Chapter 3 Mineralogical Studies and Beneficiation of Low-grade Iron Ores

This chapter deals with the mineralogical studies and beneficiation of low-grade iron ores which include characterization of raw materials (i.e. iron ores, coke, charcoal), sieve analysis of raw materials, proximate analysis of coal, chemical analysis of ore etc. Beneficiation methods, an attempt was made to find a suitable upgradation technique for an increase in total iron percentage; hence try to develop a proper beneficiation methods for iron ore.

3.1 Procurement of raw materials

The sources of raw materials were as followed:

Iron ore	:	*(i)* Bonai ranges of Badjamada Mines, Jharkhand-Odisha sector (Monet Ispat Ltd.) *[Sample – O]* (ii) Bhilwara – Rajasthan state (Red and Yellow ochre – weathered) *[Sample – R-1 and R-2]*
Coke	:	procured from local market of Rajkot
Charcoal	:	procured from local market of Vadodara
Binder	:	Polyvinyl alcohol (PVA) - procured from local market of Vadodara
Flux	:	Lime - procured from local market of Vadodara
Charge material for smelting reduction	:	TMT steel bar – available at Metallurgical and Materials Engineering Department, M. S. University of Baroda

3.2 Mineralogical and Characterization Studies

Following analysis / test of raw materials were carried out in sequence.

- Megascopic studies of ores
- Hardness of ore by Mohs scale
- Density of ore

- Ore microscopy – *Phase identification, texture, Size measurement*.
  a) Polarized microscope
  b) Reflected (Stereo and Conventional)
- Phase identification (by XRD)
- SEM & EDS Analysis - Surface morphology and topography.
- Chemical analysis of iron ore (by XRF)
- Proximate analysis of coal/coke

3.2.1 Megascopic studies of ores

Three samples of iron ore were collected from two different regions of India. Sample-O were received from Bonai ranges, Odisha which is having a size range of 10-50 mm. Rajasthan – 1 (R-1) & Rajasthan – 2 (R-2) samples were collected from two different mines of Bhilwara district, Rajasthan is associated with the Late Archean Hindoli group in Rajasthan state, India. Size range of Rajasthan ores is having a size range of 80-150 mm. For mineralogical studies, an as-received run of mine (ROM) ore samples were taken. As-received samples from different locations are shown in Figure 3.1.

Odisha (O) Rajasthan-1 (R-1) Rajasthan-2 (R-2)

Figure 3.1: As received ROM iron ore sample (O) Banded iron ore in pebble shape and sample (R-1 and 2) as rock ore

The low-grade iron ore from Odisha is brownish in colour, metallic lustre and hard and compact in nature. The bands are observed of hematite and quartz i.e. banded hematite quartz (BHQ). Ores are procured from Rajasthan, basically limonite type of ore which are basically weathered ore in nature. Limonite ore is mainly a goethite iron bearing mineral which a weathering product is obtained in oxygenated environments.[121] These types of ore are hard in nature although it generates huge amounts of fines during mining, mineral processing as well as handling operations. Lumps of R – 1 have a brownish colour. The streak colour is also the same in colour. These lumps are less hard as compared to R– 2. Lumps of R – 2 have yellowish brown streaks and cleavages in one direction. Vast amounts of fines are generated during aberrational contact with the hard surface. The lump is hard but less compact in nature. Lumps have enormous amount of surface porosities.

3.2.2 Hardness of ore by Mohs scale

Hardness is a mineral's resistance to abrasion or scratching. Relative hardness (H) is determined by trying to scratch the surface of one mineral with an edge or corner of a second mineral. If a scratch or abrasion results, the first mineral is softer. Indenting tool on the surface also gives the value of hardness which is as same as the scratch test. Relative hardness can be determined by conducting scratch tests to compare the hardness of an unknown mineral to the minerals in the Mohs hardness scale alternatively, good approximations of hardness may be made by comparing mineral hardness to a fingernail, penny, pocketknife, glass or several other tools.[121]

Measured values of the relative hardness of three samples i.e. sample O, R-1 and R-2 are obtained in the range of 3 - 4. Usually, the hardness value of iron ore recorded in between 5 – 6. Low values of hardness are observed in low-grade iron ore compared to high grade iron ore because of higher quantities of gangue minerals present, irregular size distribution of minerals, and high amount of porosity.

3.2.3 Density of ore

Density of ore was measured by Archimedes' principle.

Procedure:

- Moisture free samples were taken. A thin layer of silicon grease was applied on the surface to make the sample impervious to water.
- Weight of the sample in air (w_1, gm) was taken using physical balance.
- The sample was tied with a thin wire and an arrangement was made to take the weight of the sample submerged in water (w_2, gm)
- Apparent weight loss of sample [(w_1 - w_2) gm] was found and the density of sample was calculated as follows:

$$\text{Density } (\rho) = \frac{\text{weight of the sample in air } (w_1)}{\text{weight of the sample submerged in water } (w_1 - w_2)} \quad \text{...........(3.1)}$$

Table 3.1: Density of low grade iron ores

Sample	**Density (gm/cc)**
Odisha (O)	3.61
Rajasthan (R-1)	2.80
Rajasthan (R-2)	2.47

Table 3.1 shows the result of density measurement for low grade iron ore samples. Density of iron ores is recorded in between 2.5 to 3.6 gm/cc.

3.2.4 Microscopic observation

Raw materials were thoroughly observed with the help of:

a) Polarized microscope,
b) Reflected microscope,

a) Polarized microscope:

Polarized microscopy is generally carried out by a geological microscope. A polarized micrograph is developed by changing the place of the prism like crossed Nichol and plane polarized. Transparent phases can be identified easily.[122] Thin slides are prepared for samples and observed under the microscope at Geology Department, M. S. University of Baroda. One of the sample slides is reported in Figure 3.2.

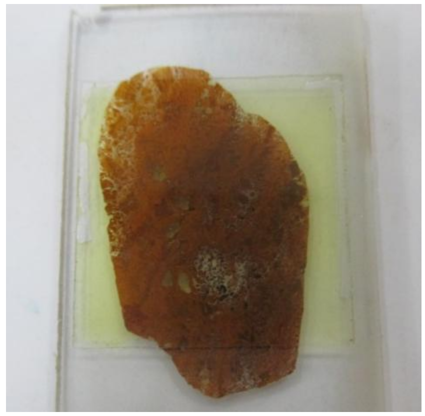
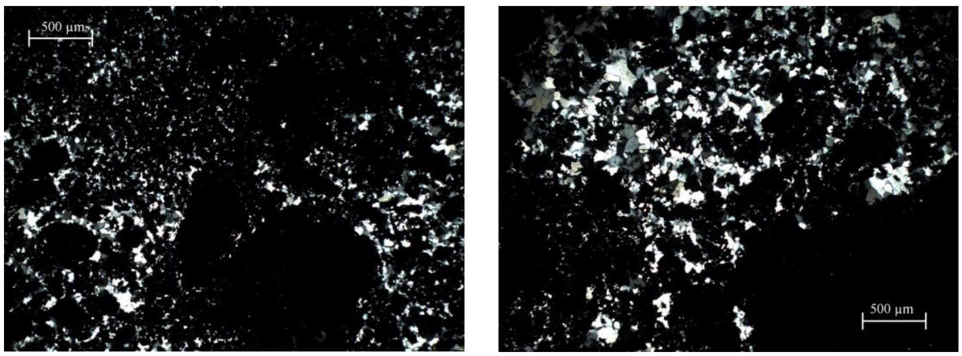


Figure 3.2: Prepared sample slide for polarized microscopy observation

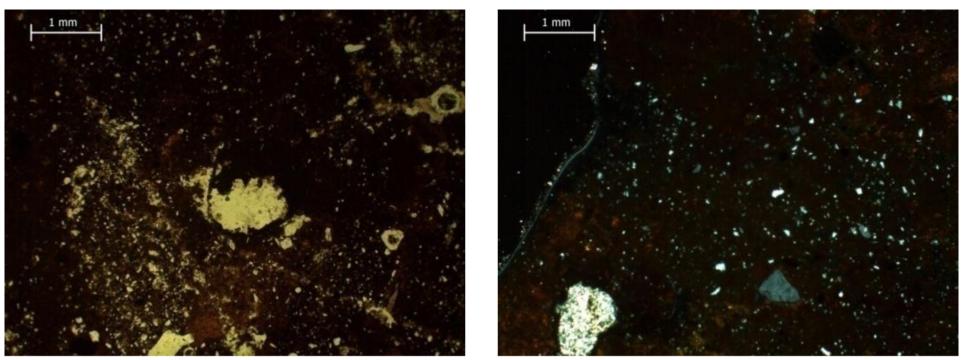
Photographs by polarized microscopes are reported in Figure 3.3. Sample-O shows a fair amount of hematite phase (black portion) in larger grain size and shape. Hematite phase was observed as coarse grain (~300 μm) and fine grain (100-150 μm) as a major phase. Fine grain size hematite is interlocked equigranular with goethite and quartz (50-75 μm) as minor phases. Admixture of fine grains of quartz (whitish portion) and hematite grains is also observed at micron level. The shape of grains is mostly irregular. Fairly easy liberation of phases is expected due to angular interlocking.[2,3]

Sample R-1 shows a quartz phase in whitish colour and goethite in brown colour plane polarized; whereas in crossed Nichol, it becomes brighter than other phases. Goethite observed as a major phase (70-100 μm), quartz and kaolinite as minor phases (20-50 μm). Minor phases are disseminated in major phases. Complete liberation is difficult or impossible due to fine sized minor phases dispersed with major phases. It has inconsistency in the distribution of the hematite phase and is greatly locked with the gangue phases. Tiny gangue mineral phases were interlocked around the surface of ore mineral phases. A clear distinguish of goethite was problematic due to the blackish colour in nature. It consists approximately of 60 to 70 pct of goethite ore minerals by area of its distribution. Almost all ore minerals accommodate the various size and shapes of gangue minerals.[2,4,5]

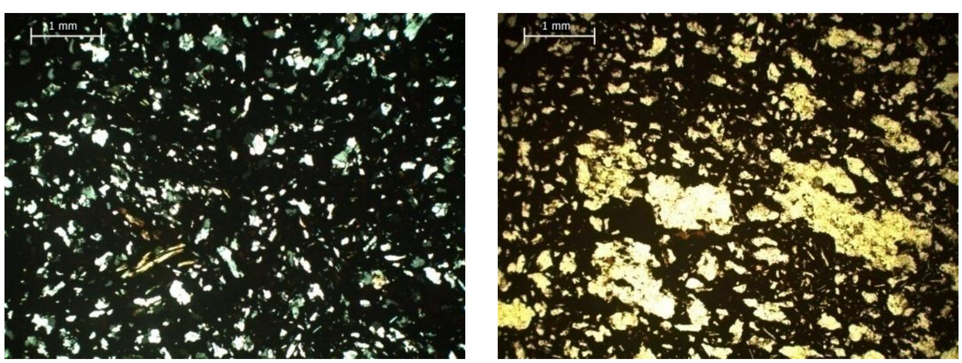
Sample R-2 shows goethite (in dark reddish brown) and quartz (in bright whitish to yellowish) phases are compounded in ore. Ore minerals are coated or enveloped of gangue minerals like quartz, kaolinite and calcite by layer. Gangue phases such as quartz and kaolinite are finely dispersed phases. The size of goethite phase varies from 100-200 μm; whereas quartz has huge size range from 100-500 μm. Liberation of ore minerals is difficult due to fine size of gangue minerals; it's layered on ore mineral phases.[1, 2, 5]



Sample O



Sample R-1



Sample R-2

Figure 3.3: Photo micrographs of iron ores by polarized microscopes (100X); phases (colour) - hematite (black), goethite (brown) and quartz (white)

b) Reflected microscope:

A reflected microscope is used to observe surface morphology and topography at low magnification. Stereo and conventional reflected microscopes were used for observations. Samples were prepared by the standard procedure which is quite similar to metallography except etching operation. Samples were observed under the microscope (Olympus) at Electrical Research and Development Association, Vadodara.

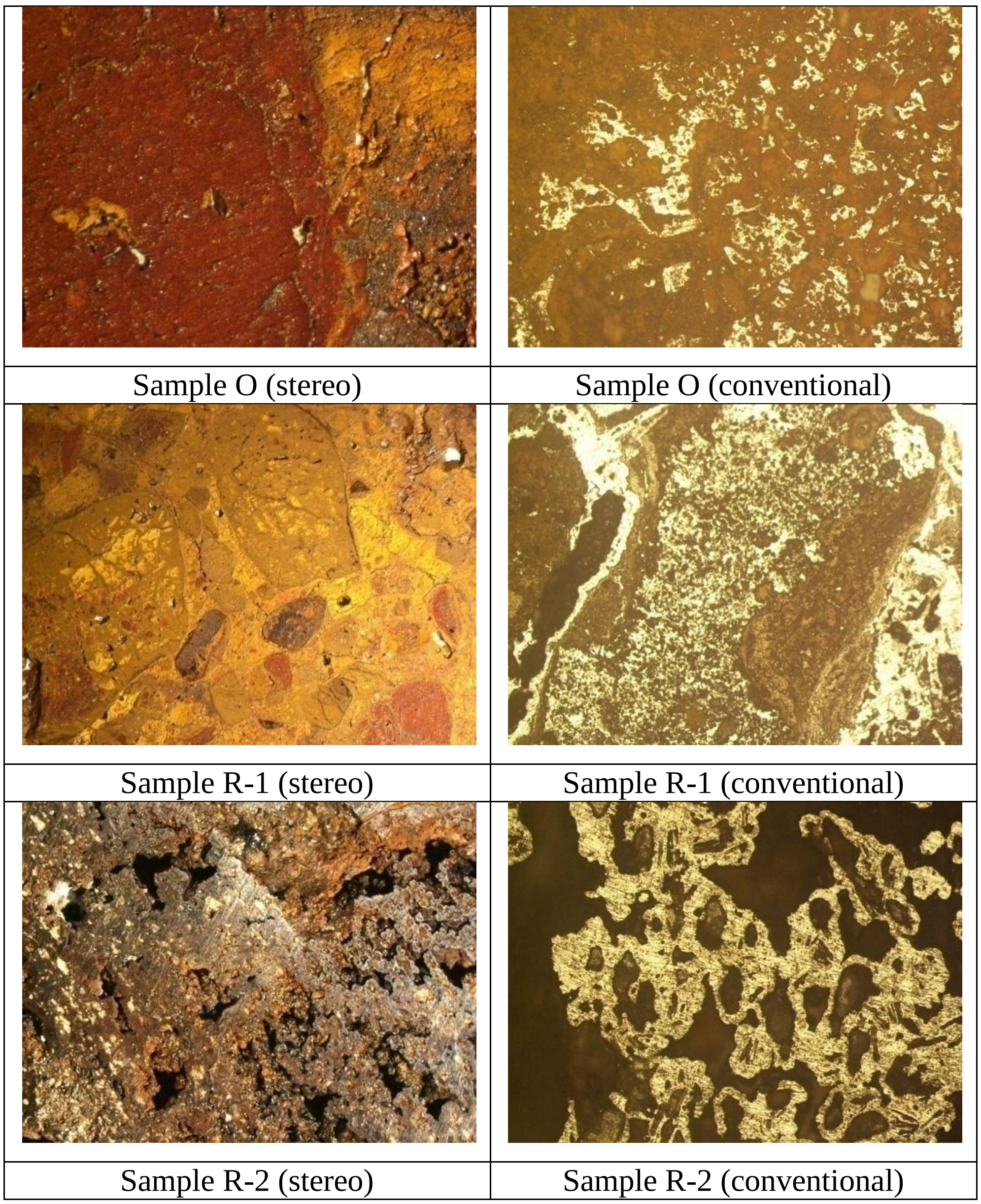


Figure 3.4: Photo micrographs of iron ores by reflected (stereo and conventional) microscopes (Left-10X and Right-50X); iron bearing minerals interlocked with fine sized gangue minerals

Photographs by stereo and conventional microscopes are reported in Figure 3.4. For Sample O, cleavages and different phases on the basis of colour are clearly observed in stereo microscope photographs while quartz (bright white phase) can be easily distinguished by its colour contrast in conventional microscope.[125] Sample R-1 is observed with cracks and porosities clearly at surface level in stereo photograph whereas a large quantity of goethite is surrounded by quartz and other gangue phases in a conventional microscope. For sample R-2, different phases are interlocked which is observed in a stereo photograph while a mixture of goethite, quartz (white phase) and kaolinite are unevenly distributed at the micro level which is observed in a conventional microscope.

3.2.5 Phase identification (by XRD)

An X-Ray Diffraction (XRD) analysis is used for the identification of the phase present in samples. Samples of Odisha (O) and Rajasthan ores (R-1 and R-2) were taken for XRD analysis. XRD Diffractometer (Model: X'pert Pro, Make: PANalytical) is available at Metallurgical and Material Engineering Department, M. S. University of Baroda. XRD graphs of three samples are mentioned in Figure 3.5, Figure 3.6 and Figure 3.7 respectively. Phase identification data are reported in Table 3.2, Table 3.3 and Table 3.4 respectively.

Sample-O has major phases of hematite and minor phases of quartz and kaolinite. Sample R-1 and R-2 have goethite and quartz as major phases while kaolinite, calcite and rutile are minor phases. The major peak of both samples has a quartz phase. Sample-O and R-2 have a prominent amount of rutile phase.

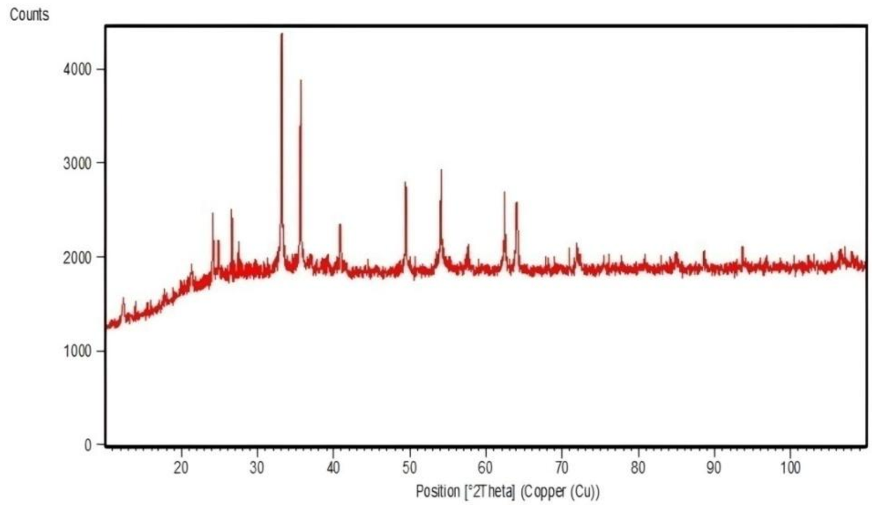


Figure 3.5: XRD graph of Odisha ore (sample-O)

Table 3.2: XRD analysis of Odisha ore (sample-O)

2θ	**d spacing (Å)**	**I/I_0 (pct)**	**Phases present**
24.1427	3.68335	31.65	Hematite
24.8638	3.57814	22.67	Quartz
33.1263	2.70212	100.00	Hematite
35.6029	2.51967	76.05	Hematite
36.8754	2.43555	9.07	Kaolinite
40.8157	2.20906	19.55	Goethite
49.4162	1.84984	33.76	Hematite
54.5702	1.69610	37.80	Quartz
57.5702	1.59970	9.28	Quartz
62.4390	1.48615	29.23	Hematite
63.9422	1.45475	26.42	Hematite
71.8751	1.31248	10.04	Rutile
88.6076	1.10285	4.75	Pyrrohotite
93.8213	1.05479	3.87	Pyrrohotite

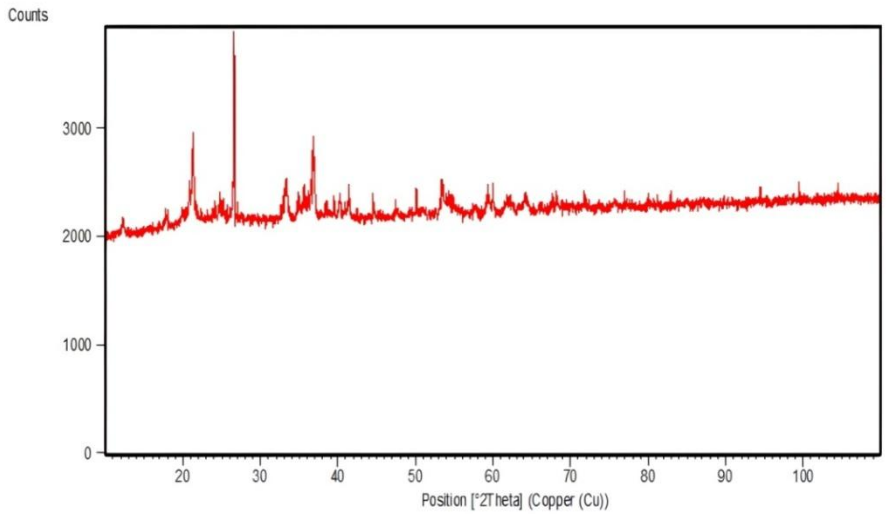


Figure 3.6: XRD graph of Rajasthan ore (R-1)

Table 3.3: XRD analysis of Rajasthan ore (R-1)

2θ	**d spacing(Å)**	**I/I_0 (pct)**	**Phases present**
17.7669	4.98818	6.51	Goethite
20.8570	4.25559	23.65	Quartz
26.5985	3.34859	100.00	Quartz
33.4083	2.67995	24.13	Goethite
35.6336	2.51752	16.09	Calcite
36.8254	2.43874	41.11	Rutile
40.2778	2.23731	10.79	Goethite
41.3916	2.17964	12.16	Goethite
44.5277	2.03314	7.75	Rutile
53.4546	1.71275	16.95	Goethite
59.2975	1.55716	9.71	Chalcopyrite
61.9215	1.49732	5.49	Rutile
68.1668	1.37455	5.17	Quartz
71.8726	1.31252	4.37	Magnesite

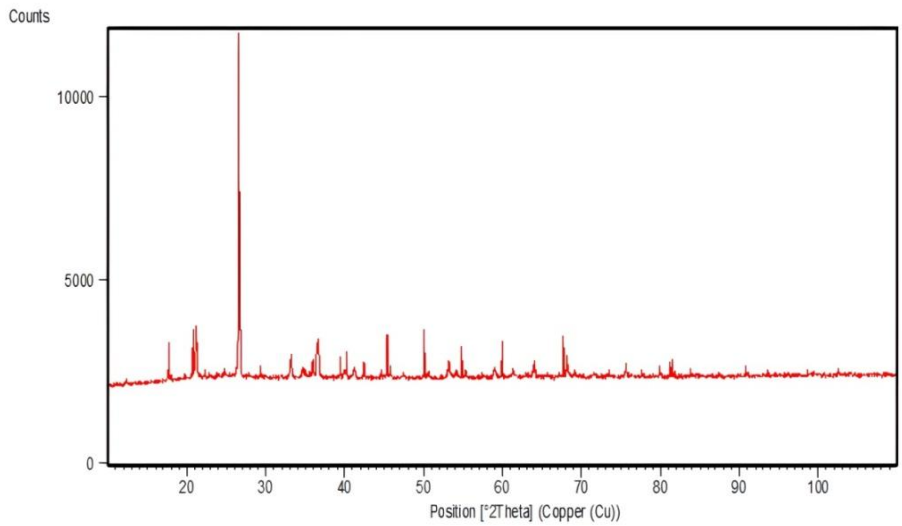


Figure 3.7: XRD graph of Rajasthan ore (R-2)

Table 3.4: XRD analysis of Rajasthan ore (R-2)

2θ	**d spacing (Å)**	**I/I_0(pct)**	**Phases present**
17.7417	4.99520	12.33	Goethite
20.7382	4.27971	11.14	Kaolinite
20.8150	4.26408	15.29	Quartz / Kaolinite
26.5819	3.35064	100.00	Quartz
26.7597	3.32878	28.83	Pyrite
29.3635	3.03925	3.39	Calcite
33.1960	2.69661	7.51	Chalcopyrite
36.4800	2.46103	8.78	Quartz
36.5883	2.45400	13.29	Kaolinite
39.4011	2.28505	5.39	Rutile
40.2231	2.24023	7.26	Goethite
45.3585	1.99781	11.14	Pyrhotite
50.1127	1.81885	12.18	Quartz /Kaolinite
53.1773	1.72103	4.78	Magnetite
54.8392	1.67273	8.06	Calcite
59.0392	1.56336	2.86	Calcite
59.9255	1.54234	9.32	Quartz
63.9999	1.45362	4.25	Rutile
67.7069	1.38277	10.20	Quartz
68.1182	1.37542	5.07	Quartz / rutile

3.2.6 SEM & EDS Analysis

Scanning Electron Microscopy (SEM) and Energy Dispersive Spectroscopy (EDS) are important analytical instruments for surface topography, texture, size analysis and chemical analysis of spot or identified surface area. SEM and EDS analyses were carried out for all three samples (O, R-1 and R-2). A Jeol SEM (Model: JSM—5610 LV) coupled with Oxford EDAX system, is available at Metallurgical and Material Engineering Department, M. S. University of Baroda.

a) SEM analysis

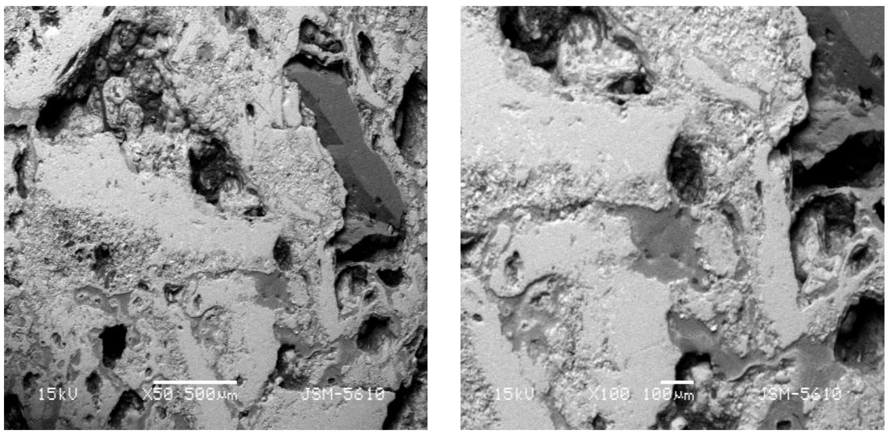


Figure 3.8: SEM photographs of raw iron ore Odisha sample-O at 50X and100X; highly interlocked minerals observed with significant amount of pores

Figure 3.8 shows iron bearing minerals i.e. hematite and goethite in greyish colour grains. Grains are mostly angular to irregular in shape. The blackish mixture is of kaolinite and quartz particles. The particle size of quartz varies from a few microns to 200 μm. An admixture of iron bearing minerals with gangue minerals was also observed at higher magnification. Several Porous areas were significantly present in the lump of ore. Mostly these areas were observed in the gangue minerals region or at the interface of gangue-iron bearing minerals.[123]

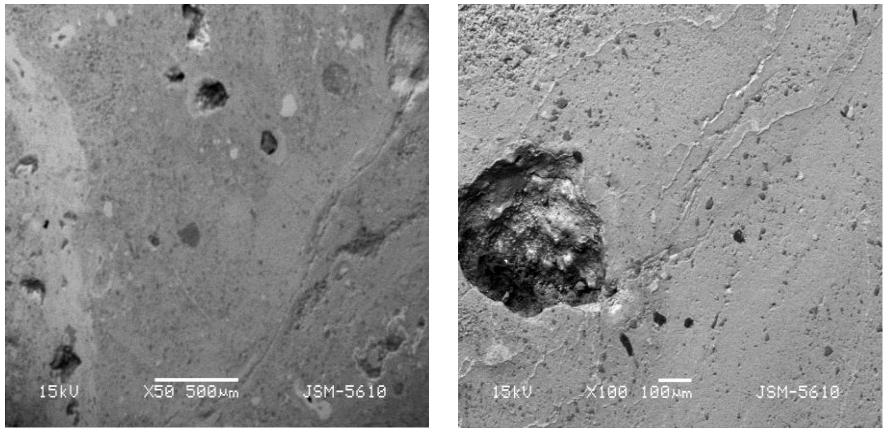


Figure 3.9: SEM photographs of raw iron ore R-1 sample at 50X and100X; micro-cracks and fine particles of gangue mineral revealed at 100X

Figure 3.9 reveals SEM photograph of Rajasthan 1 low grade iron ore at different magnifications. Black spots and particles confirm the iron bearing mineral as a goethite. Blackish spots and bands are of kaolinite and calcium based minerals with the association of Quartz gangue mineral. Micro-cracks were observed in the SEM examination. Open Pore volumes were observed at different locations. This pore volume contains hydrous minerals due to the presence of water molecules. Goethite is a hydrous type of mineral which also confirms its presence.[124]

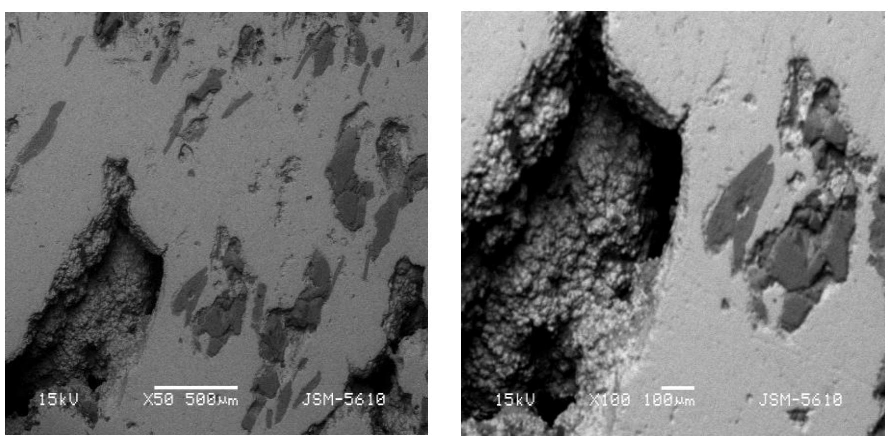
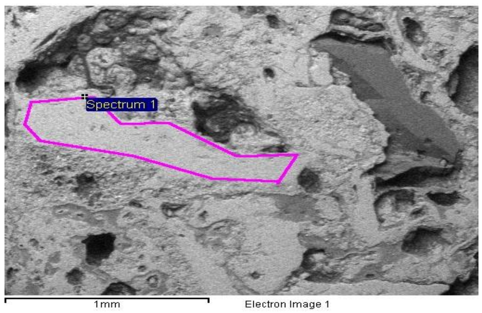


Figure 3.10: SEM photographs of raw iron ore R-2 sample at 50X and100X; quartz (black colour) distributed evenly in ore

Figure 3.10 observed that black grains are goethite minerals, whereas black phases contain huge amounts of tiny particles of silicon, aluminium and calcium based compounds. At the open pores, phases are weathered. This is due to water molecules trapped into the cavities and reacted with phases. Compounds are mostly hydroxide in nature. At 100X, the weathered phases are clearly shown at the open pores.[2,5]

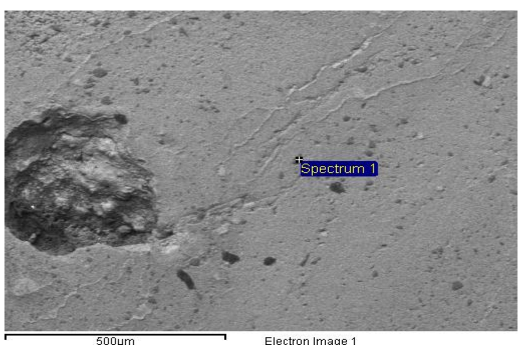
b) EDS analysis



Element	Weight (pct)	Compound (pct)	Formula
Al K	3.55	6.70	Al2O3
Si K	1.75	3.74	SiO2
Fe K	69.62	89.56	Fe2O3
O	25.09		
Total	100.00		

Figure 3.11: EDS analysis of Odisha sample; grey portion indicate iron bearing minerals

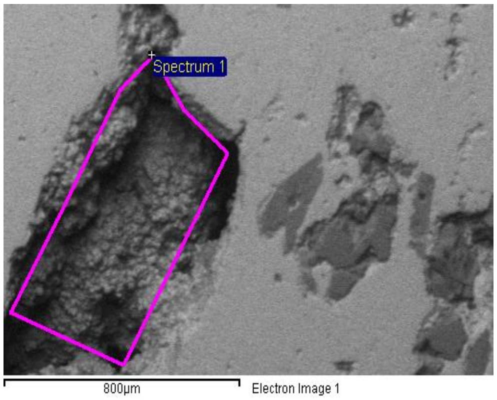
Figure 3.11 shows EDS analysis in the greyish region. This greyish region has a mixture of hematite and kaolinite phases.



Element	Weight (pct)	Compound (pct)	Formula
Al K	1.14	2.15	Al_2O_3
Si K	41.39	88.54	SiO_2
Fe K	7.24	9.31	Fe_2O_3
O	50.24		
Total	100.00		

Figure 3.12: EDS analysis of R-1 sample; black coloured fine sized particle of quartz mineral

Figure 3.12 shows the spot analysis of the R-1 sample. A higher amount of quartz is present at a particular spot.



Element	Weight (pct)	Compound (pct)	Formula
Al K	5.81	10.97	Al_2O_3
Si K	4.92	10.52	SiO_2
Fe K	61.03	78.51	Fe_2O_3
O	28.25		
Total	100.00		

Figure 3.13: EDS analysis of R-2 sample; segregation of minerals in cavity

Figure 3.13 shows the area analysis of a blackish region of the R2 sample. Due to weathering effect porosity is observed. In this region, goethite has a major amount. Quartz and kaolinite are present.

3.2.7 Chemical analysis of iron ore (by XRF)

Table 3.5: Chemical analysis of iron ore (by XRF)

Analyte	**Odisha (pct)**	**Rajasthan-1 (pct)**	**Rajasthan-2 (pct)**
Total Fe, $Fe_{(t)}$	53.55	33.02	34.02
FeO	0.90	0.98	0.81
SiO_2	8.50	24.12	31.27
Al_2O_3	7.30	13.23	7.37
CaO	0.15	0.11	0.35
MgO	0.14	0.17	0.39
MnO	0.24	0.22	0.21
TiO_2	0.42	2.16	0.25
P	0.02	0.058	0.27
S	0.007	0.008	0.01
Na_2O	0.043	0.048	0.051
K_2O	0.021	0.148	1.09
ZnO	0.004	0.035	0.210
LOI	5.31	11.03	8.46

An X-Ray Fluorescence (XRF) spectrometer is an x-ray instrument used for Non-Destructive chemical analysis of rocks, minerals, metals, sediments and fluids. It works on wavelength-dispersive spectroscopic principles. It is quite a functional analytical instrument for the analysis of both homogeneous and heterogeneous materials, especially for samples of unknown chemistry. XRF Spectrometer (Model: EDXRF-800, Make: Shimazdu, Japan) is available at Metallurgical and Material Engineering Department, M. S. University of Baroda. Measurement conditions were: Sample – powder form; Atmosphere – vacuum; Analysis range of elements: C to U. Three different samples of iron ores were analyzed by XRF and reported in Table 3.5.

3.2.8 Proximate analysis of coke and charcoal

Proximate analyses of coke/charcoal were done as per the standard method.[126] It deals with the determination of the following constituents: (a) moisture, (b) volatile matter, (c) ash and (d) fixed carbon. The knowledge of these constituents is useful in the selection of coal and coke for an appropriate function.

Procedure of Proximate analysis:

a) **Determination of Moisture**:

Moisture is determined by heating coal at 383 K in a crucible for one hour. A known amount of the finely powdered coal sample, taken in a silica crucible, is heated in an electric oven at 383 K for one hour. After which the crucible is taken out, cooled in desiccator and weighed.

$$\%\ of\ moisture = \frac{Loss\ in\ weight}{wt.of\ coal\ taken} \times 100$$

b) **Determination of Volatile Matter**:

After moisture determination same silica crucible is taken and covered with a lid. It is then placed in muffle furnace, heated at 1223 K for 7 minute. The crucible is then taken out and cooled first in air, then in a desiccator and weighed again.

$$\%\ of\ volatile\ matter = \frac{Loss\ in\ weight}{wt.of\ coal\ taken} \times 100$$

The volatile matter present in the coal includes H_2, CO methane and other low Hydrocarbons, CO_2 and N_2.

c) **Determination of Ash**:

After volatile matter determination same silica crucible without lid is heated in open crucible at 1123 K in presence of oxygen in air for half an hour in a muffle furnace. The crucible is cooled down in furnace, then take out crucible and weighed of crucible.

$$\% \; of \; ash = \frac{wt. of \; ash \; formed}{wt. of \; coal \; taken} \times 100$$

Ash is non-combustible matter, which is left behind when all the combustible substances have been burnt off.

d) **Determination of Fixed Carbon**:

After the determination of moisture, volatile matter and ash contents; the remaining material is 'Fixed Carbon'.

i.e. *% of Fixed Carbon = 100% - (% of Moisture + % of Volatile Matter + % of Ash)*

The results (average of two samples) of coke and charcoal have been reported in Table 3.6.

Table 3.6: Proximate analysis of coke and charcoal

Analyte	**Coke**	**Charcoal**
Moisture	01.35	02.30
Volatile Matter	07.46	18.21
Ash	22.81	04.12
Fixed Carbon	68.38	75.37
Total	**100.00**	**100.00**

3.3 Beneficiation studies of low-grade iron ore

Two different types of iron ore were selected for beneficiation studies: (i) Hematite - BHQ – Jharkhand – Odisha sector and (ii) Limonite - Rajasthan. For beneficiation studies, iron ores were crushed and ground as per the requirement of beneficiation methods.

3.3.1 Beneficiation of Odisha (O) low-grade iron ore

Raw iron ore from Odisha which is having a size range of 10-50 mm. Ore was crushed by jaw and roll crushers. After the crushing operation, ore size was obtained in the range of -5+2 mm (i.e. -4 +10 mesh). A jigging operation was employed on crushed ore. After the initial beneficiation method, an intermediate upgraded ore (i.e. concentrate product) was further ground in a ball mill. For ball mill operation, small and large sized cast iron balls (20-50 mm in diameter) were kept in a mill. The grinding operation was done at 64 rpm with continuous observation to avoid over-grinding (i.e. fine size grinding). Size analysis of an upgraded ore was done and the result is shown in Table 3.7. A ground ore was subjected to the tabling operation. The concentrate and middling products were mixed for maximum utilisation of ore.

Table 3.7: Sieve analysis of Odisha iron ore (after grinding)

Sieve size B.S. Mesh No.	Sieve size Micron (μm)	Weight retained (gm)	pct weight retained	Cumulative pct weight retained	Cumulative pct weight passed
85	180	30.486	30.49	30.49	69.51
100	150	37.912	37.91	68.40	31.60
120	125	13.361	13.36	81.76	18.24
150	106	12.008	12.01	93.77	6.23
200	75	3.763	3.76	97.53	2.47
pan		2.467	2.47	**100.00**	**0.00**
	Total	**99.997**	**100.00**		

For beneficiation studies, Odisha iron ore is crushed and ground as per the requirement of beneficiation methods. Different sized ore was beneficiated by using various methods like jigging, tabling (wilfley table), air classifier and hydraulic classifier. After beneficiation trials, the two methods were finalised. Washing and scrubbing of ore were applied after each step of comminution and beneficiation operations which helped to remove free gangues on the surface and subsurface. The process route using two beneficiation methods is finalised where jigging was used in the first stage and followed by the tabling operation. The concentrate and middling can be mixed for further utilization. All the tests were carried out at Metallurgical and Materials Engineering Department, M.S. University of Baroda as per standard procedures.

The laboratory sized jig instrument's stroke speed is 120 strokes/min and operation time is 5 min. While wilfley table has a stroke length - 80 mm, a speed of table – 100 strokes/min and an angle of table - 30º.

Recovery of iron from ore is calculated by:

$$Iron\ recovery\ (pct) = \frac{(W_p \times F_p)}{(W_f \times F_f)} \times 100 \text{ ....(3.2)}$$

where, W_f – Weight of feed sample,

W_p - Weight of product after beneficiation

F_f – fraction of $Fe_{(T)}$ in feed sample and

F_p – fraction of $Fe_{(T)}$ in product sample after beneficiation.

Results of jigging and tabling operations were shown in Table 3.8 and Table 3.9 respectively.

Table 3.8: Result of jigging operation for Odisha ore

	Fe (pct)	SiO_2 (pct)	Al_2O_3(pct)	Weight (gm)	Recovery (pct)
Initial	**53.5**	**8.5**	**7.3**	**500**	
Concentrate	59.02	7.05	6.11	294.98	65.57
Middling	49.63	11.8	10.25	132.23	24.72
Tailing	46.12	12.13	11.65	67.1	11.65

Table 3.9:Result of tabling operation for Odisha ore

	Fe (pct)	SiO_2 (pct)	Al_2O_3 (pct)	Weight (gm)	Recovery (pct)
Initial	59.02	7.05	6.11	200	
Concentrate	62.13	5.04	3.30	106.13	55.86
Middling	58.39	8.51	4.09	57.70	28.54
Tailing	51.25	12.241	3.717	28.06	12.18

For maximum outcome of iron recovery from ore is calculated by:

$$Iron\ recovery\ (concentrate + middling)\ (pct) = \frac{(W_C \times Fe_c + W_m \times Fe_m)}{(W_C + W_m)} \times 100 \quad .....(3.3)$$

where, W_c – Weight of concentrate,

W_m - Weight of middling

Fe_c – fraction of $Fe_{(T)}$ in concentrate and

Fe_m – fraction of $Fe_{(T)}$ in middling.

The flow sheet of two stage beneficiation of Odisha ore is shown in Figure 3.14. The recovery of iron by each beneficiation operation's result is shown in Table 3.10 and Figure 3.15. The Jigging operation gave better recovery of iron from iron ore. Concentrate of jigging operation is considered for the next beneficiation operation i.e. tabling operation. Furthermore, the concentrate and middling product of tabling operation observed comparatively higher iron recovery with respect to concentrate product of tabling operation. So, the recovery of iron is calculated by equations 3.2 and 3.3. Overall beneficiation of iron ore is shown in Table 3.10. Overall these two operations are selected in sequence for beneficiation of Odisha iron ore.

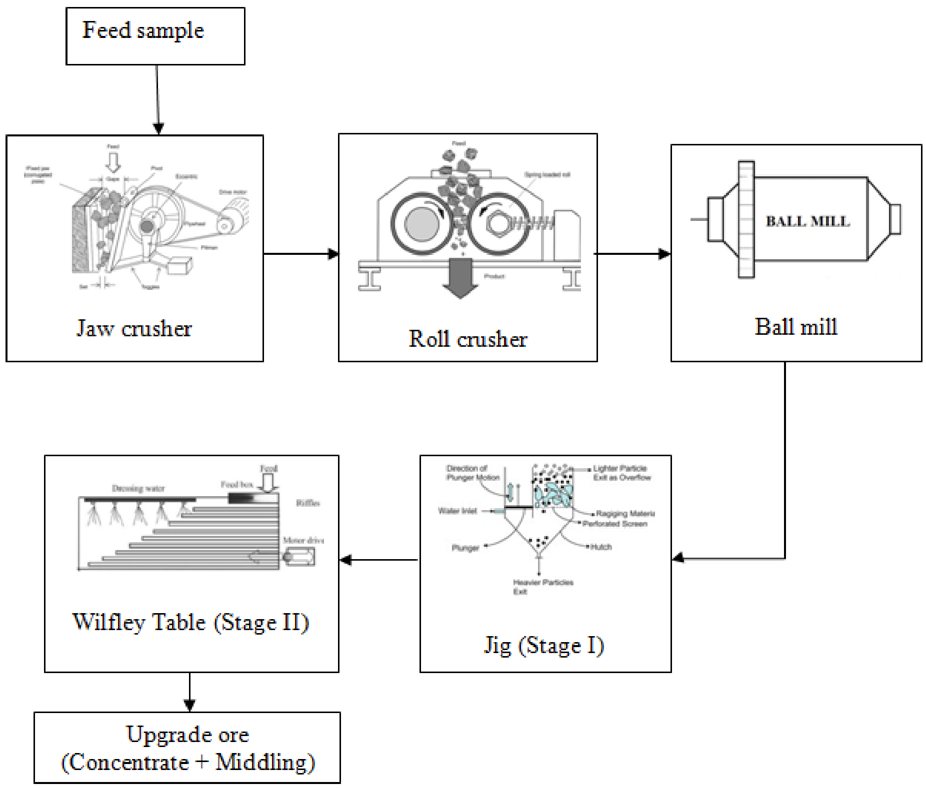


Figure 3.14: Flow sheet of two stage beneficiation for Odisha ore

Table 3.10: Result of Beneficiation of Odisha iron ore

Operation	**Initial weight (gm)**	**pct Fe**	**SiO_2(pct)**	**Al_2O_3 (pct)**	**Weight retained (gm)**	**Recovery (pct)**
Initial		**53.1**	**8.5**	**7.3**		**0**
Jigging	500	59.02	7.05	6.11	294.98	65.57
Tabling (Concentrate)	200	62.13	5.04	3.30	106.13	55.86
Table (Middling)		58.39	8.50	4.09	57.70	28.54
Final output i.e. Tabling (Conc +Middle)	200	60.26	6.76	3.69	163.83	59.75

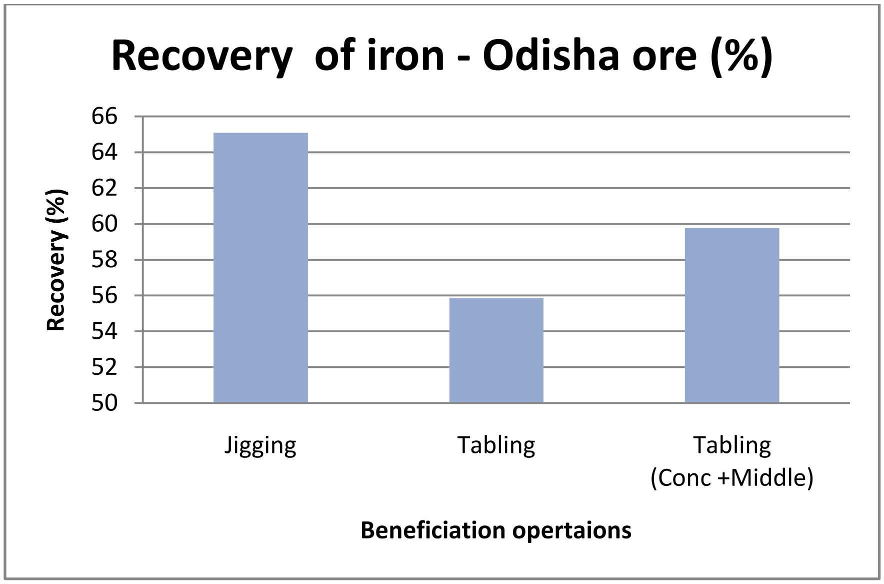


Figure 3.15: Iron recovery by beneficiation operations

3.3.2 Beneficiation of Rajasthan (R) low-grade iron ore

For beneficiation studies, Rajasthan iron ore is crushed and ground as per the requirement of beneficiation methods. Raw iron ore from Rajasthan which is having a size range of 8-15 cm. Ore was crushed by jaw and roll crushing operation. Due to the weathered nature of ore, rock is tough to break but a notable amount (~3 pct) of fines is generated during crushing operations. After the crushing operation, ore size was obtained in a range of -5+2 mm (i.e. -4 +10 mesh). Different sized ore was beneficiated using various methods like jigging, tabling (wilfley table), and classifiers. Satisfactory results were not achieved in the conventional beneficiation operations. Crushed ore is further ground in a ball mill for magnetic roasting studies. Small and large sized cast iron balls (20-50 mm in diameter) were kept in a mill. The grinding operation was done at 64 rpm with continuous observation. Sieve analysis of a ground ore is shown in Table 3.11. Mineralogical studies indicated ore minerals interlocked with fine gangue minerals. So, ore is subjected to fine ground for beneficiation. Rajasthan ore is less compact in nature so it does not require intensive grinding. Due to the presence of hydroxides in ore, it has a sticking nature on the internal surface of ball mill and on the balls' surface.

Table 3.11: Sieve analysis of Rajasthan iron ore (after grinding)

Sieve size B.S. Mesh No.	**Sieve size Micron (μm)**	**Weight retained (gm)**	**pct weight retained**	**Cumulative pct weight retained**	**Cumulative pct weight passed**
85		13.994	14.03	14.03	85.97
100	150	7.752	7.77	21.81	78.19
120	125	8.587	8.61	30.42	69.58
150	106	11.385	11.42	41.84	58.16
200	75	14.716	14.76	56.60	43.40
300	53	18.96	19.01	75.61	24.39
350	45	4.721	4.73	80.35	19.65
Pan		19.598	19.65	**100.00**	**0.00**
	Total	**99.713**	**100.00**		

As per the literature survey, magnetic roasting is quite encouraging for low to lean grade iron ores which have a complex association of gangues with ore minerals. So, a magnetic roasting process was applied to upgrade a lean grade ore. Several trials were taken with different parameters (particle size of ore, time and temperature). Magnetic roasting was carried out with two different carbonaceous materials - coke fines and charcoal fines as per stoichiometric reactions. The roasted ore was subjected to wet magnetic separation for beneficiation.

Proximate analyses of coke and charcoal are shown in Table 3.6. Fixed carbon of coke and charcoal are 68.38 pct and 75.37 pct respectively. Coke has high ash content and low volatile matter while charcoal has low ash content and high volatile matter. For magnetic roasting studies, coke and charcoal were ground. Sieve analysis of coke and charcoal are shown in Table 3.12 and Table 3.13.

Table 3.12: Sieve analysis of coke (after grinding)

Sieve size B.S. Mesh No.	**Sieve size Micron (μm)**	**Weight retained (gm)**	**pct weight retained**	**Cumulative pct weight retained**	**Cumulative pct weight passed**
85		40.969	40.97	40.97	59.03
100	150	15.141	15.14	56.12	43.88
120	125	9.598	9.60	65.72	34.28
150	106	9.647	9.65	75.36	24.64
200	75	8.289	8.29	83.65	16.35
300	53	6.156	6.16	89.81	10.19
350	45	3.194	3.19	93.00	7.00
Pan		6.995	7.00	**100.00**	**0.00**
	Total	**99.989**	**100.00**		

Table 3.13: Sieve analysis of charcoal (after grinding)

Sieve size B.S. Mesh No.	**Sieve size Micron (μm)**	**Weight retained (gm)**	**pct weight retained**	**Cumulative pct weight retained**	**Cumulative pct weight passed**
85		5.150	5.15	5.15	94.85
100	150	16.150	16.15	21.30	78.70
120	125	20.980	20.98	42.28	57.72
150	106	25.530	25.53	67.82	32.18
200	75	16.890	16.89	84.71	15.29
300	53	5.460	5.46	90.17	9.83
350	45	2.290	2.29	92.46	7.54
Pan		7.540	7.54	100.00	0.00
	Total	**99.99**	100.00		

Magnetic roasting process: ore and carbonaceous material (coke or charcoal) were mixed as per stoichiometry and put in an alumina crucible for different temperatures and times in a muffle furnace. During this process, additional coke or charcoal was placed on the top and beside a crucible to maintain reducing atmosphere in the furnace. Roasted ore was slowly cooled down in the furnace.

Initially, iron ore roasted by coke at 873 K and 1173 K for 60 min. Roasted ore characterized by XRD technique for phase identification and stoichiometric calculation. Graphs are shown in Figure 3.16 and Figure 3.17. Identified phases are reported in Table 3.14 and Table 3.15. At 873 K, goethite is partially transformed into maghemite (γ–Fe_2O_3) phase (*magnetic in nature*) and hematite. Untransformed goethite is also found out during analysis. At 1173 K, goethite is transformed majorly in the maghemite phase. The remaining goethite phase is untransformed at 1173 K also. Roasted ore at 1173 K had achieved a better magnetic nature due to a maghemite phase present which will be helpful in the enrichment of iron ore by magnetic separation.

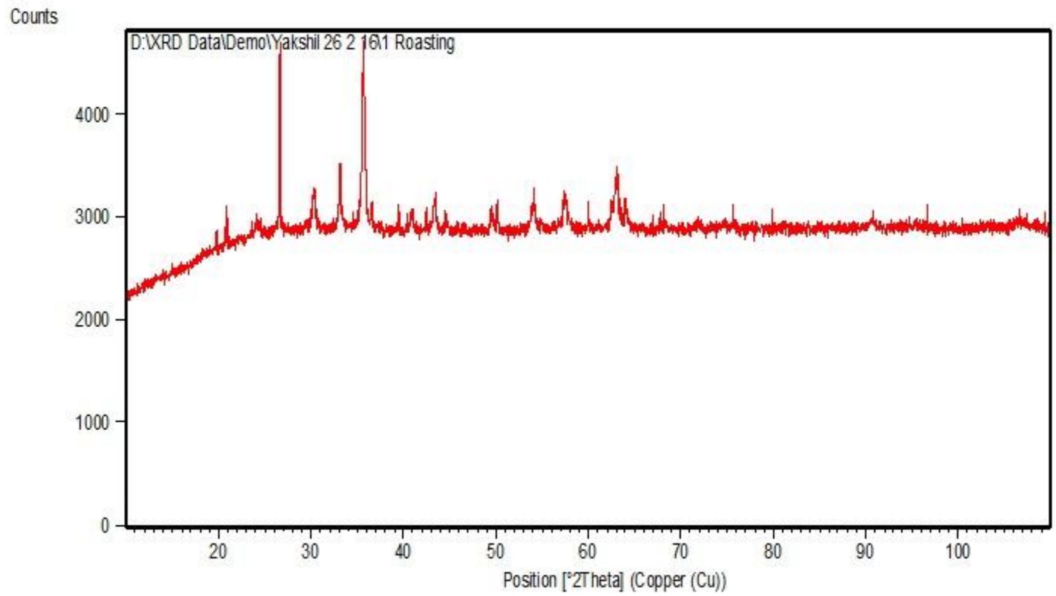


Figure 3.16: XRD graph for roasted ore at 873 K

Table 3.14: XRD analysis of roasted ore at 873 K

2θ	**d spacing(Å)**	**I/I_0 (pct)**	**Phases present**
20.8597	4.25505	25.50	Quartz
24.1501	3.68225	12.86	Hematite
26.6171	3.34629	90.14	Quartz
30.3634	2.94141	22.28	Maghemite
33.1725	2.69846	36.39	Goethite
35.6810	2.51429	100.00	Maghemite
36.5259	2.45805	11.03	Quartz / Kaolinite
39.4726	2.28107	9.47	Quartz / Kaolinite
40.8608	2.20672	9.39	Goethite
43.3965	2.08347	18.98	Maghemite
44.5817	2.03080	5.81	Rutile
49.4548	1.84149	9.17	Hematite
50.1518	1.81752	8.77	Quartz / Kaolinite
54.0972	1.69391	17.58	Quartz
57.4465	1.60285	17.17	Maghemite
59.9880	1.54088	4.81	Quartz
63.0949	1.47227	29.44	Maghemite
63.9304	1.45503	12.05	Hematite
68.0613	1.37643	4.64	Quartz
71.9507	1.31129	3.40	Magnesite
90.5974	1.08373	4.74	Maghemite

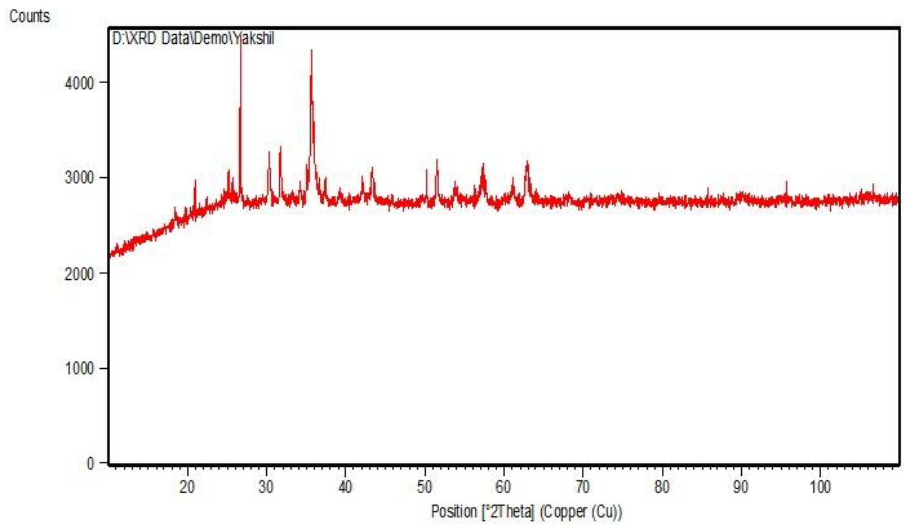


Figure 3.17: XRD graph for roasted ore at 1173 K

Table 3.15: XRD analysis of roasted ore at 1173 K

2θ	**d spacing(Å)**	**I/I_0 (pct)**	**Phases present**
20.9046	4.24602	23.94	Quartz
25.1339	3.54029	24.30	Quartz
26.6902	3.33730	92.55	Quartz
30.2880	2.94856	32.77	Maghemite
31.6902	2.82122	31.80	Alumohydrocalcite
34.1530	2.62320	10.80	Chalcopyrite
35.0353	2.55914	22.53	Kaolinite
35.6543	2.51611	100.00	Maghemite
35.9901	2.49340	47.02	Goethite
37.4016	2.40248	12.57	Quartz / Kaolinite
39.2143	2.29550	5.35	Rutile
42.1150	2.14386	13.39	Goethite
43.3275	2.08663	19.87	Maghemite
50.1596	1.81726	15.50	Quartz
51.4481	1.77473	22.33	Magnetite
53.9608	1.69787	8.94	Maghemite
57.2555	1.60775	20.59	Maghemite
61.0163	1.51735	9.22	Quartz
62.9567	1.47517	25.33	Maghemite
89.9104	1.09022	4.54	Maghemite

Stoichiometric Calculation of magnetic roasting:

On the basis of XRD analysis of roasted ore; the following reaction took place in the process:

$$2\ FeO(OH) + C \rightarrow Fe_2O_3 + CO_2 + H_2O$$

Wt. (gm) 2*(89) = 178 12

For the roasting of 178 gm of FeO(OH), carbon required = 12 gm.

∴ For the roasting of 100 gm of FeO(OH), carbon required $= \frac{100 \times 12}{178}$

Stiochiometric carbon required (S_C) = 6.74 gm

From proximate analysis of coke, fixed carbon is 68.38 gm in 100 gm of coal.

For the 68.38 gm of Carbon, Coke required = 100 gm.

∴ For the 6.74 gm of Carbon, Coal required $= \frac{6.74 \times 100}{68.38}$

Coal required = 9.86 gm

From Chemical analysis of Iron ore, $Fe_{(T)}$ = 33.02pct in ore.

For the 33.02 gm of $Fe_{(T)}$, ore required = 100 gm.

∴ For the 100 gm of $Fe_{(T)}$, ore required $= \frac{100 \times 100}{33.02}$

Ore required = 302.85 gm

For magnetic roasting,

For the 302.85 gm of ore, coke required = 9.86 gm.

∴ For the 100 gm of ore, coke required $= \frac{100 \times 9.86}{302.85}$

coke required = 3.255 gm

For the magnetic roasting, iron ore and coke are required 100 gm of iron ore and 3.255 gm of coke as per stoichiometric calculation. Coke is taken 10 pct additional, i.e. 3.58 gm. Similarly, the amount of charcoal is calculated by stoichiometric calculation for magnetic roasting. Iron ore and coke are required 100 gm of iron ore and 2.953 gm of charcoal as per stoichiometric calculation. Charcoal is taken 10pct additional, i.e. 3.25 gm. Process parameters for magnetic roasting are as follows:

Temperature (K) – 1023, 1073, 1123, 1173

Time (min) – 30, 60, 90, 120

For magnetic roasting, weight loss is calculated by using the formula as follows:

$$Weight\ loss\ (pct) = \frac{(Initial\ weight - final\ weight)}{Initial\ weight} \times 100 \quad ....(3.4)$$

Table 3.16: magnetic roasting of iron ore

Tempra-ture (K)	Time (min)	Initial weight (gm)	Final weight (gm)	Weight loss (gm)	weight loss (pct)	Remark
1023	30	42.940	36.512	6.428	14.97	
	60	42.943	36.816	6.127	14.27	
	90	42.948	36.419	6.529	0.00	
	120	42.941	36.293	6.648	15.48	
1073	30	42.939	36.414	6.525	15.20	
	60	42.940	36.303	6.637	15.46	
	90	42.942	36.126	6.816	15.87	
	120	42.938	36.004	6.934	16.15	
1123	30	42.947	36.257	6.690	15.58	
	60	42.943	36.124	6.819	15.88	
	90	42.945	36.257	6.688	15.57	
	120	42.941	36.068	6.873	16.01	
1173	30	42.947	36.281	6.666	15.52	
	60	42.938	36.370	6.568	15.30	
	90	42.944	35.958	6.986	16.27	
	120	42.848	35.757	7.894	16.81	
1173	120	44.956	33.124	11.832	26.31	Charcoal as carbonaceous

By using equation 3.4, weight loss calculation for magnetic roasting is reported in Table 3.16. The best results were obtained for the roasting operation at 1173 K for 120 minutes. After the roasting process, roasted ore is further upgraded by wet magnetic separation. Iron recovery for magnetic roasting is calculated by equation 3.1. By using coke fines, 34.6 pct total Fe of roasted ore was achieved which was further upgraded to 49.1 pct by wet magnetic separation. The result of the beneficiation process for iron ore by coke is shown in Table 3.17.

Table 3.17: Beneficiation results of iron ore by coke

Process	**$Fe_{(T)}$ (pct)**	**SiO_2(pct)**	**Al_2O_3(pct)**	**Weight (gm)**	**Recovery (pct)**
Initial	33.02	24.121	13.227	42.848	0
Magnetic Roasting	34.635	23.462	13.095	35.757	87.53
Wet magnetic separation	49.129	12.372	11.835	22.989	91.19

The parameter of the best result achieved is 1173 K and 120 min. These parameters are adapted for the beneficiation of iron ore by charcoal. By using charcoal fines, 38.02 pct total Fe of roasted ore was achieved which was further upgraded to 49.6 pct total Fe by wet magnetic separation. The result of the beneficiation process for iron ore by charcoal is shown in Table 3.18. The flow diagram for the beneficiation of Rajasthan ore is shown in Figure 3.18. Comparatively both carbonaceous materials i.e. coke and charcoal can be useful for the beneficiation of low grade iron ore. Figure 3.19 shows nominal changes in the recovery value of iron. So, Charcoal can be replaced coke as carbonaceous material.

Table 3.18: Beneficiation results of iron ore by charcoal

Process	**$Fe_{(T)}$ (pct)**	**SiO_2 (pct)**	**Al_2O_3 (pct)**	**Weight (gm)**	**Recovery (pct)**
Initial	33.02	24.121	13.227	44.956	0
Magnetic Roasting	38.023	23.937	13.124	33.124	84.84
Wet magnetic separation	49.601	19.1	2.65	22.928	90.29

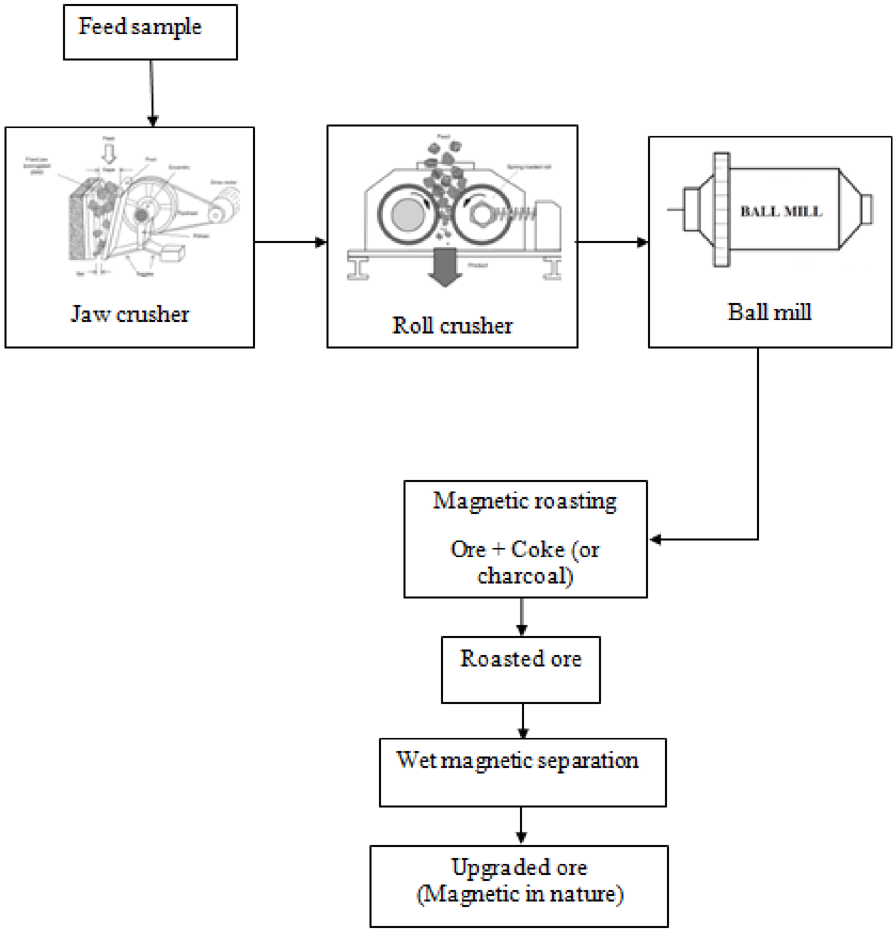


Figure 3.18: Flow sheet for beneficiation of Rajasthan ore

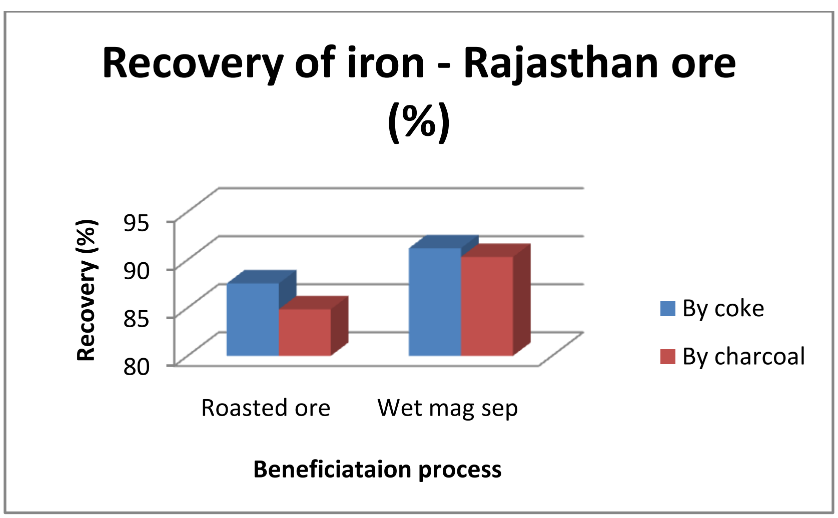


Figure 3.19: Comparison of carbonaceous material on recovery

Conclusion:

1. Mineralogical studies provide key information about texture, morphology, and phases present in ore which help to decide a suitable beneficiation process.
2. Odisha ore is BHQ type. The size of gangue and ore minerals is in microns. So, ore can be processed by conventional beneficiation route but at fine size only. $Fe_{(T)}$ is achieved from 53.1 pct to 60.26 pct with a recovery value of 59.75 pct by jigging and tabling operations. By this beneficiation route, Odisha ore is upgraded to high grade and it can be used in the agglomeration process. Based on extensive work on Odisha ore and detailed literature survey, it is concluded that this ore can easily utilise in iron making process.
3. Rajasthan ore is a limonite type of ore that has goethite as a major ore mineral and quartz as a major gangue mineral. Goethite is always challenging to beneficiate by any beneficiation process. Ore texture and particle size were not preferred for the conventional beneficiation route.
4. For Rajasthan ore, the magnetic roasting process achieved recovery of iron in the range of 85-90 pct by a transformation of goethite into maghemite at 1173 K and 120 min. $Fe_{(T)}$ is attained from 33.02 pct to 49.12 pct and 49.60 pct by coke and charcoal respectively. Coke and charcoal did not impact the recovery of iron significantly. Due to the presence of less amount of ash content in charcoal, it can be advantageous over coke. Upgraded ores by coke and charcoal are taken for further studies.