2.1 INTRODUCTION

Characterization of the raw materials is an essential part of any research work. Characterization helps to determine the elements & their oxides, phases and different constituent present in the material. It is assessed their suitability for use in various possible applications.

From the literature, it is observed that iron ore, coal and limestone are found in various forms in the earth's crust. Different types of iron ore have different characteristics, for example, colour, specific gravity, percentage iron and their chemical formula. Chemical formula decides that it is magnetite, hematite, limonite or pyrites. The formula of the ore indicates, all the properties of that particular material. There are primarily two kinds of iron ore (i.e. hematite and magnetite) used for the extraction of iron. Hematite spelt as hematite is the mineral form of iron (III) oxide (Fe_2O_3). It is the primogenital known iron oxide mineral and common in rocks. Hematite crystal belongs to the family of rhombohedral lattice system. Hematite is brittle in nature. It is harder than pure iron. Hematite is found in banded iron form. Magnetite is a rock mineral with the chemical formula Fe_3O_4 [84]. It was the first crystal structure to be found using X-Ray diffraction. The structure of magnetite is inverse spinal. In the structure O^{2-} ions form a face-centered cubic (FCC) lattice and iron cations occupy interstitial sites. Magnetite contents both divalent and trivalent iron (ferrous and ferric ions), therefore it is different from most other oxides. XRF technique can be used to determine the various elements present in the iron ore while the XRD technique can be used to find out different phases.

Coal is available in various forms in the world. The quantity of carbon, moisture, volatile matter and ash content make it different from each other. The amount of volatile matter affects the rate as well as the amount of reduction of any species with carbon. Volatile matter dissociates in the form of carbon and hydrogen and thus provides the extra amount of reducing agents. Ash mainly contained silica, so high ash contained coal increases the amount of silica in bulk as slag. Proximate analysis of coal can be used to determine the different properties present in the material [85].

In the blast furnace, lime is charged as the lump form of limestone (calcium carbonate). Calcium carbonate requires a tremendous amount of heat energy for its dissociation in the form of lime (as solid) and carbon dioxide (as gas). Thus, it consumes a considerable amount of heat of the furnace and makes the process less effective. If lumpy lime charged instead of limestone in case of BF and fine lime charged in case of BOF/EAF steelmaking process, a sufficient amount of it will come outside with the off-gases as fines/ powder form which reduce the efficiency and also makes the environment polluted [8]. For the sake of above reasons directly lime can be used in the pellets of iron ore to increases the effectiveness as well as productivity of the process. XRF technique can be used to determine the chemical form of lime powder.

Hot metal (cast iron/ pig iron) is the product of the blast furnace, which is convert into steel by using further refining process. Silicon, Manganese, Carbon, Sulphur and Phosphorous are the elements present in the hot metal that have to be removed during the steel making process. The amount of silicon in hot metal decides whether it will be used further for the cast iron or steel making. If the amount of silicon is less than 2.5%, it can be used for steel making. So the amount of silicon decides the selection/rejection of pig iron for the steelmaking technology. Hot metal-containing carbon ranging from 4.0 to 4.5%, silicon 0.4 to 1.5%, manganese 0.15 to 1.5%, phosphorous 0.045 to 0.25% (normally 0.06-0.25%) and sulphur maximum 0.15% can be used for the conversion into steel in the Basic Oxygen Furnace (BOF) steelmaking process. To produce internal crack free products with appropriate surface quality it is compulsory to minimize the level of sulphur and phosphorous to less than 0.01% even some times less than 0.005% [8] [2]. To get the desired amount of sulphur and phosphorous in the product, charging of hot metal (pig iron) having low sulphur and phosphorous may be a useful method. The chemical analysis of different elements present in the pig iron can be done by using Optical emission spectroscopy (OES) technique.

2.2 EXPERIMENTAL

Figure.2.1 shows the steps involved in the preparation and characterization of the raw materials which is used in the current study.

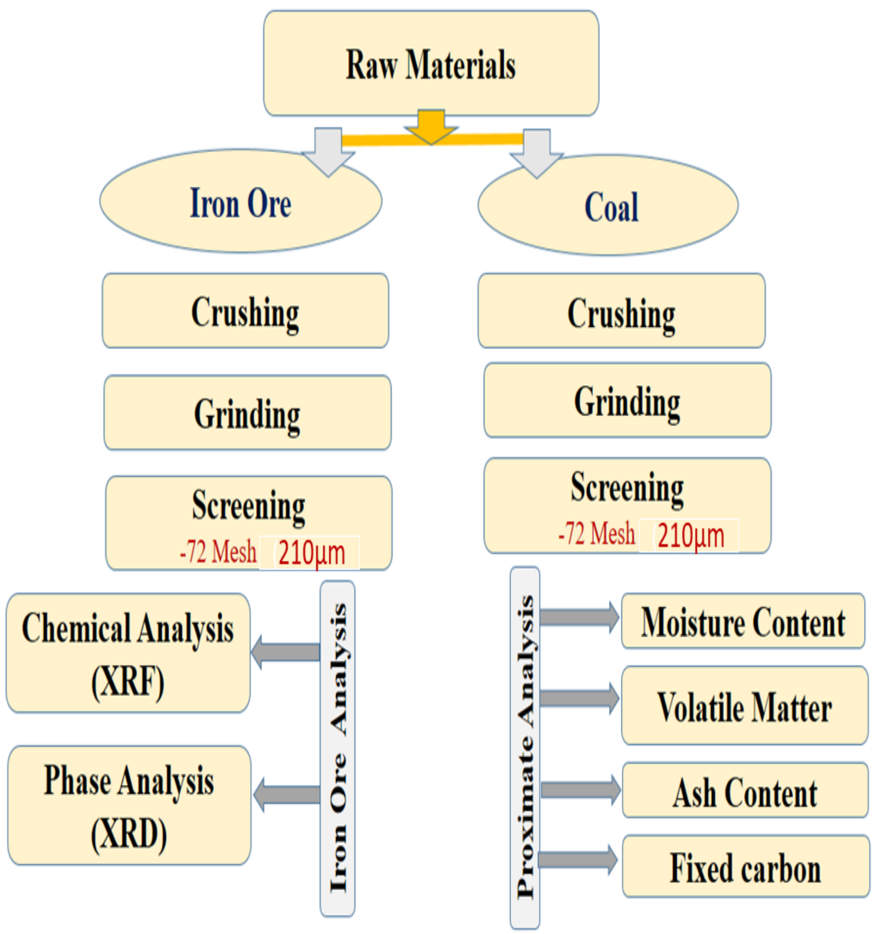

Figure 2.1 Flow chart for preparation and characterization of raw materials

These processes are described in following sub-sections of different heading.

2.2.1 MATERIALS PROCUREMENT

Iron ore was collected from the TATA Steel, Jamshedpur (Jharkhand), India. Lump iron ore which received for the process has lump of size 60-80mm.

Lime powder was purchased from local supplier. It is in the form of fine powder to directly use in the pellet making. It has almost 95% calcium oxide (CaO) in its total quantity.

Coal was also purchased from the local supplier. The original size of coal is as lump of 60-80mm.

Cast iron (pig iron) as pigs was collected from the TATA METALLIKS, Kharagpur, West-Bengal, India.

2.2.2 RAW MATERIALS PREPARATION

Raw materials (Iron ore/Coal) collected are larger in size and can't be used directly in the pellets making, that's why they are converted first into fines. All the processes (i.e. crushing, grinding and screening) are done in the Mineral Beneficiation Laboratory, Department of Mining Engineering, Indian Institute of Technology (BHU), Varanasi, India. The operations used to convert lumps into fine form are described in following sub-sections respectively.

2.2.2.1 Crushing

Crushing is the process in which large size material as iron ore and coal converted first into small pieces followed by powder making. There are two types of crusher used in this process. In the first step, a fixed jaw crusher was used to break the material from its original form to approximately -5 mm size particle. After this second type crusher (roll crusher) is used to convert into fines of -1mm size. These crushers are shown in **Figure2.2**.

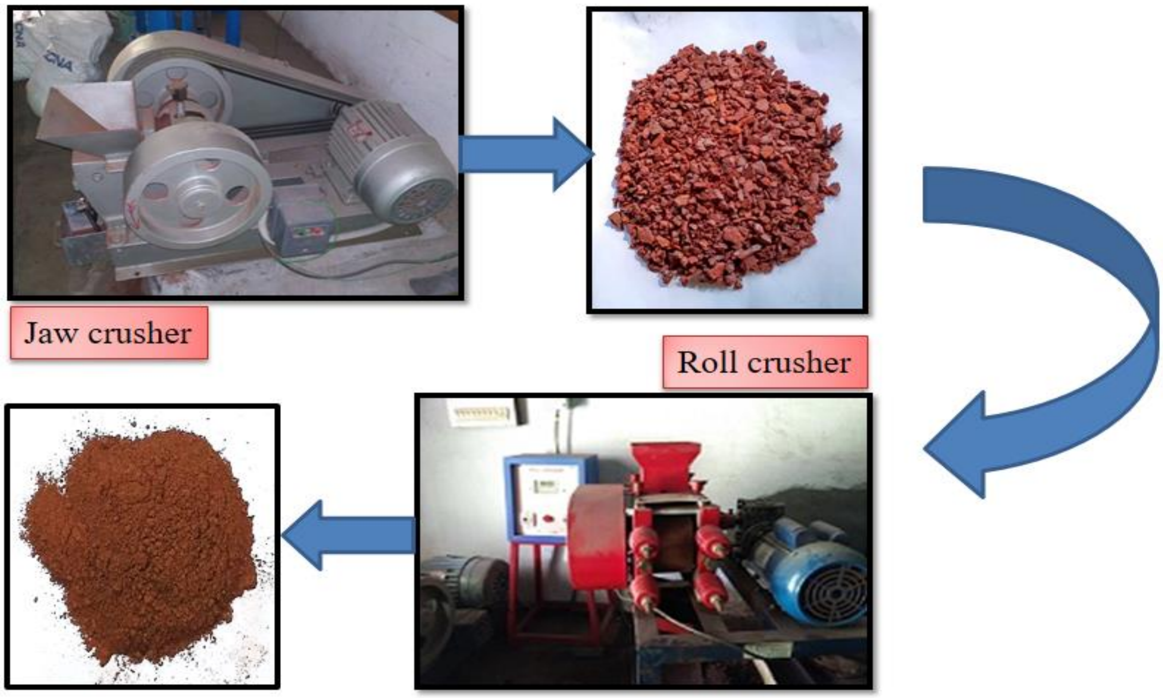

Figure 2.2 Jaw and Roll Crusher used to crush raw materials up to fines of (-1mm)

2.2.2.2 Grinding

Fines (-1mm) obtained after crushing is not enough for making pellet. So, further work is done to make it finer, and this process is known as grinding. This process used to convert the iron powder and coal into a fine powder which has a size less than 0.2 mm. In this process a 5 Kg capacity ball mill which contains steel balls of 20 mm diameter was used as shown in **Figure 2.3**. Iron ore fines were powdered to fineness passes through 72 mesh to get optimum value of particles which are further used to pellet making. This size of the particle is generally followed in the industry.

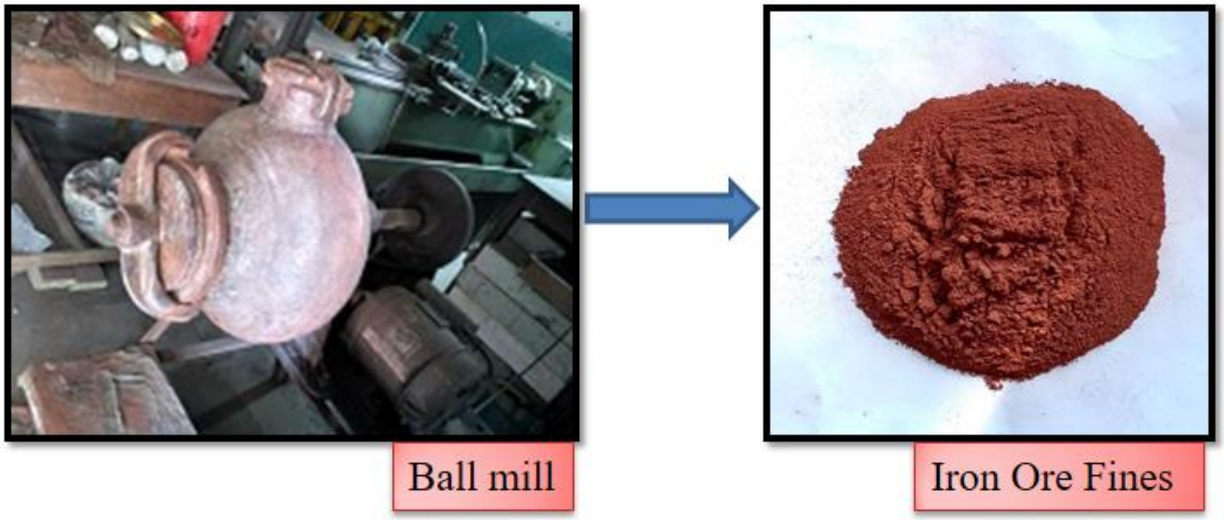

Figure 2.3 Ball Mill used to grind iron ore into powder

2.2.2.3 Screening/Sieving

Screening is the operation to get the desired size of the feed material by mechanical separation using various types of sieves. All the particle after the ball milling do not have the same size. For this project work, required size of the fines is < 0.2 mm. C136/C136M – 14 standard is followed for the screening process. For achieving the appropriate size of the material, screening was done by using up to 72 mesh size sieves. After screening through 72 mesh it contains a large variation in particle size. Size of the particles affect the hardening and reduction mechanism of the pellets therefore it is essential to see the quantity of different size particles present in the iron ore fines. To determine the quantity of different particles present in the iron ore fines sieve analysis (Set up is shown in **Figure 2.4**) was performed in the Foundry Metallurgy Division of Department of Metallurgical Engineering, Indian Institute of Technology (BHU), Varanasi. For the sieve analysis 100 gm powder of iron ore was taken and it was shake for the 10 minute in the setup of particle size analyzer. Weight of the material present in the different sieves after 10 minute shaking was weighed. This process repeated 5 times and on the behalf of these results cumulative values calculated to get the particle size of the iron ore powder.

Figure 2.4 Set up used for particle analysis of iron ore

2.2.3 TESTING OF RAW MATERIALS

Chemical analysis of pig-iron, iron ore & lime, Phase analysis of Iron Ore and proximate analysis of coal are explained one by one in the following subsections.

2.2.3.1 Chemical Analysis of Iron Ore & Pig Iron

Chemical analysis is performed to know the different elements present in the raw materials with the use of XRF and Optical Emission Spectrometer (Foundry Master). XRF test for iron ore was performed at Mahindra Sanyo special steel Pvt. Ltd Khapoli, Raigad, India. Pig iron was analyzed with the use of Optical Emission Spectroscopy (OES) technique in the Department of Metallurgical Engineering, Indian Institute of Technology (BHU), Varanasi, India.

2.2.3.2 Phase Analysis of Iron ore

X-Ray Diffraction (XRD) (Model Rikagu Miniflex-600 model with Dtex ultra detector and Cu-Kα radiation λ=1.54nm, acceleration voltage=40Kv, current=15mA) was used to determine the phases present in the iron ore. Samples were scanned at the rate of 2 degree per minute from 15° to 90°. Expert High Score and PCPDF Software used to determine the phases of the material.

2.2.3.3 Proximate Analysis of Coal Powder

Proximate analysis of coal was performed to know the presence of moisture, volatile matter, ash and fixed carbon content, in the Industrial Metallurgy Division, Department of Metallurgical Engineering, Indian Institute of Technology (BHU), Varanasi. The average result was reported for three set of experiments. Methods are describing in the following sub-section.

2.2.3.3.1 Moisture Test

Loss in weight of coal produced by heating of weighed quantity of coal sample for one hour at 110°C is the moisture content. A glass dish (50mm diameter & 10mm deep) with lid is used to determine moisture content. One gram finely powdered (-72 mesh, i.e.-200mm) coal sample is taken in the glass dish with lid and weighed on digital balance. Sample is exposed to 110 ± 5°C temperature for one hour in the oven without lid on dish. Sample is cooled after heating by keeping it in a desiccator with lid in position to avoid moisture adsorption. After cooling, the sample is weighed keeping the lid in position. Loss in weight is accounted for inherent moisture content [3].

% Inherent Moisture = (Loss in weight of coal / Initial weight of coal)*100

2.2.3.3.2 Volatile Matter Test

Specially designed silica crucible (22mm inner diameter and 38mm deep) with lid is used to determine the volatile matter content in coal. Deep crucible with lid allows heating the sample in absence of air. One gram coal sample is taken in the crucible and weighed with lid accurately on a digital balance. This crucible is then placed over a tripod (made of Nichrome wire) to keep its bottom 5mm above the floor of the furnace. The crucible with tripod was heated in a furnace at 925± 5°C for 7 minutes. The crucible was taken out, cooled in air with lid in position and then weighed to notice the loss in weight due to the elimination of volatile matter and inherent moisture [3].

% Volatile Matter = [(Loss in weight of coal / Initial weight of coal)*100] - [% inherent Moisture content]

2.2.3.3.3 Ash Determination

Silica dish (50mm diameter and 10mm deep) without lid is used for this purpose. One gram coal sample was taken and weighed accurately on digital balance. The coal is spread uniformly in the crucible by gentle tapping. This was heated at 400±5°C for 30 minutes in furnace to oxidize all the sulphur and part of carbon in coal. Then that crucible was transferred to another furnace at 800±5°C to oxidize the sample for 60 minutes and repeated for several times for complete combustion to achieve a constant weight of residue. Then dish was cooled to weigh the residue ash content [3].

%Ash in coal= (weight of residue ash formed/ Initial weight of coal)*100

2.2.3.3.4 Fixed Carbon Estimation

It is estimated indirectly by subtracting the total percentage of moisture, volatile matter and ash content from 100 [3].

% Fixed Carbon = 100 - (% Moisture + % Volatile Matter + % Ash Content)

2.3 RESULTS AND DISCUSSION

The obtained results were critically examined with proper analysis which is presented in the following section.

Chemical analysis of iron ore and lime powder are shown in **Table-2.1**. From the data, it can be clearly seen that hematite present as the main phase (92.50 wt. %) in the iron ore. Silica and alumina presented as the major gangue material with a trace amount of other elements. Lime powder contained ~95 wt. % calcium oxide with a little amount in loss of ignition.

Table-2.1 Chemical Analysis of Iron Ore & Lime

Chemical Composition of Raw Materials (Wt. %)								
Iron Ore							Lime	
Fe_2O_3	Al_2O_3	SiO_2	MnO	P_2O_5	SO_3	Others	CaO	LOI
92.50	3.70	2.94	0.09	0.35	0.12	0.30	95	5

Chemical analysis of pig iron is shown in **Table-2.2**. Chemical analysis of pig iron shows iron present here in the major quantity (93.05wt. %) with 3.83wt% carbon. The amount of impurities which are considered for the study are sulphur (0.11wt. %) phosphorous (0.12wt. %) silicon (2.36 wt. %) and manganese (0.46wt %).

Table-2.2 Chemical Analysis of Pig Iron

Chemical Composition (Wt. %)						
Iron	Carbon	Silicon	Manganese	Sulphur	Phosphorous	Titanium
93.05	3.83	2.36	0.46	0.11	0.12	0.07

The phase analysis is essential to know the form of bonds inside the material. Existence of element with other elements can be determined with the help of a phase in which constituents present. XRD technique was used to know the phases present in the iron ore powder. XRD pattern of the given iron ore presented in **Figure 2.5**. It can be clearly seen from the figure that the measure phase present in the iron ore is hematite. The amount of other phases is very less as it was shown in above **Table-2.1** so they cannot be detected with the help of XRD.

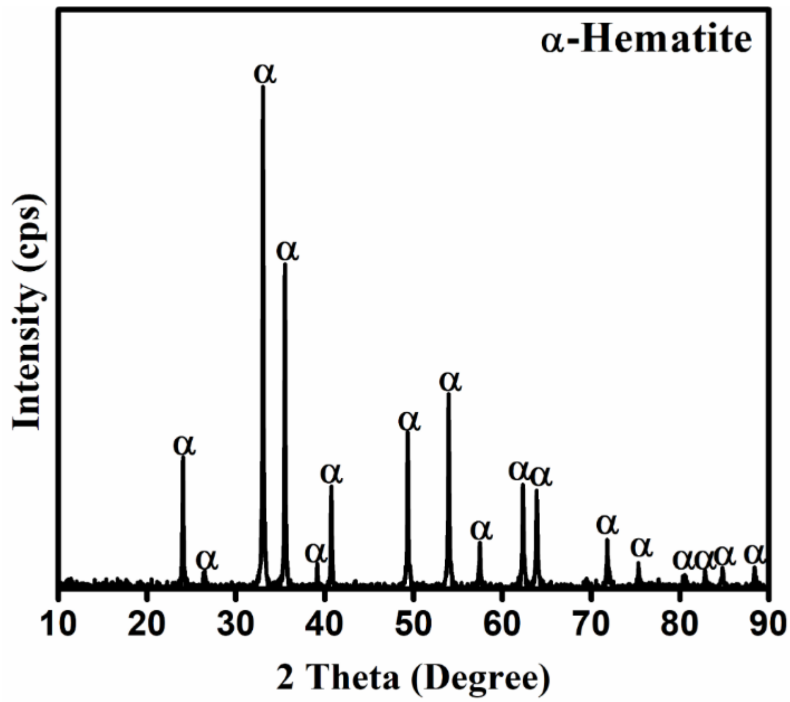

Figure 2.5 X-Ray Diffraction pattern of iron ore

Report of proximate analysis of coal is represented in the **Table-2.3**. From the table, it can be seen that fixed carbon content in the coal is 62.50 wt. % with 8.1 wt. % moisture and 14.7 wt. % volatile matter.

Table-2.3 Proximate Analysis of Coal

Proximate Analysis of coal (wt. %) [average of three set of experiments]				
Reductant	Fixed Carbon	Moisture	Volatile Matter	Ash Content
Coal	62.5	8.1	14.7	14.7

Quantity of different particle size present in the iron ore fine is shown in **Figure 2.6**. It can be seen from the figure that iron ore fines contain all the particles are smaller than 210µm. It contain D_{90} means 90% particles smaller than 140µm, D_{50} means 50% particles smaller than 70µm and D_{10} means 10% particles smaller than 40µm.

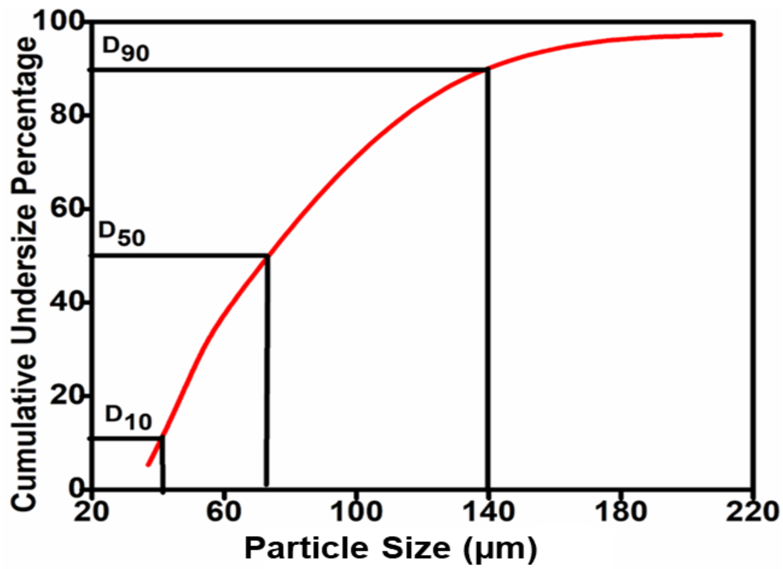

Figure 2.6 Particle Size Analysis of Iron Ore fines

2.4 FINDINGS

- Iron Ore

- Hematite presents as the major phase (92.5 wt. %) in the iron ore fines with Silica (2.94wt. %) and alumina (3.70wt. %) presented as the major gangue material with a trace amount of other elements.

- Particle size of the iron ore fines (<210 µm) has a mixture of different sizes of D_{10}-40µm, D_{50} -70µm, D_{90} -140µm.

- Lime Powder

- CaO (95wt. %) presents as major chemical constituent in the lime.

- Coal

- Coal contain carbon 62.50wt. % with 8.1wt. % moisture and 14.7wt. % volatile matter.