Selective Flocculation of Banded Hematite Quartzite (BHQ) Ores

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Abstract: The recovery of fine hematite particles from banded hematite quartzite (BHQ) ore with potato starch has been investigated using selective flocculation. Microscopic features, coupled with x-ray diffraction (XRD), and Fourier transforms infrared (FTIR) spectroscopic data provide evidences for adsorption of starch molecules on hematite surface. The starch molecules show good selectivity for hematite particles in the pH range of 8.5-9.5 and in the reagent concentration range of 20-40 mg/l. The Scanning electron microscope (SEM) studies show that flocs of hematite particles are larger and appear to be denser than that of quartz. Initial results with 1:1 hematite-quartz mixture indicated that it is possible to achieve an iron concentrate of 64.5% Fe with 92.0% of iron recovery from a feed of 34% Fe. However under optical conditions of pH and reagent concentration, the BHQ ore could be upgraded from an initial grade of 38.9% Fe to 57.2% Fe with 71% recovery. The overall results indicate that separation of very fine grained hematite present in the BHQ iron ore is feasible by selective flocculation.

Keywords: BHQ ore, Selective flocculation, Potato starch, XRD, FTIR, SEM.

INTRODUCTION

Selective flocculation is a useful technique commonly used to enrich ultra fine mineral particles that respond poorly to conventional beneficiation techniques. The beneficiation techniques, such as gravity separation, magnetic separation, or flotation, are generally ineffective in treating the ultra-fine particles. Hence in practice these particles are usually discarded as tailings prior to concentration. For example, in India, 10-12 million tons of iron ore slimes are being generated every year during the concentration of hematite ore and discarded without any further utility. In order to recover values from ultra-fines, several industries are employing selective flocculation technique to treat fines and slimes generated during mining and processing of the ore.

Similarly, the process to recover iron values from the banded iron ore formation (BIF)—banded hematite quartzite (BHQ), banded hematite jasper (BHJ) and banded magnetite quartzite (BMQ)—are yet to be explored due to the fine nature of the ore which is difficult to treat by straight-forward selective flocculation means. These low grade BIF ores contain 28-40% Fe and 40-55% SiO_2. The minerals are liberated at extremely fine sizes and in addition, these ores typically consist of bands of iron-bearing hematite or magnetite intergrowth with quartz and for this reason, upon breakage, the nature of particle surface appears quite complex. These aspects of particle characteristic tend to have a strong bearing on any technique that relies on surface properties to induce separation. It is therefore imperative to develop a recipe to adopt selective flocculation technique to treat low grade ores and waste slimes of banded nature. The possible beneficiation techniques that have been tried to recover iron values from these banded ores is by grinding to liberation size followed by high intensity magnetic separation and flotation [1]. However, adaptation of selective flocculation technique has been a challenge.

A number of successful selective flocculation studies have been achieved at laboratory as well as pilot-plant scale using various mineral/ore slimes such as bauxite, coal, phosphate, chromite, hematite, magnetite, etc., [2-12]. The success of such technique relies heavily on the chemistry of the process. A review of the literature shows that the use of various starch molecules from potato, corn, tapioca, cassava, etc., as flocculants have worked well for iron-bearing particles compared to other synthetic flocculants. For example, selective flocculation study of hematite and hematite-silica mixture clearly indicates the superior selectivity of starch in comparison to polyacrylic acid (PAA) and polyacrylamide [13, 14]. Selective flocculation studies employing starch as the flocculant to treat naturally occurring iron ore slimes (hematite) of India have shown that a concentrate containing 65% Fe, 1.8% Al_2O_3 and 1.4% SiO_2 could be achieved from a plant tailing containing 52.5% Fe, 7.4% Al_2O_3 and 7.8% SiO_2 [15]. Some fundamental studies on starch have indicated that native starch is a polysaccharide mainly consisting of two different types of glucose polymers: amylopectin and amylose. The amylopectine component of starch takes part in flocculation but the amyloses are unable to flocculate any mineral. Several scientific studies such as thermogravimetry, electrophoresis and FTIR spectroscopy have indicated that adsorption of starch onto hematite surface is due to the availability of higher concentration of hydroxylated metal adsorption sites and that the flocculation occurs by the classical bridging mechanism and formation of a surface complex [16, 17].

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Use of starch is quite common for flocculation purpose and several applications of starch can be found in areas like flotation, adhesives, drug deliveries, and selective flocculation [18-22]. Given the structure of the starch we believe it is an excellent surface modulator which is particularly suitable to act on the surface of BIF ore which in itself is structurally complex. In this paper, we report a proven application of causticised potato starch as a selective flocculant for ultrafine hematite particles present in banded hematite quartzite (BHQ).

MATERIALS AND METHODS

Sample Preparation

The BHQ ore, pure quartz, and pure hematite were collected from different mines of Odisha, India. The chemical compositions of mineral samples under study are shown in Table **1**. The hematite sample contains 97.2% Fe_2O_3 with very low amount of impurities. The quartz sample is 99.4% pure. The BHQ sample contains 55.3% Fe_2O_3, 42.5% SiO_2, and 1.0% Al_2O_3. It is observed that silica is the major impurity present in the BHQ sample. The percentages of other impurities are comparatively small. For flocculation study, the as received BHQ ore was crushed in a laboratory jaw crusher followed by roll crusher to reduce the particle size. The crushed ore was further reduced to 20 μm size in a laboratory ball mill (300x300 mm) with required weight of balls. The lumpy hematite or quartz samples were also crushed, hammered, and carefully ground in a ball mill and wet screened to below 20 μm. The ground quartz particles were further leached with dilute HCl to remove any contaminated iron particles in it. It was washed several times with distilled water and finally dried at 105^0C. A laser particle size analyzer named CILAS-1064 was used for the measurement of particle size. The results are presented in Fig. (**1**) for all the three different types of samples. These results indicate that the average particle size of BHQ, quartz, and hematite are 17.16, 17.56, and 3.22 μm respectively.

Table 1. Chemical Analysis of BHQ, Hematite and Quartz Samples

Constituents %	BHQ	Hematite	Quartz
Fe (T)	38.7	68.0	Traces
Fe_2O_3	55.3	97.2	0.005
SiO_2	42.5	0.3	99.4
Al_2O_3	1.0	0.5	0.01
CaO	0.02	0.02	-
MgO	0.72	0.05	-
MnO	0.032	0.08	-
Na_2O	0.06	Traces	-
LOI	0.54	0.5	0.1

Characterization Studies

The microscopic study of the different size fractions of BHQ sample was carried out to identify the different minerals associated with the ore. The samples were examined by FTIR spectra (Shimadzu FTIR, IR Prestige-21) using KBr as the reference to understand the interaction between iron and starch molecules. The structural characterization of the samples was carried out using a Philips X-ray diffractometer (PANalytical, X'pert) which was operated with Cu-Kα radiation at 40 kV and 30 mA. SEM studies were carried out in a Hitachi 3400N equipment to analyze the coagulation pattern between hematite particles in the presence of starch.

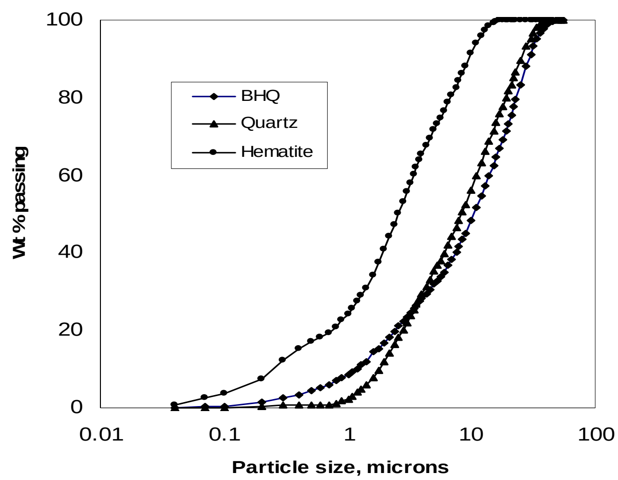

Fig. (1). Particle size analysis of BHQ, hematite and quartz samples.

Flocculation Procedure

The laboratory grade potato starch was used as the flocculant. A 1.0% starch solution was prepared by dissolving 1.0 gm of starch and 0.5 gm of sodium hydroxide to a volume of 100 ml of water. The resultant solution was heated to 84^0C and then rapidly cooled to room temperature using ice. The heating and cooling of the solution was carried out within five minutes. The flocculant was prepared daily before the start of the experiments. Flocculation experiments with BHQ, hematite, quartz and hematite-quartz mixtures were performed in a stirred beaker using the conventional jar and cylinder test. All the flocculation experiments were carried out at a pulp density of 5% solids by weight. The stirring speed was kept at 600 rpm. After adjusting the pH, starch was added very slowly to the slurry under continuous mixing at low shear condition which was ensured by reducing the speed of the stirrer. The stirring was then continued for another two minutes and the resultant suspension was transferred to a measuring cylinder. The cylinder was inverted 3 times and the flocs formed were allowed to settle for one minute. The supernatant of the upper 80% part of the cylinder was siphoned off and the separated mass was washed with water. Finally, the floc and non-flocs were dried, weighed to determine the flocculated solids content and iron. In all the experiments, NaOH, HCl or Na_2CO_3 of analytical grade was used as pH modifier. However, In some cases sodium hexameta-phosphate was used as the dispersant.

RESULTS AND DISCUSSION

Flocculation of particles relies on surface characteristics of the particles, nature of the aqueous environment, reagent type, etc. Microscopical studies indicated that BHQ sample

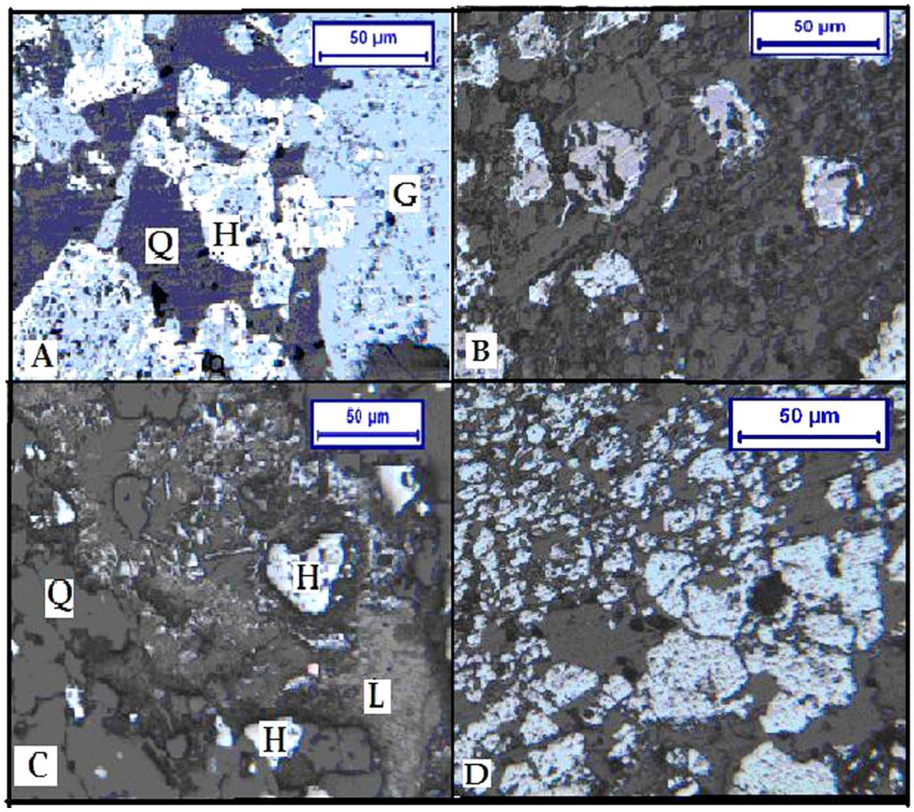

Fig. (2). A. Hematite (H) encloses grains of goethite (G) and quartz (Q). **B**. Hematite (white) rimed round with magnetite and silicate grains. **C**. Few grains of hematite(H) (flakes and needle type) are enclosed with limonitic **D**. Wide different size ranges of hematite (white) grains present along the inter-granular spaces of the silicates (quartz).

consists of hematite and quartz as the major minerals. Minor amounts of magnetite, goethite, limonite as well as kaolinite are also present in the sample. The microphotographs of BHQ sample are shown in Fig. (**2**). Widely varying hematite grains of different sizes are present along the inter-granular spaces of the silicates. As the particle sizes of hematite and silica are interlocked to each other, it is necessary to grind the material to a very fine size for effective liberation.

Flocculation Studies

Effect of pH

The effect of pH on amount of material settled in case of BHQ, hematite and quartz samples without addition of starch is shown in Fig. (**3**). It is observed that the amount settled decreased with increase in pH, and a good dispersion is obtained in the alkaline pH region. All the three minerals showed good dispersion in the pH range of 9-11. The hematite particles appeared to have agglomerated in acidic pH region (~pH 2.2) and correspondingly about 78% hematite particles were settled. It was also observed that the amount of settled mass of BHQ and quartz in comparison to hematite was very low. Hence due to difference in settling velocity of the minerals, selective flocculation for separation of hematite particles could be carried out below pH 9.0.

Fig. (**4**) shows the amount of material settled in the pH range of 2-12 for hematite, quartz, and BHQ with the addition of 20 mg/l of causticised starch. It is observed that the amount of BHQ and hematite that settled is about 98% where as, the same for quartz is only 60%. From the results, it is clear that selective separation of iron from silica using starch as flocculants at slightly alkaline pH can be achieved.

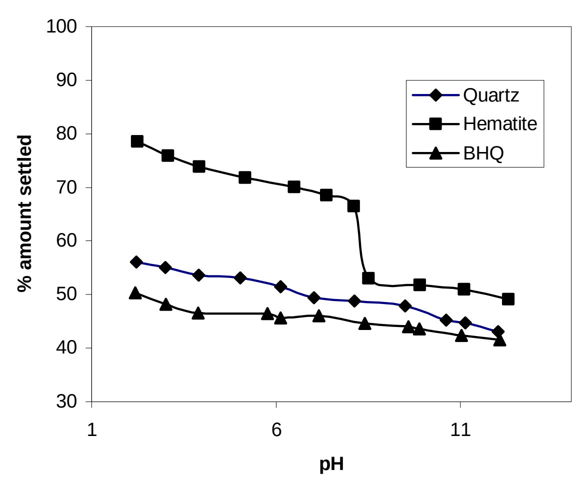

Fig. (3). Settling of minerals as a function of pH.

Effect of Starch Concentration

Flocculation studies were conducted for BHQ, hematite, and quartz at pH 9.0 as a function of starch addition. The effect of starch concentrations using 50 mg/l of sodium hexameta-phophate (Na-HMP) as dispersant is shown in Fig. (**5**). As seen from the figure, hematite and BHQ flocculate at a relatively low starch concentration. It is observed that about 95% of hematite is flocculated when starch dosage was just above 20 mg/l. In contrast, amount of quartz that settled is comparatively less at the same dosage of starch. Hence the best separation of quartz from hematite is possible at 20 mg/l of starch concentration. Such behavior of the individual minerals is a prerequisite for a successful selective flocculation of BHQ.

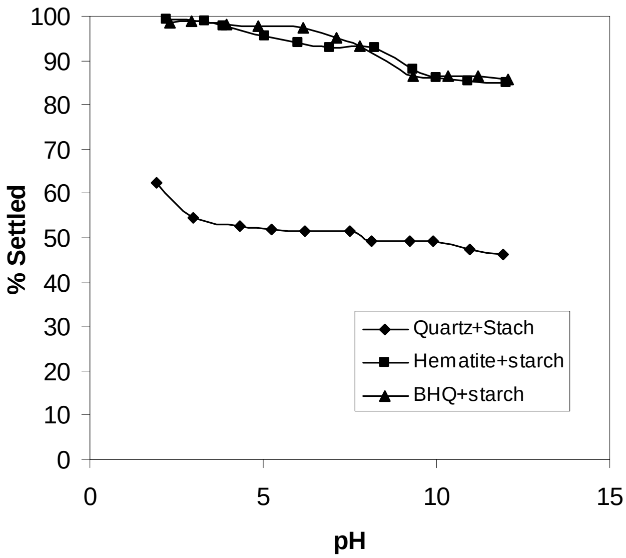

Fig. (4). Settling of minerals as a function of pH by the addition of 20mg/l of causticised starch.

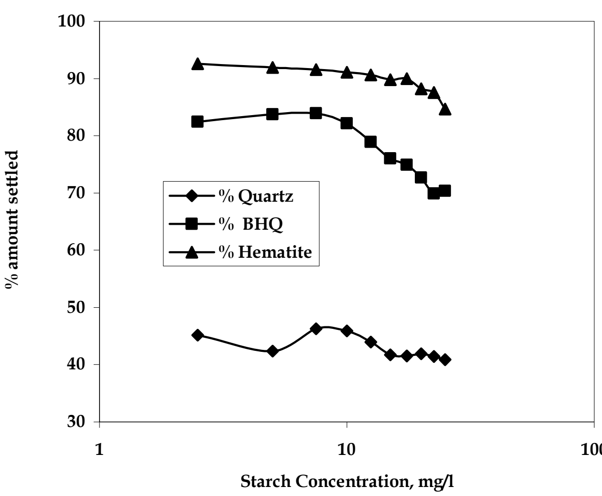

Fig. (5). Settling of minerals as a function of starch concentration, 10 mg/l, Na-HMP at pH 9.0.

Effect of Dispersant

The dispersion of any mineral prior to flocculation is important. Dispersants are used to prevent fine particles from aggregating and in many cases act to reduce the viscosity. During selective flocculation, it is essential to achieve the goal of separation so that the undesired minerals remain in dispersed state. The variation of the selective flocculation tendency of BHQ with various dispersants such as sodium silicate (Na-silicate), sodium hexameta-phosphate (Na-HMP), sodium hydroxide (NaOH) and sodium carbonate (Na_2CO_3) is shown in Fig. (**6**). Out of the four reagents studied, it is seen that Na-HMP is a better dispersant since the amount settled is less in comparison to other reagents. Hence further experiments were carried out by adding a small dosage of Na-HMP for the separation of iron particles from quartz.

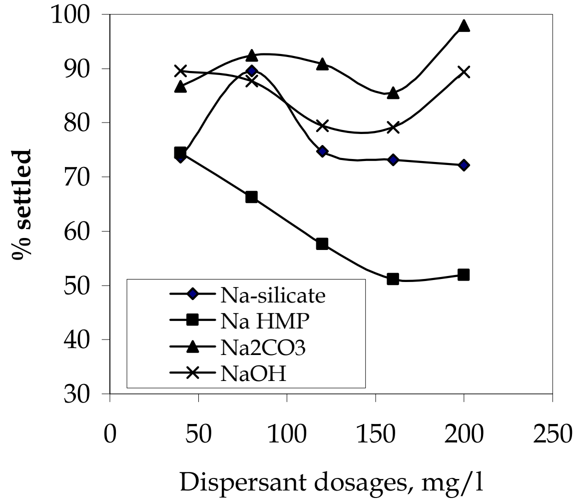

Fig. (6). Amount of BHQ particles settled as a function of dispersant concentration at pH 9.0.

Based on the above studies, selective flocculation tests were conducted for 1:1 hematite-quartz mixture at pH 9.0 as a function of starch addition. The results of the 1:1 hematite-quartz mixture by the addition of 150 mg/l of Na-HMP are shown in Fig. (**7**). It is thus possible to achieve 64.5% Fe with 92.0% of iron recovery from the resultant feed iron concentration of 34% Fe. The behavior of the mineral mixture thus indicates that flocculation of BHQ ore which essentially contains quartz and hematite would be possible. Accordingly, flocculation studies of BHQ ore under similar conditions were undertaken and the results are shown in Fig. (**8**). The results of this study indicate that it is possible to

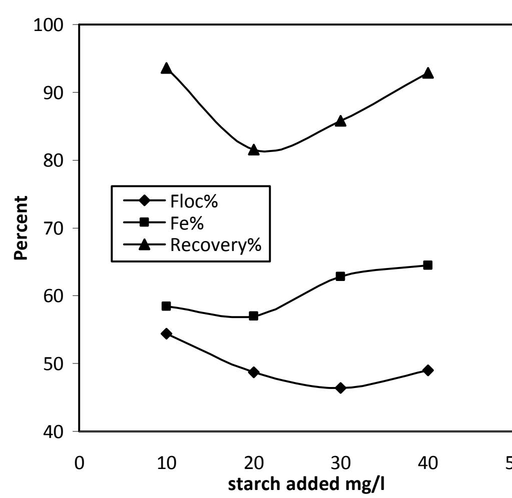

Fig. (7). Selective Flocculation of hematite–quartz mixture as a function of starch addition at pH 9.0.

achieve 57.2% Fe with 71% recovery from a feed containing around 38.9% Fe.

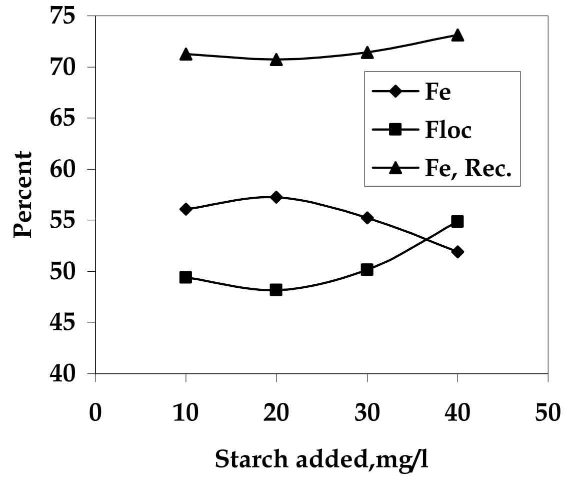

Fig. (8). Selective flocculation of BHQ ore as a function of starch addition at pH 9.0.

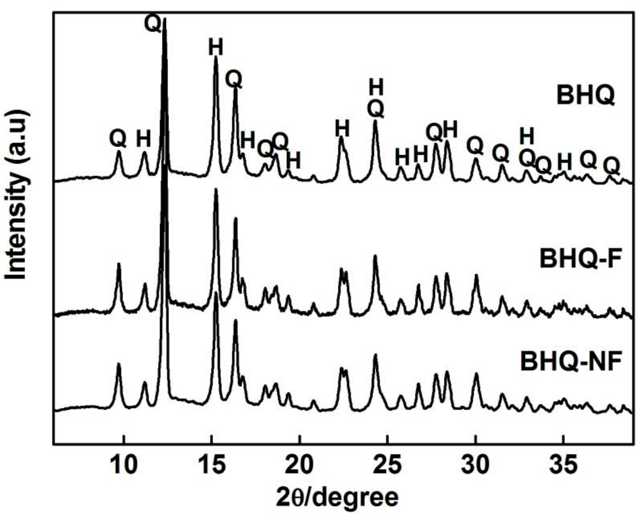

Fig. (9). XRD spectra of BHQ, flocculated (BHQ-F) and non flocculated samples (BHQ-NF).

XRD Studies

The XRD results of flocculated and non-flocculated products along with the BHQ ore sample are shown in Fig. (**9**). Two prominent mineral peaks of quartz and hematite are observed in all the three samples. It is also observed that the diffraction pattern of BHQ ore and the non-flocculated (BHQ-NF) fraction are identical. The intensity of diffraction peaks of quartz in the flocculated BHQ (BHQ-F) has slightly decreased where as, the peak intensity of hematite is enhanced slightly. According to the results of chemical analysis, the flocculated mass contains about 57.2% Fe, and the non-flocculated sample contains about 21.9% Fe. The quantitative estimation of different minerals as calculated by stereomicroscopy and by liquid mount thin sections is shown in Table **2**. The corresponding iron and silica values of the three products are also shown in the same table. The results indicate that hematite content in the flocculated material is 77.2% in comparison to 16.7% in the non-flocculated material. The corresponding quartz value in the flocculated material is only 4.5% where as, it is 80.6 and 33.0% in the BHQ and the non-flocculated material respectively. Thus these results point to the fact that flocculation of hematite particles from BHQ ore has taken place in the presence of quartz.

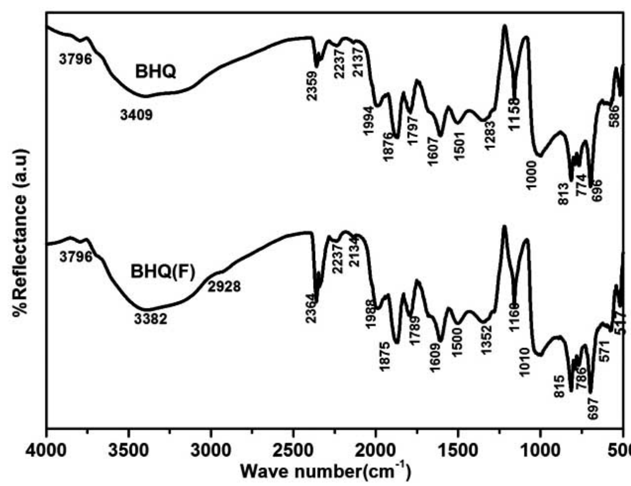

Fig. (10). FTIR spectra of BHQ and BHQ-flocculated with starch molecules.

FTIR Studies

FTIR spectra of BHQ sample and starch adsorbed BHQ sample is shown in Fig. (**10**) and their spectral assignments are given in Table **3**. The FTIR absorption profiles of the samples showed peaks at 774, 1283 and 696 cm^{-1}, corresponding to quartz. The hematite peaks have appeared near 1158, 774 and 517 cm^{-1}. The peaks at 1000 cm^{-1} are assigned due to (Si-OH) stretching. The spectrum shows that the bands above 3400 cm^{-1} is due to the presence of absorbed water. The broad peak that appears at 3134 cm^{-1} is due to OH group coupling with minerals. The FTIR spectra of starch generally shows four peaks at 3300, 610, 1350 and 1000 cm^{-1}

Table 2. Quantitative Mineralogy of BHQ ore and Products

Sample	Hematite, %	Goethite, %	Quartz, %	Fe, %	SiO_2, %
BHQ-iron ore	48.9	19.1	33.0	38.97	42.54
BHQ-Flocculated	75.2	19.3	4.5	57.2	16.3
BHQ-Non-flocculated	16.7	2.7	80.6	21.9	64.3

corresponding to water molecules that comprise of O-H group vibration, bending mode angles of O-C-H, C-C-H, and C-O-H. Absorption peaks due to water molecules are small in starch spectra. There are also some strong absorption peaks in the region between 1200 and 900 cm^{-1}. The starch peaks have shifted and appears at 3382, 571, 1352 and 1010 cm^{-1} indicating that starch molecules are adsorbed on BHQ surfaces [23-27].

Table 3. FTIR Spectral Band Position and their Assignment for BHQ and Starch Adsorbed Sample

Band Position, cm^{-1}	Assignments
3134	-OH stretching band
3796, 3400	Si-OH stretching mode, absorbed water
1607	Fe-O, Hematite
1158,734,517	Hematite
1000	(Si---OH)
1158,774, 517	quartz
1501, 1283	Asymmetric O.C-O stretching vibration
1000-500 (BHQ-Starch)	Sharp band due to starch absorption

Scanning Electron Microscope Studies

The electron micrographs of BHQ ore, flocculated and non-flocculated particles are shown in Fig. (**11**). The ore samples consisted of various size particles but mostly of below 20 µm size. The flocculated particles are relatively large in comparison to the non-flocculated material. It is noticed that the iron-bearing particles are present in coarser size compared to the silica bearing particles. In contrast, the particles in the non-flocculated fraction are relatively fine.

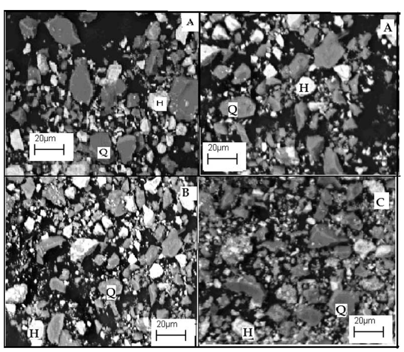

Fig. (11). SEM micrographs of BHQ, flocculated and non-flocculated particles. (A=BHQ(Feed),B=BHQ(F),C=BHQ(NF), H=Hematite, Q=Quartz).

Flocculation Mechanism

In a selective flocculation process, it is necessary to have particles of stable dispersion for which enough kinetic energy must be provided to the suspension to overcome the potential energy barrier. The energy barrier can be eliminated by neutralization of surface charge *via* adsorption of a suitable flocculant onto the particle surface which in turn act to bridge between the particles. Therefore, in the flocculation process high molecular weight flocculant is added to favor the bridging. In hematite flocculation system, it has been proved by several adsorption experiments that amylopectin has a greater affinity for hematite compared to amylose. The superior flocculation and adsorption ability of amylopectin has been attributed to its larger molecular weight and branched structure. Hanumantha Rao [28] *et al.*, have found that potato starch is a better selective flocculant containing amylopectin in the selective flocculation of iron ore slimes. The natural polymers are non-toxic, readily available in natural resources and are biodegradable compared to synthetic flocculants.

In the flocculation process of BHQ ore containing hematite and quartz, it has been observed that large and dense flocs of hematite are formed with the addition of potato starch in alkaline conditions while under the same conditions the fine quartz particles remain dispersed. These results are also in agreement with the results of individual minerals mixtures and BHQ ore tests as shown in Figs. (**3**-**8**). In fact, available literature on iron oxide and quartz separation using starch and polyacrylic acid indicates that natural starch is a better flocculant than the synthetic one [29].

The photographs of flocculated and non-flocculated particles are shown in Fig. (**12**). It is seen that most of the iron particles are blackish grey in color where as the non-flocculated material which is mostly a mixture of quartz and fine hematite are reddish in colour.

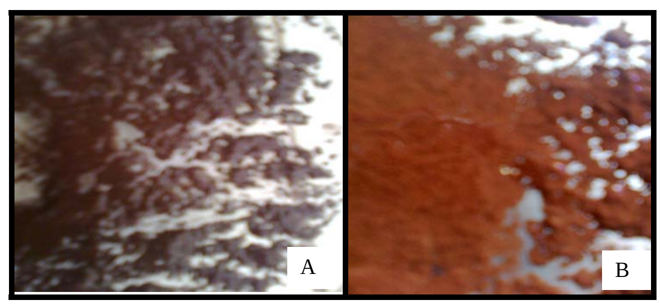

Fig. (12). Flocculated and non-flocculated particles. (A=Flocculated iron particles B=Non-flocculated quartz particles).

CONCLUSIONS

The banded hematite quartzite iron ore is a low grade iron ore and contains only 39% Fe, with 42.5% SiO_2, and 1.0% Al_2O_3. The mineralogical characterization studies indicate the presence of hematite and quartz as the major minerals. The initial flocculation experiments with synthetic mixture of hematite and quartz indicates a hematite concentrate of 64.5% Fe with 92.0% recoveryis achievable from a feed containing 34% Fe. Selective flocculation results with custicised starch as the flocculant to recover iron values from the sample indicates that it is possible to separate the iron values from other impurities at pH 9.0. The change of absorption peaks of starch through FTIR provides evidence the adsorption of starch on hematite particle present in BHQ. These particles agglomerate as viewed under SEM. The results of selective flocculation studies at particles size of below 20 µm indicates that a concentrate of 57.2% Fe with 71% recovery could be achieved. The concentrate can be blended with some high grade iron concentrate to use in iron and steel making.

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