# Chapter 4 Experimental Set-up for Reduction Studies and Smelting Reduction

The fourth chapter discusses the briquette preparation and experimental setup for isothermal reduction studies; smelting reductions, apparatus and procedure are also discussed. Characterization and testing of materials are discussed in this chapter. The testing techniques and measurement of degree of reduction and activation energy are calculated.

Rajasthan iron ore was upgraded by coke and charcoal. Four types of composite briquettes were prepared for an isothermal and non-isothermal reduction (TG-DTA) and smelting reduction studies. These are as follows:

- **AA** - Upgraded iron ore (Rajasthan) by coke + Coke
- **AB** - Upgraded iron ore (Rajasthan) by coke + Charcoal
- **BA** - Upgraded iron ore (Rajasthan) by charcoal + Coke
- **BB** - Upgraded iron ore (Rajasthan) by charcoal + Charcoal

## 4.1 Briquette Preparation

Cylindrical shaped iron ore – coke / charcoal composite briquettes were prepared. Briquettes were prepared using a mandrel and die assembly. Load is applied by giving free flow single impact to the moist powder mixture. One impact to the powder mixture was applied to simulate the impact (due to rotation and fall of the materials) occurred during commercial pelletizing operation. Two different sized of iron ore-carbonaceous materials were prepared based on geometry of furnaces: (i) for Isothermal reduction studies - diameter 10 mm, height 12-13 mm and weight in between of 1.6-1.9 gm and (ii) for smelting reduction - diameter 25 mm, height 24-25 mm and weight in between of 31-33 gm. Based on detailed literature survey, 10 pct Polyvinyl Alcohol (PVA) solution was used as binder. Briquettes were dried in open atmosphere for 24 to 48 hours. Further briquettes were dried in an oven at 383 K temperature for 1 hour immediately before briquettes' testing or isothermal reduction or smelting reduction studies. Figure 4.1 shows steps involved in composite briquette making.

## 4.2 Stoichiometric calculation:

For the AA type of composite briquette, the weight of iron ore and coke is calculated by stoichiometric calculations. The following reaction takes place in the reduction of composite briquettes:

$$Fe_2O_3 + 3\,C \rightarrow 2\,Fe + 3\,CO$$

**Wt. (gm) 160 36 112 48**

For the reduction of 160 gm of $Fe_2O_3$, carbon required = 36 gm.

∴ For the reduction of 100 gm of $Fe_2O_3$, carbon required $= \frac{100 \times 36}{160}$

Stoichiometric carbon required ($S_C$) = 22.5 gm

From proximate analysis of coke, fixed carbon is having68.38 gm in 100 gm of coke. (Table 3.6)

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

∴ For the 22.5 gm of Carbon, Coke required $= \frac{22.5 \times 100}{68.38}$

Coke required = 32.94 gm

From Chemical analysis of upgraded Iron ore (AA), $Fe_2O_3$ is 70.18 pct ($Fe_{(T)}$ = 49.129 pct). So,

For the 70.18 gm of $Fe_2O_3$, ore required = 100 gm.

∴ For the 100 gm of $Fe_2O_3$, ore required $= \frac{100 \times 100}{70.18}$

Ore required = 142.49 gm

142.92 gm iron ore and 32.94 gm coke are required for the briquette making by stoichiometric calculations. Similarly, the desired weight of iron ore and coke / charcoal was calculated and reported in Table 4.1.

Table 4.1: Calculated weight of raw materials for briquette making

| Types | Carbonaceous material | $Fe_2O_3$ (pct) | Fixed Carbon (pct) | Ore required (gm) | Coke / charcoal required (gm) |
|---|---|---|---|---|---|
| AA | Coke | 70.18 | 68.38 | 142.49 | 32.90 |
| AB | Charcoal | 70.18 | 75.37 | 142.49 | 29.85 |
| BA | Coke | 70.86 | 68.38 | 141.12 | 32.90 |
| BB | Charcoal | 70.86 | 75.37 | 141.12 | 29.85 |

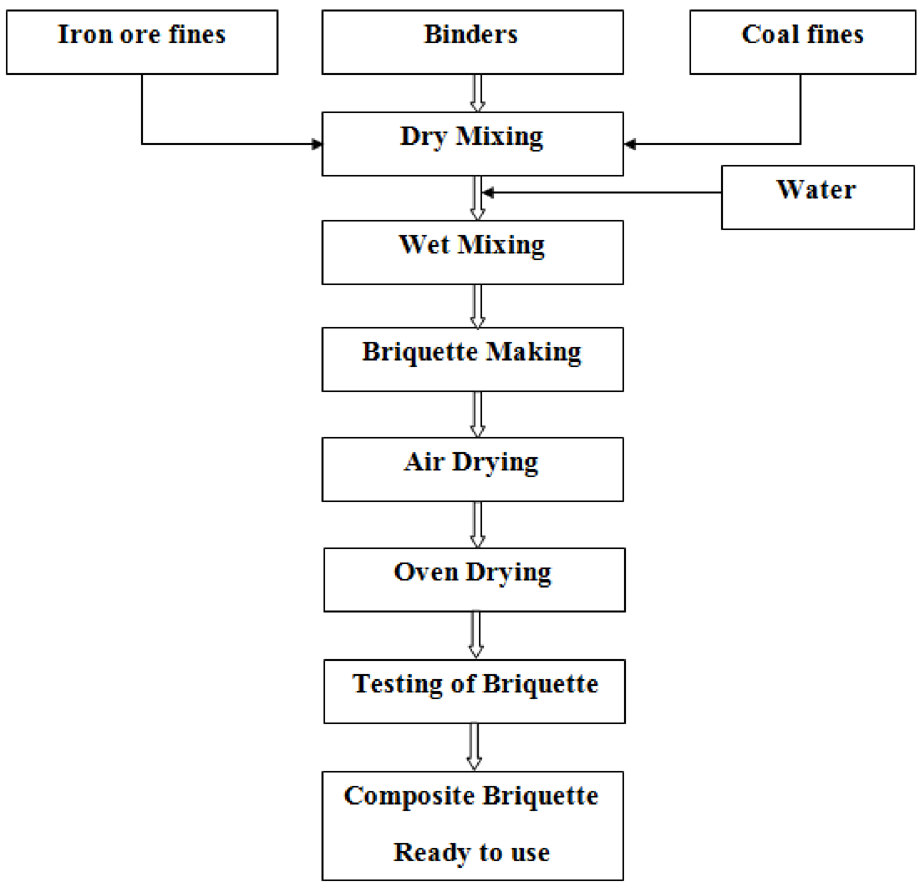


Figure 4.1: Flow diagram for composite briquette making

## 4.3 Evaluation of the Properties of Composite Briquettes

The composite briquettes were subjected to the following tests:

I. ***Drop test:*** In the Drop test, the briquettes / pellets were dropped repeatedly from a height of 450mm on a 10 mm thick steel plate until they broke. The final value was taken as the average of five such test values.

II. ***Compression test:*** the briquettes / pellets were tested on a compression strength-testing instrument (Montanso Tensometer, Japan; crosshead speed: 30 mm/min) at Metallurgical and Materials Engineering Department, M. S. University of Baroda. The final value was calculated as the average of five such test values.

III. ***Shatter Index test:*** The known amounts of the briquettes/pellets were dropped four times from a standard height of 2000 mm on a 10 mm thick steel plate. The broken sample pieces were put on a 100 mesh sieve. The amount of material, expressed as a percentage of the original weight passed through 100 mesh sieve was considered to be the shatter index:

$$Shatter\ index = \left(\frac{Weight\ of\ -100\ mesh\ size\ particles}{Total\ weight\ of\ sample\ taken}\right) X\ 100$$

Table 4.2: Test results for composite briquette

| Sr. No. | Type | Diameter of briquette (mm) | Green Drop Strength | Dry Drop Strength | Compressive Strength (N/briquette) | Shatter Index (pct) |
|---|---|---|---|---|---|---|
| 1 | AA | 10 | 90 | 157 | 880 | 0.15 |
| 2 | AB | 10 | 70 | 102 | 986 | 0.94 |
| 3 | BA | 10 | 109 | 179 | 1170 | 0.1 |
| 4 | BB | 10 | 86 | 126 | 871 | 0.57 |
| 5 | AA | 25 | 4 | 10 | 470 | 5.92 |
| 6 | AB | 25 | 3 | 10 | 703 | 3.17 |
| 7 | BA | 25 | 2 | 8 | 380 | 7.11 |
| 8 | BB | 25 | 3 | 11 | 630 | 4.89 |

The test result values of the average of the three tested values of briquettes are shown in Table 4.2. The values varied mostly by ± 20 pct from the average value of a sample.

## 4.4 Construction of tube furnace

The tube furnace was constructed using an alumina tube and Kanthal FeCrAl coil wire which can withstand up to 1650 K. Maximum 1475 K working temperature is maintained inside the tube. The one end of the tube is connected with a three way valve. A valve allows only gas to flow in a furnace at a time. On the other end of the tube, there is a thermocouple arrangement to measure the temperature in the tube.

*Dimensions:*

- Length of tube = 600 mm
- Outer Diameter of tube = 50 mm
- Inner Diameter of tube = 45 mm
- Length of hot zone = 300 mm

Figure 4.2: Tube Furnace

## 4.5 Isothermal studies of composite briquette

The degree of reduction (α) of iron ore can be obtained by the weight loss method at constant temperature, where a carbonaceous material as a reductant is used. Weight loss of the sample is not enough; some additional measurements are required for estimating the degree of reduction (α), which is measured as follows:

$$\text{Degree of reduction } (\alpha) = \frac{\text{Weight of oxygen removed from iron oxide}}{\text{Total weight of removable oxygen present in iron oxide}} \times 100 \quad ..(4.1)$$

Sah and Dutta[120] applied the following equation for calculations of the degree of reduction to measure of the loss of volatile matters:

$$\alpha = \left[\frac{4 \times \{f_{wl} - (f_{coal} \times f_{vm})\}}{7 \times \{f_{ore} \times \rho_{ore} \times f_O\}}\right] \times 100 \quad \ldots(4.2)$$

where, $f_{wl}$ - fractional weight loss = $[(W_i - W_f) / W_i]$,

$W_i$ - initial weight of the composite briquette,

$W_f$ - final weight of the composite briquette after reduction,

$f_{coal}$ - fraction of coal present in composite briquette,

$f_{vm}$ - fraction of volatile matters present in coal,

$f_{ore}$ - fraction of waste present in composite briquette,

$\rho_{ore}$ - purity of iron oxide ($Fe_2O_3$) in waste,

$f_O$ - fraction of oxygen present in pure $Fe_2O_3$.

The experiments were planned to study the reduction kinetics of the composite briquettes in isothermal conditions at a range of temperatures. A horizontal electrically heated tube furnace was used for experiments. Initially, the furnace is calibrated by Pt, 10 pct Pt-Rh type thermocouple. Temperature profile is shown in Figure 4.3. The boat is made from quartz which is used for the briquettes to be positioned into the core of the tube furnace. Briquettes were dried at 383 K for 1 hr before insertion it into the furnace. Two briquettes of 10 mm diameter were taken. The weights of briquettes were in the range of 1.6-1.9 gm. The briquettes were placed into a boat which was put into the preheated tube furnace in a nitrogen (99.5 pct pure) atmosphere. After the reaction time, the boat was shifted into desiccators for cooling. Isothermally reduced briquette is shown in Figure 4.4. The cooled briquettes were weighted to calculate the fraction of reduction. The variables for Isothermal reduction of composite briquettes are shown in Table 4.3.

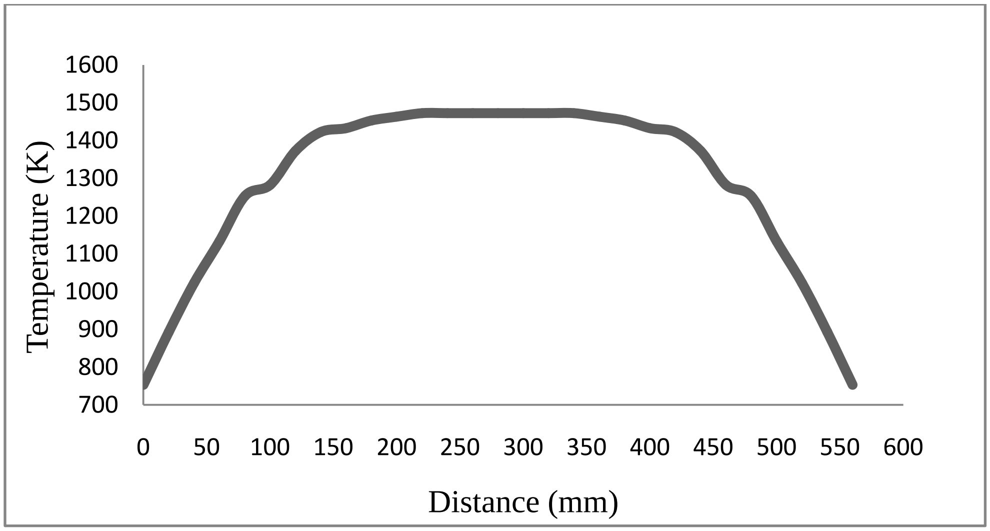


Figure 4.3: Temperature profile of tube furnace

Figure 4.4: Isothermally reduced composite briquette

Table 4.3: Variable for isothermal reduction of composite briquettes

| Sr. No. | Variable | Number | Remarks |
|---|---|---|---|
| 1 | Iron ore | 2 | Upgraded ore by coke and charcoal |
| 2 | Carbonaceous material | 2 | Coke and charcoal |
| 3 | Temperature, T (K) | 3 | 1223, 1273, 1323 |
| 4 | Time, t (s) | 5 | 150, 300, 450, 600 and 1200 |

### 4.4.1 Phase identification (by XRD)

An X-Ray Diffraction (XRD) analysis is majorly used to identify phases present in samples. Isothermally reduced iron ore was taken for XRD analysis. Characterization was done on an XRD Diffractometer (Model: EMPYREAN, Make: PANalytical) at Metallurgical and Material Engineering Department, Indian Institute of Bombay, Mumbai.

### 4.4.2 SEM Analysis

Scanning Electron Microscopy (SEM) is an important analytical instrument for surface topography, texture and size analysis of a spot or identified surface area. SEM analysis was used for reduced briquette at 1323 K and 20 min. A Jeol SEM (Model: JSM—5610 LV) is available at Metallurgical and Material Engineering Department, M. S. University of Baroda.

## 4.6 Calculation of Rate of reduction (k) and activation energy ($E_A$)

With the help of equation 4.2, the fraction of reduction (f) of composite briquettes is calculated. f *vs* t plots are drawn from the experimental data. From the obtained straight line of the plot, the slope of that straight line is established to measure the rate of reduction (k).

Therefore, $k = \frac{df}{dt}$ …. (4.3)

The activation energy ($E_A$) can be calculated from Arrhenius equation:

Rate of reduction, $k = A \times e^{-\left(\frac{E_A}{RT}\right)}$ … (4.4)

where, A is constant,

$E_A$ is activation energy,

R is gas constant, and

T is absolute temperature.

Therefore, $\ln k = \ln A - \left(\frac{E_A}{RT}\right)$ … (4.5)

Equation (4.5) is used for calculations of the reduction kinetic data. A plot of ln k *vs* 1/T gives a slope ($E_A$/R) of line; from which the activation energy ($E_A$) can be measured.

Therefore, the activation energy ($E_A$) = slope x R … (4.6)

## 4.7 Non-isothermal reduction studies by TG-DTA

Thermogravimetry (TG) is used to measure changes in the weight of a sample which may result from chemical or physical transformations, as a function of temperature or time. The sample may either lose weight to the atmosphere or gain weight by reaction with the atmosphere Differential thermal analysis (DTA) can be used to detect the physical and chemical changes, which are accompanied by a gain or loss of heat in a material as a function of temperature or time.[127] A powder mixture of upgraded ore with coke and charcoal was examined in EXSTAR TG/DTA 6300 instrument at Institute Instrumentation Centre, Indian Institute of Technology, Roorkee. A powder is tested in Argon (200 ml/min) atmosphere up to 1473 K with heating rate of 10 K/min.

## 4.8 Smelting reduction studies

Due to the lack of high grade iron ore and coking coal, alternative ironmaking arises as a prominent route for achieving the steel demand. An attempt to utilize the upgraded iron ore as a composite briquette for steelmaking is carried out with the help of an induction furnace.

The induction furnace consists of a refractory container, capable of holding the molten bath, which is surrounded by a water-cooled helical coil connected to a source of alternating current. Induction heating is the heating of an electrically conducting object immersed in a varying magnetic field. An alternating electromagnetic field induces eddy currents in the metal thereby electrical energy gets converted to heat. The quantity of heat evolved depends on the resistivity of the charge. The object being heated may not be a magnetic material to heat efficiently. All that is required is that it should have reasonably good electrical conductivity.[128]

A laboratory induction furnace has a 15 kW power rating and 5 kg melt capacity at Metallurgical and Materials Engineering Department of M. S. University of Baroda (Make: Inductotherm, India). The unit operates on 460 V, three-phase, 50 Hz, 20 kVA power source with an output of 9600 Hz. (Figure 4.5) It is a variable induction power (VIP) furnace with an external water storage and supply system that provides a water to cool the shell and heat exchanger of the internal system and the furnace coil. The furnace bottom was properly grounded with alumina ramming mass manually. The ceramic bonded clay graphite crucible was filled with steel scrap as charge material and then put into the induction furnace for melting.

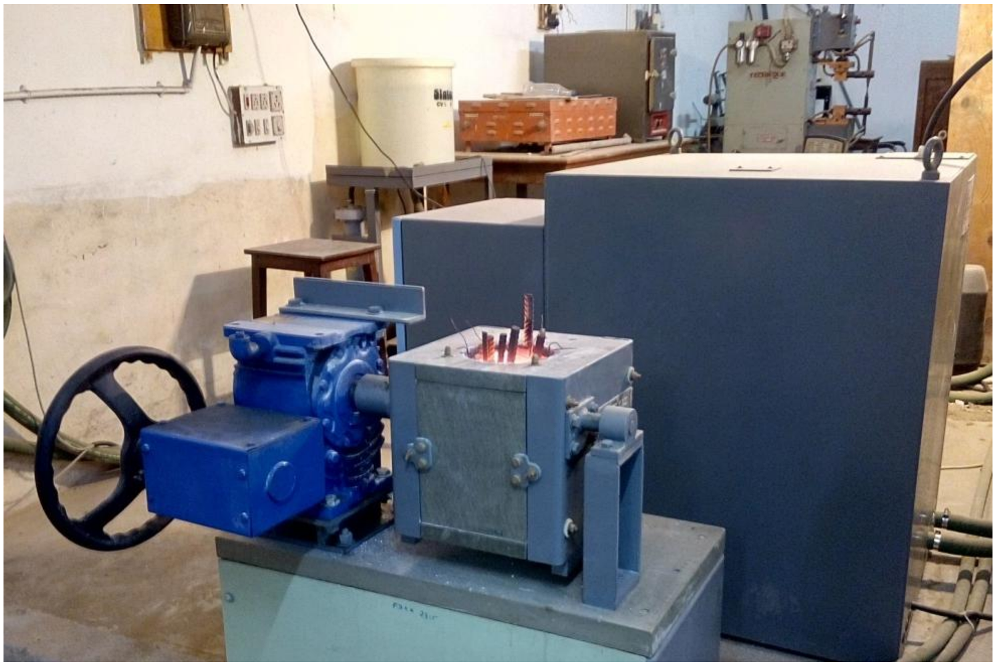

Figure 4.5: Induction furnace

## 4.9 Mould preparation

A sand mould was prepared at least 24 hr before the melting practice. Sand and bentonite (2 pct) were mixed properly in a sand muller with approximately 5 pct water to prepare the moulding sand. A single piece mould box was first filled with the moulding sand and then ramming was done. Mould cavities were made in a vertical position by piercing cylindrical wooden rods in the rammed moulding sand. Venting was done to ease the escape of gases generated during casting. Moulds were thoroughly dried by an oil fired burner heating method before a liquid metal pouring (Figure 4.6).

Figure 4.6: Dried mould - ready for pouring

## 4.10 Smelting Reduction of Composite briquette in Induction Furnace

Smelting reduction of composite briquettes was carried out in a laboratory induction furnace. The experiments were designed to investigate: dissolution behavior and bulk dissolution of composite briquettes in molten bath, to make steel and to evaluate the recovery of iron. The variables for smelting reduction of composite briquettes are shown in Table 4.4.

Table 4.4: Variable for smelting reduction of composite briquettes

| **Sr. No.** | **Variable** | **Number** | **Remarks** |
|---|---|---|---|
| 1 | Composite briquette | 4 | AA, AB, BA, BB |
| 2 | Steel scrap | 1 | Metallurgy Dept., MSU |
| 3 | pct of briquette charged | 4 | 5, 10, 15, 20 |

### *4.10.1 Dissolution behaviour and bulk dissolution of composite briquettes*

Steel scrap-filled crucible was put into a furnace. The furnace was switched on with an initial power supply of 4 to 5 kWh and gradually raised to 10 kWh. Within half an hour the steel scrap was melted and homogenization took place chemically i.e. hot heel. A sample of liquid metal was taken for chemical analysis (i.e. *Initial sample*). A Liquid metal bath was ready to dissolve the composite briquette. To study the dissolution behaviour of composite briquettes in a molten bath, a single composite briquette was put in the molten bath and visually observed how the composite briquette dissolved in the molten bath and the time required for complete dissolution was noted. This was repeated several times. It was observed that the 25 mm diameter composite briquettes completely dissolved in the molten bath within 45-50 seconds.

The experiments were carried out to observe the bulk dissolution of composite briquettes in a molten bath to produce steel and to calculate the iron yield. Preheating of briquettes took place due to the radiation from the hot heel and subsequently reduction and dissolution of briquettes occurred in the hot heel. A proportionate amount of lime powder was added for easy slag formation. After the complete dissolution of briquettes and slag-off, the liquid molten bath was allowed to homogenize for some time. The fluidity of molten metal increased with time which led to the homogenization of the bath due to convective stirring within liquid molten bath. The slag was removed with the help of a steel (TMT) rod. Liquid molten metal was poured into a dried sand mould. The casting was knocked out after solidification. Chemical analysis of initial and final product samples was done at (Model: S5 - Solaris Plus, Make: G.N.R., Italy) Aadhya Engineering Service, Makarpura, Vadodara.

Yield or recovery of iron was calculated by:

$$Iron\ yield (pct) = \frac{Total\ iron\ in\ output\ cast\ sample}{Total\ iron\ in\ input\ materials} \times 100$$

$$= \frac{Total\ iron\ in\ output\ as\ cast\ sample \times 100}{Total\ iron\ from\ scrap + Iron\ from\ composite\ briquette + Iron\ form\ stirring\ rod}$$

$$= \frac{(W_5 \times F_6) \times 100}{\{[(W_1 - W_2) \times F_1] + (W_3 \times F_2 \times F_3 \times F_4) + (W_4 \times F_5)\}} \ldots (4.7)$$

where, $W_1$-weight of TMT scrap, kg

$W_2$-weight of sample taken for chemical analysis before briquette addition

$F_1$-fraction of Fe present in initial melt

$W_3$-weight of composite briquette charged, kg

$F_2$- fraction of iron oxide present in composite briquette

$F_3$- fraction of purity of iron oxide

$F_4$- fraction of Fe present in iron oxide

$W_4$-weight of TMT rod dissolved during stirring the melt, kg

$F_5$- fraction of Fe present in TMT rod

$W_5$-weight of product, kg

$F_6$- fraction of Fe present in product

## 4.11 Microstructure of steel products and hardness measurement.

Steel produced from the induction furnace was normalized at 1173 K for 1 hr. The normalized microstructure was etched by 2 pct Nital solution and observed under a microscope at different magnifications. The hardness of normalized steel was measured by the Rockwell testing machine. Due to high carbon content in steel, B scale was used for hardness measurement.