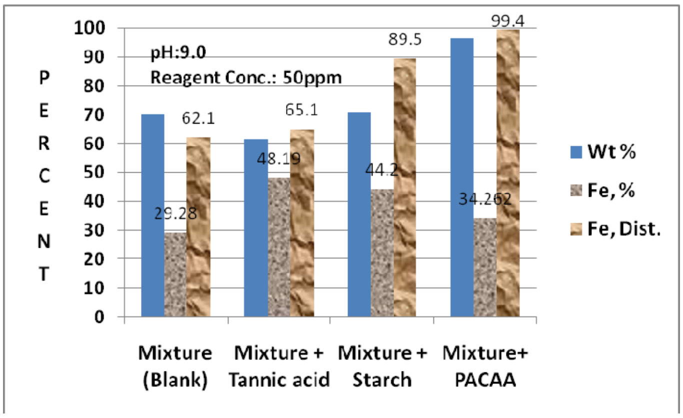

Fig 4: Settled wt% with assay and distribution of Fe of mixture of hematite, quartz and kaolinite

4. Conclusions

- Flocculation-dispersion may be applied to fine iron ore particles, wherein the fine iron ore minerals can be aggregated and flocculated using starch and other polymeric reagents.
- There is a good correlation between zeta potential and supernatant turbidity value.
- Selective separation of fine grained hematite particles could be possible from the ternary mixture of hematite-quartz-kaolinite by adopting flocculation method with suitable reagent and pH condition.
- By selective flocculation, a yield of 71 % iron bearing minerals with 44.2% Fe achieved at pH ~ 9 using starch as flocculating agent.

5. Acknowledgements

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