JOURNAL GEOLOGICAL SOCIETY OF INDIA
Vol.93, April 2019, pp.443-454

Mineralogical and Chemical Characterization of Low Grade Iron Ore Fines from Barsua Area, Eastern India with Implications on Beneficiation and Waste Utilization

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ABSTRACT

The consumption of iron ore has increased rapidly over the past decade due to the tremendous growth of iron and steel industry. The depletion of high grade iron ore resources make it inevitable to utilize the existing low grade iron ores/ fines/ tailings with effective beneficiation to meet the present specification and demand. Enormous amounts of fines are produced both from the natural geological process as well during the mechanized mining operations which is hitherto in unknown resource present in the form of waste. Beneficiation and utilization of these fines/tailings still remains a challenging task. In order to find out the effective way of utilization of these fines, an in-depth characterization study is essential. A detailed insight into the different mineralogical attributes involving microscopic SEM-EDX, EPMA, XRD, FTIR, TGA, physical and chemical characterization are undertaken on the Barsua iron ores fines. These studies revealed that hematite and goethite are the major iron bearing minerals with gibbsite, kaolinite and quartz present as gangue that makes up the deleterious Al and Si content. Traces of magnetite is also observed along with martite. The liberation size of the sample is found to be below 150 µm. The bulk chemical composition shows around 57.67% Fe, 6.29% Al_2O_3, 3.52% SiO_2 and 6.93% LOI. Based on the detailed characterization, possible routes of beneficiation of the iron ore fines are discussed.

INTRODUCTION

Iron ore is the primary raw material for steel and metallurgical industries. The burgeoning demand for steel puts pressure on iron ore resources of India in respect of its grade and reserve. As per National Steel Policy, in order to support steel production of 110 million tonnes by 2020, the requirement of quality grade iron ore is placed at 190 million tonnes per year. Though India is one of the leading producers of iron ores in the world and also endowed with large reserves of high grade hematitic iron ores but the same are depleting day by day (Upadhyay et al., 2010). Considering the resource position with the gradual depletion of high-grade iron ores and increase in demand of good grade ore for steel production, there is a great emphasis on utilisation of low grade iron ores, fines, rejects etc. (Das et al., 2010; Vidyadhar et al., 2010; Anupam et al., 2010; Panda et al., 2011; Dash et al., 2012; IBM, 2012; Mohanty et al., 2012, Prasad et al., 2017).

Around 10-15% fines are being generated during the mechanical mining or other operations. It is also estimated that around 10 million tons of fines/slimes are being generated every year during the processing of hematite ore and lost as tailings containing around 48% - 62% Fe. No commercial plant has been set up so far to recover the iron values from such ores/fines for any industrial applications (Vidyadhar et al., 2010; Panda et al., 2011; Dash et al., 2012; IBM, 2012; Mohanty et al., 2012; Jena et al., 2015). Hence, comprehensive utilization of fines/rejects is important in conserving non-renewable fast depleting natural resources and also for sustainable development (Roy et al., 2007; Singh and Mehrotra, 2007; Roy and Das, 2008; Upadhyay, et al., 2009; Roy and Venkatesh, 2009 a, b; Roy, 2009; Upadhyay, et al., 2010; Dash et al., 2012).

Hematitic ore is the principal iron ore that is extensively used for manufacture of iron and steel in India (Mohanty et al., 2012). The quality/grade of ore is determined based on different iron making processes. The iron and steel industries are more conscious about the need for improving productivity. Therefore, the approach is towards obtaining cleaner feed having higher Fe content with least gangue and of homogeneous or consistent quality (Singh and Mehrotra, 2007; Mohanty et al., 2012; Jena et al., 2015).

Iron ore is being beneficiated all around the globe to meet the quality requirement of iron and steel industries. However, each source of iron ore has its own unique mineralogical characteristics that require specific beneficiation and metallurgical treatment to get the best product out of it. The selection of beneficiation strategy depends on the nature of the gangue present and its association with the valuable mineral (Mohanty et al., 2012; Gupta et al., 2012). Though India has large reserves of iron ore containing average grade around 58% Fe, but the performance of blast furnaces has been at lower levels in comparison with developing countries, mainly due to the presence of high levels of impurities such as alumina, silica and phosphorous affecting the blast furnace performance (Upadhyay et al., 2006; Singh and Mehrotra, 2007; Upadhyay et al., 2011; Mohanty et al., 2012).

Several beneficiation techniques such as washing, jigging, spiral, tabling, magnetic separation, flotation etc. are being employed to enhance the quality of the iron ore (Pradip, 1994; Yuhua and Jianwei, 2005; Mowla et al., 2008; Das et al., 2010; Mohanty et al., 2011; Dash et al., 2012; et al., 2014; Jena et al., 2015).

In India primarily washing, jigging and classification are being carried out for the beneficiation of iron ores at coarse size range and in some plants spiral, magnetic separation and flotation techniques are being employed to treat the fines generated during the process (Roy et al., 2007; Das et al., 2008; Upadhyay et al., 2009; Roy and Venkatesh, 2009; Upadhyay et al., 2010; Roy, 2010). However beneficiation of such fines generated during the process and stock piled at the mine site is really a challenging task.

A comprehensive characterization of these fines is a very important and has to be given due attention before any attempt for its processing (Mohanty et al., 2012; Jena et al., 2015). Mineralogical characterization of the raw feed material is very much essential to obtain information

0016-7622/2019-93-4-443/$ 1.00 © GEOL. SOC. INDIA | DOI: 10.1007/s12594-019-1199-4

on the mineralogy and different textural attributes (Roy and Venkatesh, 2009; Upadhyay, et al., 2010; Mohanty et al., 2012; Jena et al., 2013; Jena et al., 2016). It is also important to find out the modal distribution of ore and gangue minerals to assess the grade of ore. Grain size of the minerals and their textural relationship helps in deciding the size reduction and liberation (Das et al., 2008). The selection of suitable beneficiation process depends on the physical characters of iron minerals and gangue present in the ore (Mohanty et al., 2012; Jena et al., 2015). A combined study of different characterization techniques will give a better insight into the different mineralogical attributes of the ore and their bearing on processing of these fines (Mohanty et al., 2012; Jena et al., 2016). In this context, a detailed characterization of the iron ores fines of Barsua iron mine of Steel Authority of India Limited (SAIL,) Tensa in eastern India has been undertaken and possible methods of beneficiation routes are identified.

GEOLOGICAL SETTING

The Singhbhum-Orissa craton (SOC), eastern India, hosts one of the world's major iron ore resources. Large bodies of iron ores along with banded hematite jasper occur within this region (Fig.1). Three major iron ore belts occur as on the western, eastern, and southern portions having horse-shoe shape. The study area, Barsua iron ore deposit forms part of the western iron ore belt i.e. Noamundi-Jamda-Koira belt and is being currently mined by Steel Authority of India. Extensive works have been carried out in 1979 by Majumder and Chakraborty, then modified by Bhattacharya et al. in 2007, later on updated by Prasad et al., 2017, to understand the structural evolution and genesis of iron ore on regional scale.

The following types of iron ores are usually found in the Barsua mines, viz., massive, laminated, flaky friable ore, blue dust and lateritic/limonitic/goethitic varieties ranging from high grade to medium-low grade ores. Owing to the continuous mining operations, the high grade ores are generally getting depleted leaving behind the medium-low grade ores. The low grade iron ores are friable in nature and occur as pockets due to the effect of leaching composed of hematite and iron hydroxide minerals in which fine recrystallized lenticular hematite grains are seen along with goethite. Broadly the iron ores are mainly composed of hematite, martite along with goethite, minor amounts of magnetite, siliceous and clay rich gangues. Due to continuous mining operations, large amounts of fines are generated along with natural geologically occurring fines such as blue dust and flaky friable ore intermingled with goethite/limonite/lateritic/clayey substances rendering them as low grade ores.

MATERIALS AND METHODS

Sample Preparation

In order to characterize this low grade iron ore, around 400 kg of representative iron ore fine (~ -6 mm) sample was collected from the Barsua iron ore mines (SAIL, Tensa) and used for the present investigations. The 'as received' sample was thoroughly mixed and different representative samples were drawn through coning and quartering for different characterization studies.

Characterization Studies

Microscopic studies were carried out by optical microscopy using Leitz Ortholux microscopes, CSIR-IMMT, Bhubaneswar, India, for different polished sections of bulk sample and size fractions.

The mineralogical composition of the sample was evaluated by X-ray diffraction (XRD) analysis using PANalytical X'Pert-PRO X-ray diffractometer fitted with a goniometer PW3050/60. Copper was used as anode material and the generator settings were maintained at 30 mA and 40 kV. The X-ray patterns were acquired in the 2è range of 10 - 80° with a step size of 0.001°/s. The room temperature was maintained at 25ºC during the measurements at CSIR-NML, Jamshedpur, India.

The samples were examined using Scanning Electron Microscope (SEM-EDX) at CRF-IIT(ISM), Dhanbad, India. SEM analysis was performed using a FEI 430 Nova Nano-scanning electron microscope equipped with a tungsten filament coated with zirconium oxide. The micro analyser was a JEOL make model JXA 8230. The acceleration tension was kept at 15 kV for all the measurements.

Qualitative mineral identiûcation for the sample was done by Fourier Transform Infrared Spectroscopy (FTIR) at CRF-IIT(ISM), Dhanbad, India, using Thermo Scientific Nicolet 6700 series (iS10) FTIR Spectrophotometer over the range 4000 - 400 cm^{-1} at room temperature, with an accuracy of 0.01 cm^{-1} and resolution of ±4 cm^{-1}.

The thermal characteristics of the sample was determined using Netzsch Thermo Gravimetric Analysis (TGA) at FME-IIT(ISM), Dhanbad, India, instrument model STA 449 F3 in the temperature range of 30 - 950 ^{0}C at a heating rate of 10 ^{0}C/min in an inert (nitrogen) environment.

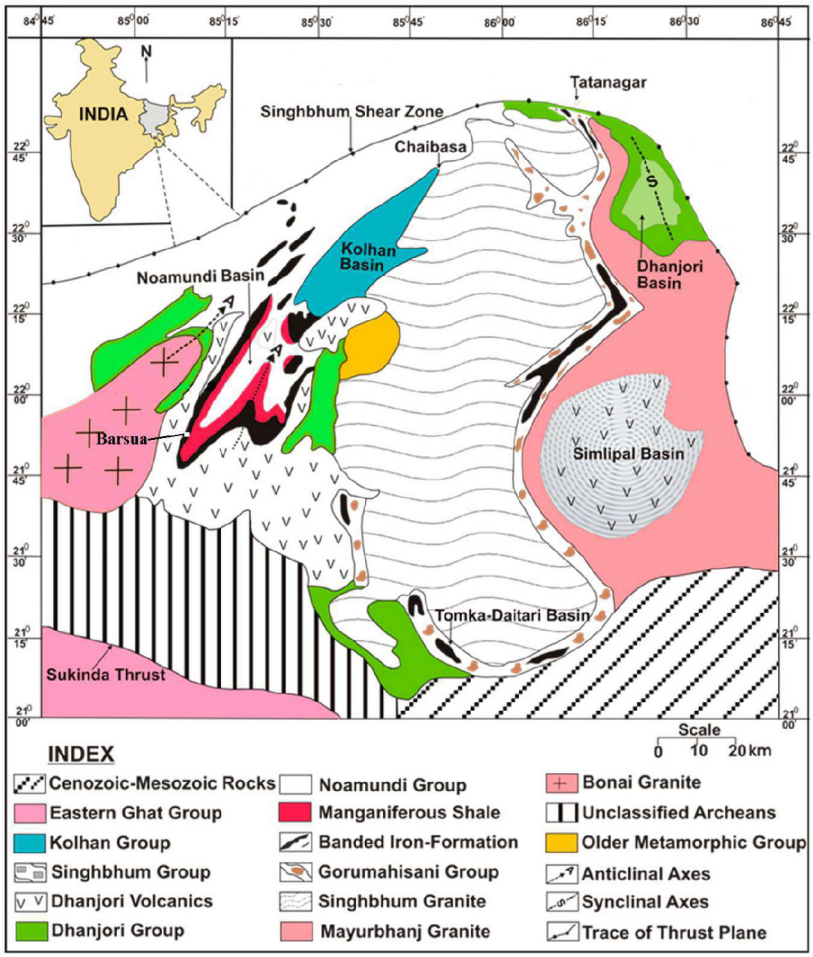


Fig. 1. Geological map of iron ore deposits of Barsua and other associate deposits of Keonjhar–Sundergarh districts, north Orissa, eastern India showing potential iron ore bodies including Barsua and other iron ore prospects of Singhbhum Orissa iron ore craton (Majumder and Chakraborty, 1979; Bhattacharya et al., 2007 and Prasad et al., 2017).

The detailed chemical composition of the sample was determined through XRF at CSIR_IMMT, Bhubaneswar, India, using PHILIPS, MagiX PRO X-ray Fluorescence spectrometer operated at 30 kV voltage and 100 mA current with counting time 5 sec, except LOI which was determined by heating the sample at 950 °C for 1 h in a muffle furnace (Jena et al., 2013).

The size and chemical analysis was carried out using standard BSS sieves by wet method for the sample. Each size fractions were then dried, weighed and analyzed for chemical constituents through XRF. Size analysis was carried out at FME-IIT(ISM), Dhanbad, India.

Thin polished section (TPS) of iron ore fines was prepared with diamond polish, then the sample was subjected to carbon coating, TPS of iron ore fines was then subjected to EPMA analysis, which was determined by fifth generation electron probe micro analyser SX Five from CAMECA France equipped with five wave length dispersive spectrometers, BSE detectors, SE detectors, cathodoluminance and sophisticated visible light optics providing image magnification ranging from 40 to 400,000. Detail of the specifications of EPMA is as follows: SX-Five with LaB6 Column.

Maximum accelerating voltage: 30KV, beam diameter in analytical mode: 100 nm @ 20 KV, 10nA:200 nm @ 10 KV, 10nA, maximum beam current: Up to 1 uA, beam stability: +-0.2% per hour @ 20kV, 10nA. EPMA was carried out at CRF-IIT(ISM), Dhanbad, India.

RESULTS

Optical Microscopic Studies

Microscopic studies of the iron ore sample under reflected/ transmitted microscope reveals that the present iron ore sample is mainly composed of iron oxide-hydroxide phases in different proportions with varying amounts of hydrated aluminium hydroxide and silicate/clay minerals like gibbsite, kaolinite and quartz. Hematite and goethite are the major iron bearing minerals, whereas small amounts of magnetite is also observed. Figure 2a-d shows that quartz, hematite and goethite occur together and their textural relationships amongst coarser to finer oxide grains and this inference has significant influence on the beneficiation of this type of iron ores. The study indicates that total separation of iron minerals from such silicate association is very difficult and may not be economic as this may involve lot of grinding to finer size for complete liberation as selection of unit operations for physical beneficiation is based on liberation of the mineral grains. These minerals are interlocked very intimately and intricately with each other along with silicate matrices as shown in Fig.2(a). Goethite shows colloform texture in association with hematite and clay as shown in Fig.2(b). Some patches of hematite (white) are also found within the goethite matrices as observed in Fig. 2(c). Figure 2(d) shows most of the hematite grains are fine-grained and interlocked within the clay/silicate matrices in a random fashion.

Size wise microscopic studies were carried out at two different sizes i.e. -6000+150 µm (Fig. 3a & 3b) and -150 µm (Fig. 3c & 3d). From the size wise microscopic study Fig. 3(a-d), the grains that are observed in the sample are hematite, goethite, gibbsite, quartz, and kaolinite. From the mineralogy it is evident that the sample has two distinct types of valuable minerals i.e. (i) predominantly micro-crystalline hematite grains (fine to medium grained) carrying disseminated goethite inclusions and (ii) micro-crystalline hematite particles intermixed with micro-crystalline goethite, gibbsite and ferruginous clay. Goethite replaces hematite in different degrees and fills up the voids and fractures during weathering.

Kaolinite occurs in intimate association with goethite and free quartz grains are also observed, therefore it is assumed that the silica is available in the form of kaolinite and quartz. Colloform texture of the weathered goethite was observed in the Fig. 3(a). Predominant

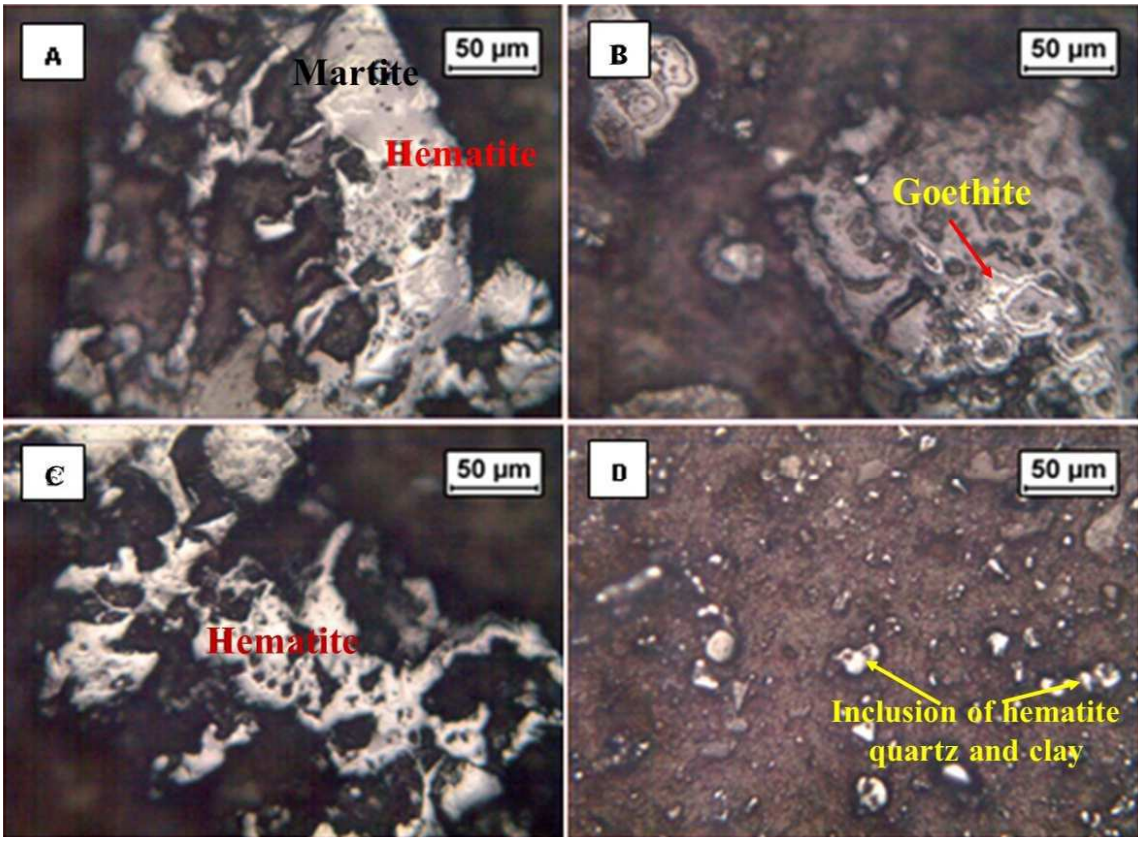


Fig. 2. Photomicrographs of the iron ore samples under reflected microscope; **(A).** Hematite and Goethite interlocked very intimately and intricately along with the clayey matrices and at places martite is also seen, **(B).** Goethite showing colloform texture in association with hematite and quartz, **(C).** Goethite encloses patches of hematite (white), **(D).** Tiny hematite grains within the clayey matrices.

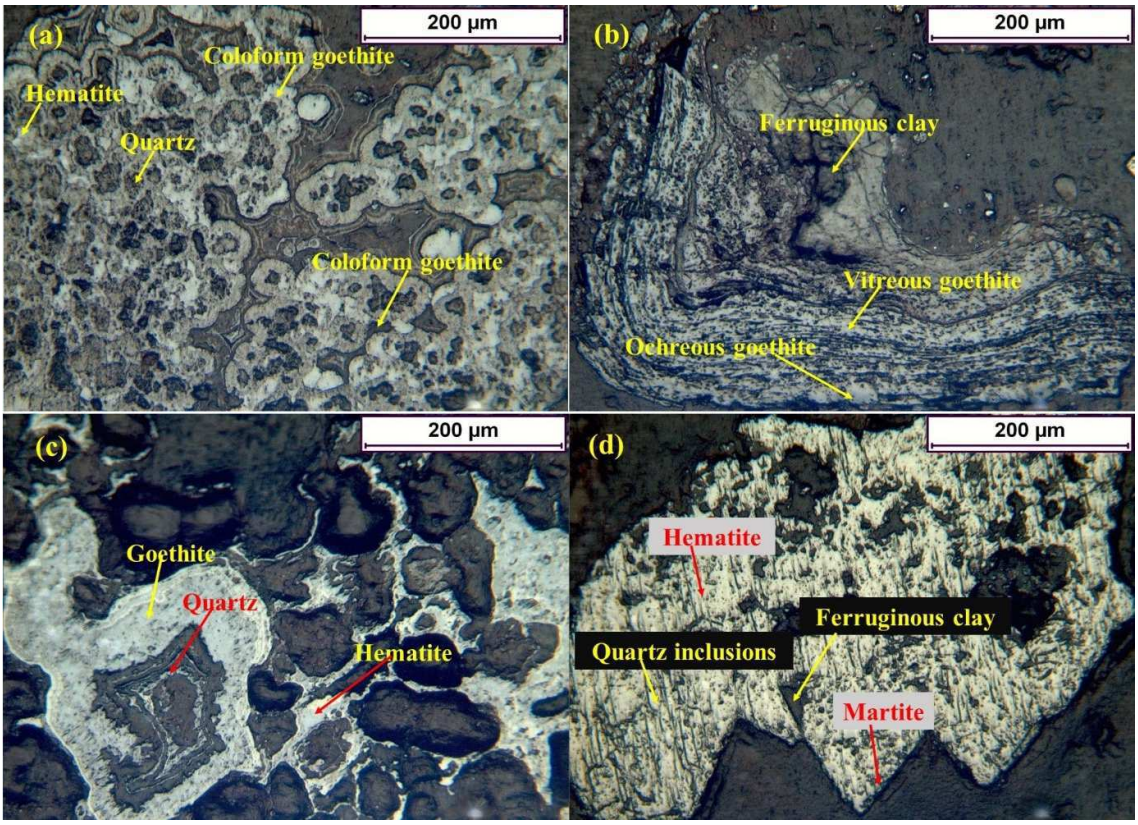


Fig.3. Size wise photomicrographs showing **(a)** Revealed that huge goethite surrounded by micro platy hematite with less amount of clay, **(b)** represents goethite associated in colloform structure with clay and hematite, **(c)** Goethite distributed unevenly with hematite and clay, **(d)** Indicates hematite patches of goethite in ropy shaped clayey matrices.

alumina-contributing mineral is gibbsite and occurs intimately intermixed with goethite and hematite (Fig. 3b). Gibbsite and clay minerals (kaolinite) grains are present as microcrystalline to cryptocrystalline aggregates and are thoroughly intermixed with goethite, and other silicate minerals. Majority of the kaolinite grains are embedded with iron oxide/ hydroxide minerals as shown in Fig. 3(d).

Figure 3(d) shows martitization which denotes the conversion (oxidation) of magnetite to hematite and hematite appears to be a martitized with relict magnetite grains. The martite, which is pseudo-morph of magnetite, in most cases, retains the shape of original magnetite. Martite, hematite and quartz are in complete crystalline form. The kaolinite is also occurring as patches in hematite and goethite. This may have resulted due to the leaching out of pre-existing minerals. Kaolinite is very fine (15-25 micron) and occurs in association with goethitic intercalations along the bands. In Fig. 3(c), patches of kaolinite and goethite in hematite, micro platy hematite intercalated with clay are prominently observed. It is also observed that quartz presents as either in liberated form or attached to the edges of the iron-bearing minerals, especially in the images from the finer fractions. Gibbsite and clay minerals (kaolinite) grains are present as microcrystalline to cryptocrystalline aggregates and are thoroughly intermixed with goethite, and other silicate minerals. Most of the quartz grains also carry very fine inclusions of hematite. Hence, the production of a high grade concentrate with a high recovery is very difficult. As, the quartz is very finely disseminated, it is extremely difficult to attain good liberation.

Modal Analysis

Concentration of minerals in two different size fractions of 'as received' sample were determined by counting the individual mineral particles under microscope in these samples. The results of the counting studies (modal analyses) for the concentration of hematite, goethite, magnetite, clay, quartz and interlocked particles for the same size fractions are given in Table 1. The results indicate that the concentrations of individual hematite and goethite mineral particles are more in both the coarse and finer fractions whereas the hematite percentage is more in finer fraction of -150 µm. Presence of free quartz particles in both the fractions is around 5 - 6%, whereas concentration of free clay particles is around 15 - 18%.

The presence of high amount of fine clay particles create hindrance while separating the valuables through beneficiation techniques. These clay particles may be deslimed prior to upgradation of valuables. Gibbsite is totally reported in interlocked particles. Minor amount of magnetite is also found. The results shows that the percentage of interlocked particles is less in finer fraction whereas around 34.1% free hematite particle concentration is found in the finer fraction.

Liberation Studies

Liberation studies show maximum interlocking in the coarser size than finer fractions. The graphical representation of liberation studies is shown in Fig. 4. The results indicate that 84.59% liberation can be obtained with the size fraction of -150 +75 µm. Percentage of liberated particles is more at finer fractions whereas less at coarser fraction.

Table 1. Modal% of different minerals present in the two size fractions of the sample.

Size, µm	Hematite %	Goethite %	Magnetite/ martite %	Clay %	Quartz %	Inter-locked/ mixed %
-6000 +150	28.0	21.4	4.0	15.7	6.0	24.1
-150	34.1	20.1	3.0	18.0	5.4	18.8

The coarser (1000+500 µm) size fraction contains around 47.05% locked particles. It is observed that more effective liberation may be possible below 150 µm size (Jena et al., 2015). Even better liberation can be achieved below 75µm, i.e. 90%. To achieve below 75 µm, the grinding cost will affect the beneficiation cost. Hence, for effective separation of valuables during physical beneficiation, the sample may be required to grind below 150 µm size.

X-Ray Diffraction Studies

The 'as received' sample was characterized by the XRD to find out the mineral constituents. The minerals are identified by standard JCPDS data file. XRD analysis of the 'as received' sample revealed that the sample is dominated by hematite followed by goethite, gibbsite, kaolinite and quartz. The major iron bearing opaque minerals are hematite followed by goethite. The aluminium bearing hydroxide mineral is identified as gibbsite, whereas the silicate minerals found are kaolinite and quartz. Traces of magnetite are also found in this sample. This observation is well supported by microscopic studies. The XRD patterns of the sample are shown in the Fig. 5.

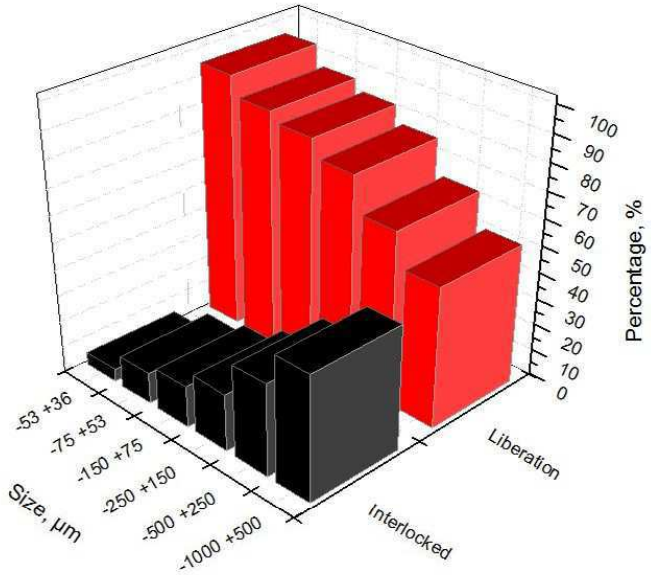


Fig. 4. Results of liberation studies showcasing the interlocked and liberated mineral assemblages, their size ranges and their recovery percentage.

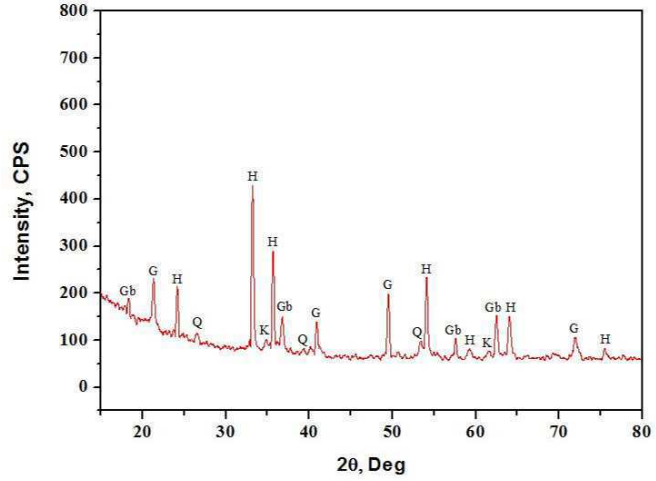


Fig. 5. XRD patterns of Barsua iron ore sample represents the presence of Hematite (H), Goethite (G), Gibbsite (Gb), Kaolinite (K) and Quartz (Q) minerals.

SEM-EDX Studies

Scanning Electron microscopic analysis shows that it is a goethite rich surface; and iron bearing mineral hematite, goethite along with martite is present. Hematite and goethite grains are stacked upon one another. The grains that are observed in the sample are hematite, goethite, gibbsite, quartz, and kaolinite. The results of the SEM studies are shown in Fig. 6. SEM studies indicate the presence of different shapes of hematite and goethite associated with each other, suggesting their formation conditions. Hematite grains stacked upon one another as fine laths enclosed by goethite. The SEM results also confirm the presence of iron and aluminium hydroxide mineral phases with minor quartz. These observations are well corroborated with the results of XRD analysis.

Size wise SE Microphotographs of iron ore fines are depicted as: Fig. 7(a) showing that stacking of goethite and hematite laths one upon another forming a complex texture; Fig. 7(b) Hematite grains distributed as ropy structure with irregular distribution of alumina; Fig. 7(c) Hematite and goethite are associated finely with quartz and alumina, goethite and quartz are in edge shape as observed; Fig. 7(d) Goethite and alumina are associated with clayey matrices below 1micron along with the presence of iron oxide minerals in spacious amount, silicon oxide and aluminum oxides are present in moderate amount whereas calcium, potassium and titanium are present in very trace amount. Sample contains mixture of oxy-hydroxides of iron and silicate gangue material and contains high alumina. Kaolinite occurs as irregular patches and thin films. Many a times, minute crystals of silicate grains are present as inclusions within the hematite.

SEM-EDX studies represents, morphology and chemical composition of the sample. Botryoidal, microporous, earthy varieties of goethite forming fine intergrowths with martitic hematite were observed. SEM-EDX revealed that the bulk sample has low iron content along with Al_2O_3 4.08% and SiO_2 2.13% contents (Fig. 8).

Similarly size wise EDX analysis revealed that the earthy-like goethite has low iron content along with Al_2O_3 (1.24–4.46%) and SiO_2 (0.77–3.49%) contents, shown in the microanalysis tables associated with the graphical results (Fig. 9(a-d)). The term earthy-like goethite was defined considering the morphology (brownish to ochre appearance) and the chemical composition. It can be classified as intergranular and intragranular goethite, according to its textural mode of occurrence.

Electron Probe Microanalysis (EPMA)

Electron probe microanalysis shows that the sample predominately contains goethite rich zone; and iron bearing mineral hematite, goethite

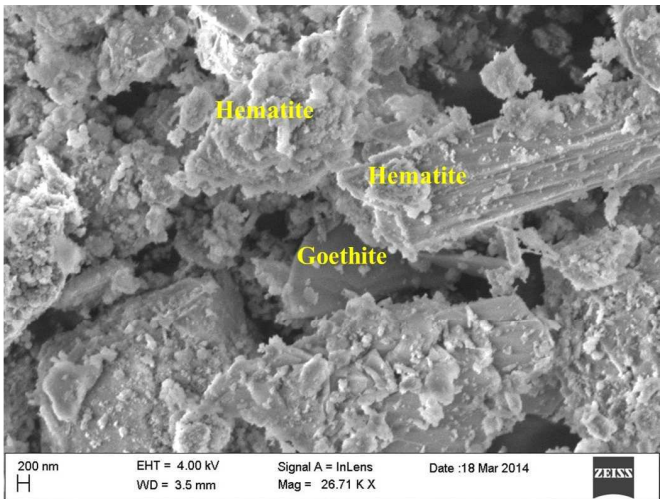


Fig. 6. SEM micrograph of the sample indicating formation and morphology of iron ore fines sample.

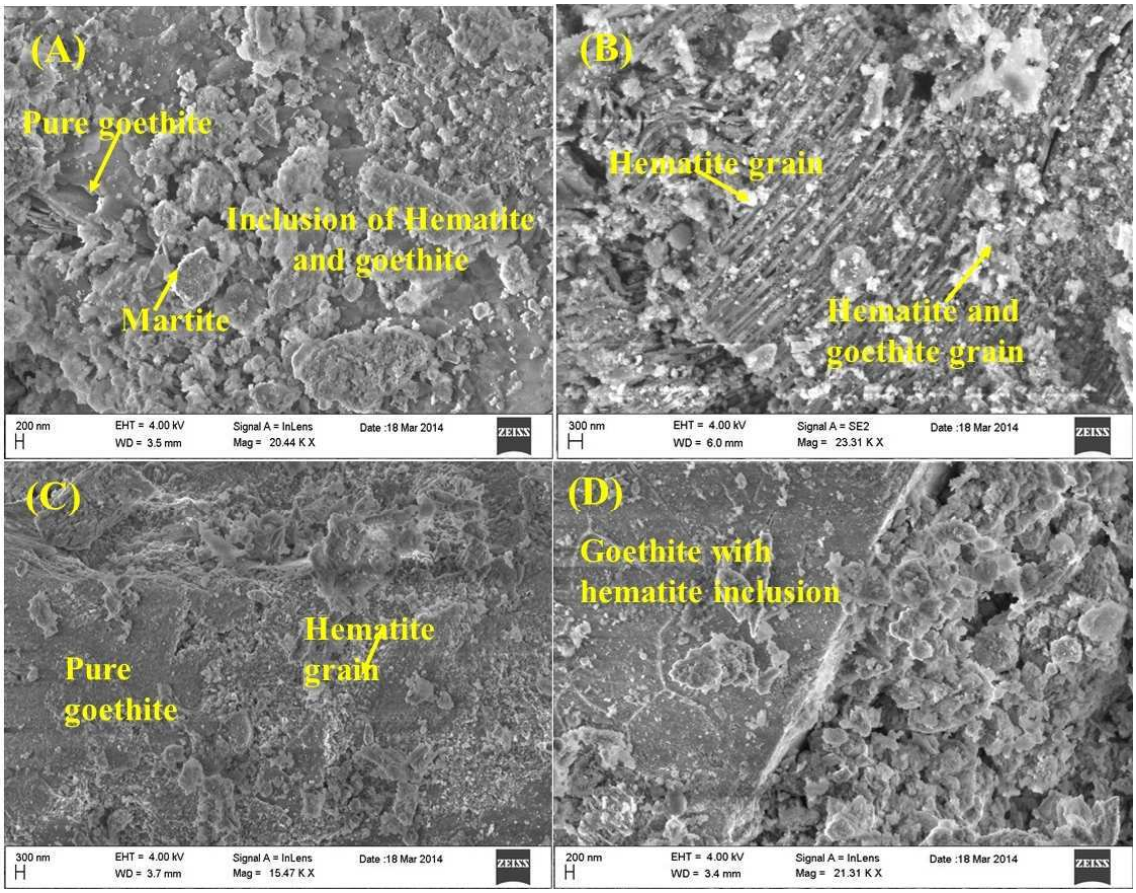
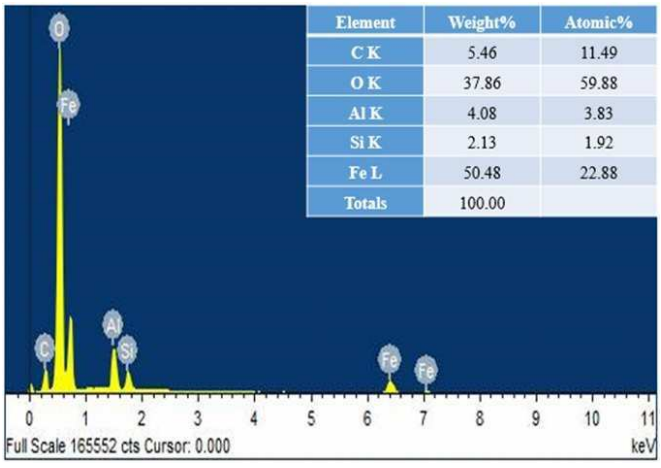


Fig. 7: SEM microphotographs showing the result of iron ore sample of following size fractions: -6000+1000µm **(a)**, -1000+150µm **(b)**, -150+53µm **(c)** and -53µm **(d)**

along with martite is also present. Hematite and goethite grains are stacked upon one another. The grains that are observed in the sample are hematite, goethite, gibbsite, quartz, and kaolinite (Fig. 10 and Table 2). From the mineralogy it is evident that the sample has two distinct types of valuable minerals i.e., (i) predominantly crystalline hematite grains having euhydral and subhydral structure (fine to medium grained) carrying disseminated inclusions seen in Fig. 10(a-d) and (ii) micro-crystalline hematite particles intermixed with micro-crystalline goethite and gibbsite are seen in Fig. 10(b). Huge cracks within the medium to fine grained Fe bearing minerals are found in most of the iron bearing minerals that are fully or partially weathered resulting in substitution of most iron oxides with clay. The aluminous clay particles intermixed with iron minerals and the clay are present



Element	Weight%	Atomic%
C K	5.46	11.49
O K	37.86	59.88
Al K	4.08	3.83
Si K	2.13	1.92
Fe L	50.48	22.88
Totals	100.00	

Fig. 8. Elemental distribution of bulk sample through SEM-EDX

with flaky texture (Fig. 10 c & d). The presence of porous surface in Fig. 10(b, d), denotes that substitution has occurred in goethite grains which are formed due to weathering/extensive oxidation of iron oxides. While the kaolinite and gibbsite are the major gangue phases noted in the sample.

It is important due to its association with iron ore and makes a chief gangue in different types of iron ores. Kaolinite in iron ores occur as void filling and fractures filling in the iron ore Fig. 10(d) and occurs as patches in hematite and goethite. This may have resulted due to the leaching out of pre-existing minerals. Various forms of mineral forms are present i.e. (a) fine grained quartz and clay inclusion into iron bearing minerals, (b) patches of kaolinite in hematite, (c) micro platy hematite intercalated with clay, (d) kaolinite occur as fracture filling, (e) hematite flakes in clay ground mass and quartz.

Fourier Transform Infrared Spectroscopy (FTIR) Studies

Figure 11 shows the IR spectrum of Barsua iron ore fines. The broad band observed in the range between 3127 and 3520 cm^{-1} is due to the bulk hydroxyl stretch and a less intense band at 3622 cm^{-1} can be attributed to surface hydroxyl group. The complete disappearance of the absorption band at 3748 cm^{-1} indicates that the phosphate was adsorbed on the surface of the iron oxy-hydroxides and the band at 3622 cm^{-1} was not replaced by adsorbed phosphates (Gadsden, 1975; Cornell and Schwertmann (2003, 2006); Russell et al., 1974). An intense band observed at 1632 cm^{-1} is due to adsorption of water and the band at 1098 cm^{-1} can be related to quartz or polysaccharide carbohydrates (Schmitt and Flemming, 1998). Kaolinite is also present in this sample, which was identified by the presence of bands at 1031 cm^{-1} (Si-O-Si) and 910 cm^{-1} (Al-O-H).

The OH bending bands at 910 cm^{-1} (d-OH) and 799 cm^{-1} (g-OH) which vibrate in and out, respectively, are important diagnostic bands

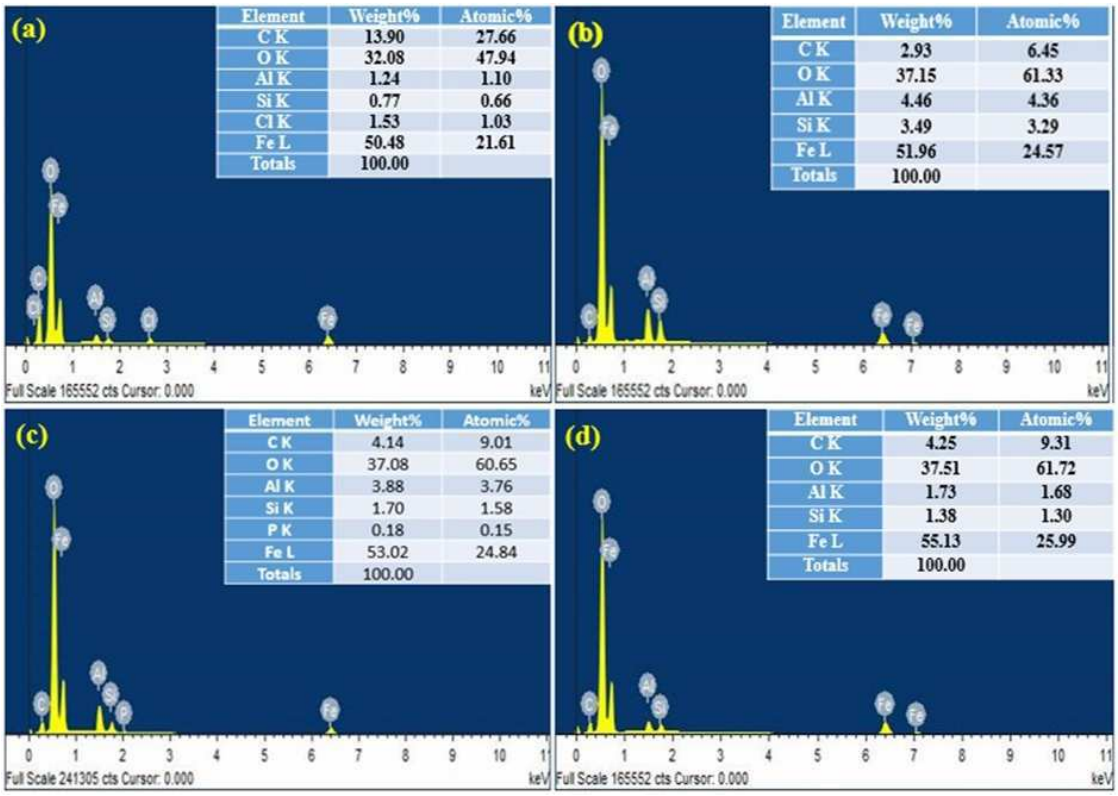


Fig. 9. Size-wise elemental distribution through SEM-EDX suggesting variable elemental constituents as per their chemical composition and presence of nature of the iron ore mineral along with gangue.

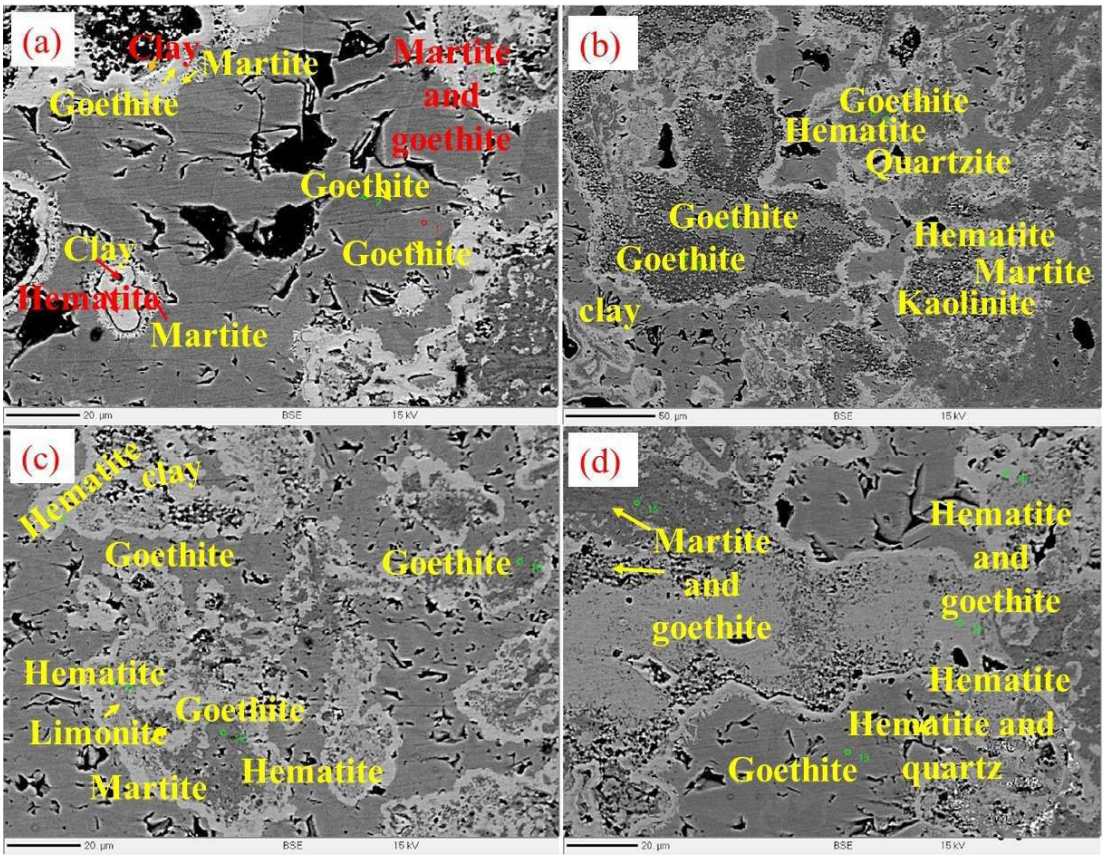


Fig. 10. Electron microscopic mapping of mineral constituents of Barsua iron ore sample showing presence of gibbsite and kaolinite phase along with hematite, goethite and martite iron ore minerals. **(a)** Showing predominantly crystalline hematite grains having euhedral and subhedral outlines (fine to medium grained) carrying disseminated inclusions of gangue and alumina bearing substances; **(b)** shows micro-crystalline hematite particles intermixed with micro-crystalline goethite and gibbsite. **(c & d)** shows the ferruginous clay inclusions accumulated on their active surface.

Table 2. EPMAMineral chemistry of selected iron bearing minerals as well as gangue constituents using EPMA from Barsua area

	Goethite													Hematite		
Sl. No.	1	2	5	6	7	8	10	11	12	13	14	15	16	3	4	9
SiO_2	0.19	0.39	0.34	0.31	0.27	0.65	0.57	0.39	0.97	0.16	0.29	0.81	0.47	0.25	0.18	0.3
Al_2O_3	0	0	3.33	0.02	2.47	1.92	1.5	2.18	2.23	0.03	2.12	4.12	1.91	1.52	1.66	1.58

and also provide information about the crystallinity as well as the extent of Al substitution in the goethite structure (Gadsden, 1975; Cornell and Schwertmann, (2003, 2006)). The most characteristic IR absorption bands of hematite are present in the low frequency region (<600 cm^{-1}). Infrared vibrations of hematite formed from the Si-free ferrihydrite were observed by two bands at 417 and 504 cm^{-1} (Vempati et al., 1990; Bikiaris et al., 1999). This FTIR analysis is in good agreement with the XRD analysis.

Thermo gravimetric Analysis

Thermo gravimetric analysis (TGA) technique can characterize materials that exhibit weight loss or gain due to decomposition, oxidation or dehydration on heating (Wendlandt, 1986). TG curve shown in Fig. 12 indicates that the weight loss below 200°C is due to the loss of adsorbed water present in the sample and its amount depends on the particle size. Goethite loses weight between 250° and 400°C due to structural OH, by the dehydroxylation reaction $2OH^- \rightarrow O^{2-} + H_2O$. The dehydroxylation endothermic peak occurs at 341°C for this sample. The temperature of this endotherm increases with increasing crystal size and influenced by amounts of Al-for Fe-substitution (Cornell and Schwertmann (2003, 2006)) and structural defects (Mackenzie and Berggren, 1970). The absence of any peak within 400°C to 850°C indicates that there is no ferrihydrite/goethite–hematite phase transformation observed in this sample (Salama et al., 2015).

Physical and Chemical Characteristics

The detailed chemical analysis, to know the major elemental composition of the 'as received' sample, was determined using XRF and the results are given in Table 3. It indicates that the sample contains around 57.67% Fe, and is rich in Al_2O_3 content with lesser silica contents. From the results it is observed that the presence of clay/aluminium bearing minerals are more compared to the quartz as evidenced from mineralogical studies as well. High LOI and alumina contents in the sample may indicate the presence of goethite, gibbsite and kaolinite (Anderson et al., 2014).

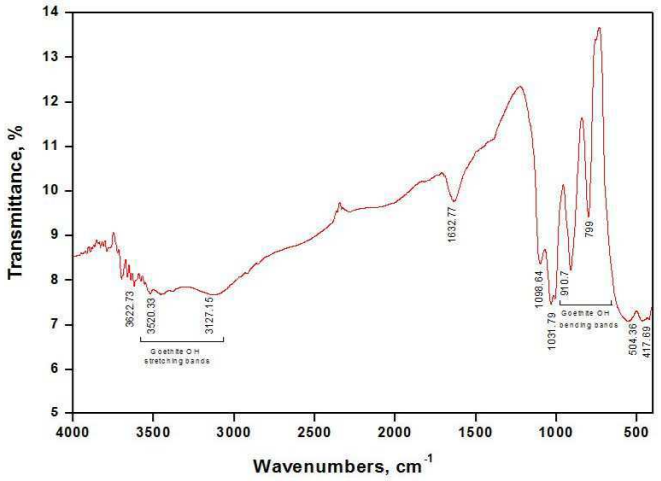


Fig. 11. Infrared spectrum of the Barsua iron ore sample showing different goethite bands indicating crystallinity and alumina substitution.

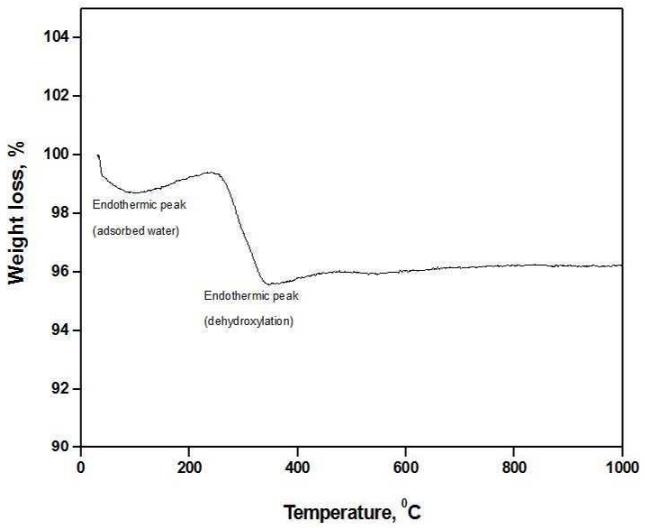


Fig. 12. Thermo gravimetric analysis patterns for the Barsua iron ore sample indicating decomposition and oxidation characteristics of the sample.

The size and size wise chemical analysis of the 'as received' sample were carried out by wet chemical method and are given in Fig. 13 and Fig. 14 respectively.

The results indicate that the 80% and 50% passing sizes of this sample are in the range of 1.9 mm and 150 µm respectively. Figure 14 shows that the grade Fe content, is increasing towards finer sizes, whereas, the silica and alumina contents are lesser in the finer size fractions. The Fe content in below 150 µm size fractions is about 60.0% which corresponds to a weight recovery of around 51.0%. It seems that most of the iron bearing minerals present in this sample are liberated below 150 µm size. This was also evidenced from the mineralogical studies. Hence, as observed from these characterization studies, further grinding of the sample to below 150 µm size may be required for effective separation of iron values through suitable beneficiation techniques (Jena et al., 2015).

DISCUSSION

In the present study, high aluminium content was observed in goethite, which occurs abundantly in the samples studied, showing varied morphologies closely associated with hematite as fine inclusions. Goethite is more friable than the other minerals in the samples; therefore, it contributes more to production of fine after processing. Furthermore, the presence of goethite inside the micropores of the martitic hematite makes its removal difficult during desliming, due to the very small size of the micropores. The abundant occurrence of

Table 3. Chemical analysis of 'as received' sample indicating percentage of valuable and gangue minerals

Constituents	Percentage, %
Fe	57.67
Al_2O_3	6.29
SiO_2	3.52
LOI	6.93

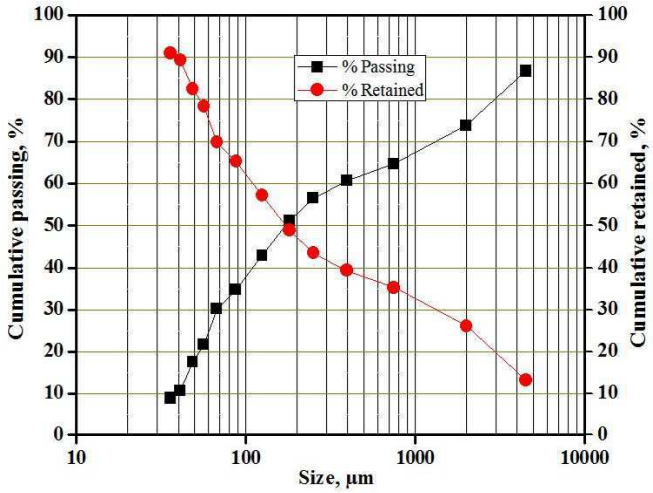


Fig. 13. Size analysis of 'as received' sample represents the average size of the sample i.e. d_{80} and d_{50}. The d_{80} and d_{50} of this sample are in the range of 1.9 mm and 150 ìm respectively.

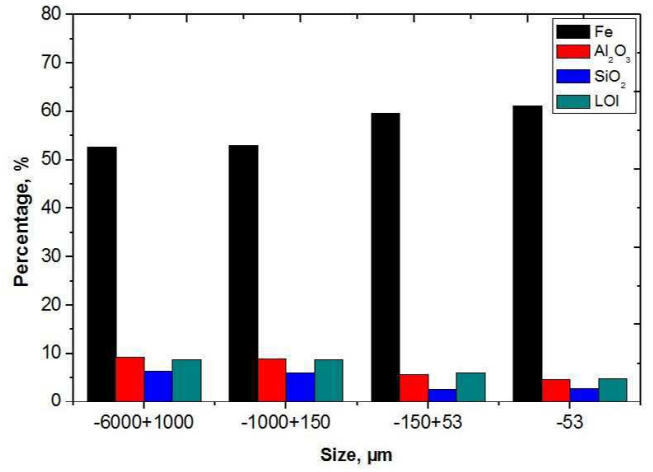


Fig. 14. Size wise chemical analysis of 'as received' sample indicating optimum grade achieved at -150 µm size range.

different types of goethite in the samples studied, mainly deep brownish goethite, generates huge amounts of fine and porous material during processing and accounts for the main alumina bearing phase of the sample.. Goethite is abundant and exhibiting secondary colloform texture in cavities along the weaker bedding planes. Such voids and inter-granular pore spaces along the weaker bedding plane are very fragile making the hematite and goethite friable during processing.

Beneficiation is mostly based on the degree of liberation of the valuable minerals that depends on the size and texture of the constituent minerals. The size of the wanted mineral decides at which grain size most of the valuables are liberated so that they can be easily separated. The texture also plays an important role in liberation characteristics. Fine grain and fine intergrowth texture indicates fine liberation size which creates a problem in separation. Hence, the knowledge about mineralogical attributes of the ore, i.e., nature of the mineral, mineral size and texture, is very important for any physical beneficiation process (O'Connor and Dahlin, 1991; Jena et al., 2016). The ore mineralogy plays a critical role in the selection of suitable beneficiation technique(s) and also dictates the process flow sheet or plant flow sheet optimization (Petruk and Hughson, 1977; Jena et al., 2016). This is more important while treating low grade iron ores/ fines during physical beneficiation method due to their fine intercalation and dissemination within clay/silicate matrices (Jena et al., 2015).

The alumina bearing minerals, kaolinite and gibbsite, are fine grained and occur in intimate association with goethite and hematite. The clay content in the present sample is high and is more in finer fraction of below 150 ìm. The mineralogical and other characterization studies including XRD, SEM-EDX, FTIR, TGA and EPMA reported that the liberation size is below 150 ìm. Hence the sample may be ground to below 150 ìm size prior to beneficiation. During grinding lot of fines/ultra-fines may be generated due to high clay/silicate minerals and also there is a chance of clay coating on the valuables, which can be minimized by adding minor amount of suitable dispersant during grinding. Scrubbing may be applied as a pre-concentration method for this sample to remove or deslime the ultra-fine clay particles before subjecting to any beneficiation technique (Zhang et al., 2014). Alternatively, classification through hydrocyclone may also be applied to deslime the ultra-fines prior to beneficiation (Singh and Mehrotra, 2007). These ultra-fines may be treated separately either by wet high intensity magnetic separation or by froth flotation method to recover the iron values (Pradip, 1994; Yuhua and Jianwei, 2005; Singh and Mehrotra, 2007; Mowla et al., 2008; Das et al., 2010; Jena et al., 2015).

Based on the characteristics reported on this sample, for concentrating the iron values beneficiation techniques such as; spiral concentration, tabling, wet high intensity magnetic separation and froth flotation may be adopted either alone or in combination (Singh and Mehrotra, 2007; Mowla et al., 2008; Das et al., 2010; Mohanty et al., 2011; Dash et al., 2012; Mohanty et al., 2012; Sahoo et al., 2014; Jena et al., 2015).

Initially gravity concentration through spiral and/or shaking table may be tried upon for its beneficiation. Later on, since hematite and goethite minerals are feebly magnetic in nature, wet high intensity magnetic separation may also be an option to recover the iron values from this sample (Singh and Mehrotra, 2007; Roy et al., 2007; Upadhyay, et al., 2009; Roy and Venkatesh, 2009; Upadhyay, et al., 2010; Roy, 2010; Dash et al., 2012). As the prepared sample is less than 150 ìm in size, froth flotation may be adopted to recover as maximum as iron values using suitable reagents. Therefore combination of gravity and magnetic separation/flotation method may be the suitable beneficiation process for treating this type of sample. Based on characterization there are so many authors has adopted different beneficiation methods to upgrade the goethitic ore, the results of literature survey are given in Table 4.

CONCLUSIONS

It has been concluded that characterization studies of the Barsua iron ore fines mainly composed of iron oxide-hydroxide phases in different proportions with varying amounts of hydrated aluminium hydroxide and silicate/clay minerals. Microscopic studies revealed that fine-grained hematite and goethite are interlocked very intimately and intricately with each other within the clay/silicate matrices showcasing its complex nature. The hydrated aluminium hydroxide is gibbsite along with other clay minerals viz., kaolinite while the major gangue mineral quartz appears as minute grains with variable sizes. XRD analysis reported that the sample is dominated by hematite followed by goethite, gibbsite, kaolinite and quartz. Traces of magnetite are also found in this sample as part of the martitization process.

Microscopic modal analysis of the sample indicated that presence of hematite and goethite minerals are more with higher clay mineral contents. The percentage of interlocked particles is in the range of 18-24%. Gibbsite is reported in interlocked particles. Liberation studies reported around 85% liberation may be possible below 150 µm size. SEM-EDX, FTIR and TGA results also confirm the presence of iron and aluminium hydroxide phases with minor quartz.

XRF analysis of the sample reported that the sample contains around 57.67% total Fe with 6.29% Al_2O_3, 3.52% SiO_2 and 6.93%

Table 4: Correlation of the present study on iron ore fines sample vis-à-vis other important iron ore/fines/low grade ores

SI No.	1	2	3	4	5	6	7	8	9	10	11	Present study
Nature of the ore	Iron ore slimes of d_{80}=50µm. Assay 57% Fe, 4% SiO_2 8.3% Al_2O_3	Al-rich Indian iron ore slimes	Very low grade ore containing porous and friable oxide and hydroxide of iron along with kaolinite and quartz occurrence of kaolinite along the cavities and weaker mineral plane renders the ore highly fragile and causes high alumina content in the slime	BHQ samples collected from Karnataka and Orissa, with low content of strongly magnetic material i.e. low iron content with very high silica and very low alumina content.	BHQ iron ore samples of very low grade containing only 38-40 wt. % of iron. Hematite and quartz are the major mineral phases. Silicate minerals are major impurities, which are associated as hard banded form with iron. Fine liberation and texture of the ore	BHQ iron ore sample that contains 39.0% Fe (T), 42.5% SiO_2, and 1% Al_2O_3. Average particle size of 50 µm	Three iron formations from. different locations BIF I is represented by magnetite and chert with a little martite, hematite and goethite and occasional presence of pyrite is observed. BIF II is mostly martitised magnetite and quartz. BIF III mostly consists of hematite and jasper (a red variety of quartz)	Very low grade iron ore sample from Barsua iron ore deposits of Eastern India, containing 38.19% Fe, 9.48%SiO2, 19.97 Al2O3 Mainly contains goethite, hematite, kaolinite, gibbsite and quartz. Micro platy hematite, goethite with clay patches are common features in this type of ore.	Low grade BMQ ore from Hospet, Karnataka, India. The sample on an average contains ~ 47% Fe, 34.5% SiO_2, and 1.15% Al_2O_3. Silica is found to be the major impurity present in the sample. Three different phases' viz. magnetite, hematite and quartz observed.	Magnetite concentrate, magnetite and silica are two main minerals. The iron content of the raw material is 62.2%. Very fine with only 4.1% over 74 µm. 79.7% Fe is distributed in 0–37 µm. Most of the Si is distributed in coarse size range.	Iron ore slime sample from the Barsua iron ore mines. Hematite, goethite are the major iron mineral phases, whereas kaolinite constitutes the major part of the gangue mineral phases. Quartz was in the form of a minor silicate phase.	The iron ore fines sample is mainly composed of iron oxide-hydroxide phases in different proportions with varying amounts of hydrated aluminium hydroxide and silicate/ clay minerals with 57.67% total Fe with 6.29% Al_2O_3, 3.52% SiO_2 and 6.93% LOI. fine-grained hematite and goethite are inter-locked very intimately intricately with each other within the clay/ silicate matrices. The hydrated aluminium hydroxide is gibbsite along with other clay minerals viz., kaolinite while the major gangue mineral quartz appears as minute grains with variable sizes.
Beneficiation methods implemented/ employed/ proposed.	Hydro-cyclone to get a product suitable for subsequent use in the sinter mix.	A critical review of the work done so far on the pro-cessing of such ores. Develop-ment of selective reagents capable of accom-plishing hematite-alumina/ gibbsite separation has also been identified.	Excellent rejec-tion of silica and alumina from iron ore slime is achievable using the conventional physical separation. Processes - classifying cyclone, shaking table, wet high intensity magnetic separator, and conventional froth flotation cell.	Samples from Odisha was bene-ficiated using DHIMS followed by WHIMS, and the sample from Karnataka region was beneficiated by hydrocyclone.	Two-stage magnetic separation and floatation using conventional cell have established the excellent rejection of silica from the low grade ore.	Magnetic separation was carried out at different magnetic intensities in a wet high intensity magnetic separator	The study highlights the major minerals and their textural behaviour such as relation between hematite and quartz and their spatial distribution in the ore.	The feasibility of beneficiation of goethite-lateritic ore is explained. Advanced gravity separation techni-ques followed by high intensity magnetic separation is suggested. If this stage also fails to achieve the required grade, froth flotation to remove the gangue could be used as the final concentration stage.	The flotation studies of the ore using oleic acid and dodecyl-amine as the collectors have indicated that the ore is more favour-able to cationic collector in comparison to anionic collector.	Cationic reverse flotation with dispersion method, like scrubbing, to improve the flotation performance to prevent hetero-coagulation of fine magnetite particle and coarse silica particle.	The sample responded positively to hydrocyclone followed by magnetic separation.	Based on the detailed characterization studies conducted on this sample, it may be concluded that effective beneficiation of the sample may be possible at below 150 ìm size using gravity, magnetic and flotation based techniques either alone or in combination towards up-gradation of iron values
References	Das et al., (1992)	Pradip, (1994)	Roy et al., (2008)	Anupam et al., (2010)	Das et al., (2010)	Mohanty et al., (2011)	Mohanty et al., (2012)	Nayak, (2014)	Sahoo et al., (2014)	Zhang et al., (2014)	Jena et al., (2015)	Studied area

LOI. The alumina and LOI content is more due to the presence of gibbsite, kaolinite and goethite.

The size and size wise chemical analysis results revealed that the 80% passing size of this sample is 1.9 mm and the grade, in terms of Fe content, is increasing towards finer sizes whereas the silica and alumina contents are less in the finer fractions. The Fe content in below 150 µm size fractions is around 60.0%. It also confirms that most of the irons bearing minerals present in this sample are liberated below 150 ìm size.

Based on the detailed characterization studies, it may be concluded that effective beneficiation of the sample may be possible at below 150 µm size using gravity, magnetic and flotation based techniques either alone or in combination towards up-gradation of iron values for further utilisation in metallurgical industries.

Acknowledgements: The authors are thankful to the Director, Indian Institute of Technology (Indian School of Mines), Dhanbad for the facilities; IIT (ISM) Central Research Facility, for Electron Probe Microanalysis, FTIR and SEM analysis. The authors gratefully acknowledge Dr. S. K. Pan, RDCIS-Ranchi for providing iron ore fines sample. Author would also like to acknowledge the technical support extended by Dr. M. K. Mohanta, CSIR-NML, Jamshedpur, for liberation, microscopic and modal analysis and Dr. M. S. Jena, CSIR-IMMT, Bhubaneswar for XRD analysis. SP would like to thank his colleagues Ms. Mousumi Gharai, Mr. Laxmikanta Sahu and Mr. Rahul Mukherjee for their support during this work. All technical staff of Fuel and Mineral Engineering Department of IIT (ISM) are thankfully acknowledged for their help during the sample preparation and chemical analysis. SP acknowledges the financial support received from IIT (ISM), Dhanbad for his research fellowship. The authors gratefully acknowledge the constructive suggestions of Prof B.V. Rao. We thank the reviewers for their comments.

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(Received: 27 December 2017; Revised form accepted: 2 June 2018)