3 EXPERIMENTAL

3.1 Material Preparation:

Two types of material were used for experimental work

1 Synthesised pure iron oxide

2 Iron ore powder

3.1.1 Synthesis of pure iron oxide:

Pure iron oxide was synthesised by precipitation of iron hydroxide from analytical grade ferric chloride with ammonia as per following reaction.

$$FeCl_3 \text{ (dilute)} + 3NH_4OH \rightarrow Fe(OH)_3 \downarrow + 3NH_4Cl \text{------} (3.1)$$

The precipitate was filtered, washed and dried at 200^0C for 3 hr and further fired at 910^0C for 1 hr to decompose the hydroxide to Fe_2O_3. The completion of reaction was checked by x-ray diffraction.

3.1.2 Iron ore powder Preparation:

The lumps of iron ore procured from Rajhara mines of Madhya Pradesh was crushed in jaw crusher, roll crusher and further ground in ball mill, to get the desired size. The colour of the ore powder was dark brown.

3.2 Powder Characterisation:

3.2.1 Size analysis:

Size analysis of pure iron oxide and ground ore was carried out by conventional sieve analysis using British Standard sieve set .

3.2.2 Chemical analysis:

Conventional wet analysis was used for analysis of the ore powder and pure oxide.

3.2.3 X-ray diffraction :

X-ray analysis of the pure iron oxide, iron ore and the products of iron ore after firing it at different temperature as, 355^{0}C, 650^{0}C, 950^{0}C , 1200^{0}C and 1250^{0}C in air, oxygen and in inert atmosphere were carried out by Rigaku D Max III Geiger flex x-ray diffractometer of 3 KVA capacity using copper target and nickel filter. The instrument consists of horizontal goniometer with scanning range of 3^{0} to 160^{0} , scintillating counter (NaI) as signal sensor and software facilities to process data. The samples were normally scanned between 10^{0} to 80^{0} with a scan speed of 3^{0}/min to 5^{0}/min and the observed raw data were processed by instrument software for base line correction and calculation of d, Io (peak intensity), I/Io, and peak width values for desired ranges. Figure 3.1 shows the photograph of x-ray diffractometer.

3.2.4 Thermal analysis (TG/DTG/DTA):

Thermal Analysis was carried out on SIEKO TG/DTA 32/5100 simultaneous thermal analysis system with following specifications:

Temperature range	Ambient — 1500 ^{0}C
Balance Type	Horizontal Differential
TG measurement range (Sensitivity)	+ 200mg (0.2ug)
DTG measurement range	0.5 mg/min — 1g/min
DTA measurements range (sensitivity)	+ 2500 μV (0.06 μV)
Gas system with maximum carrier gas amount	1000ml/min
Programmable heating rate cooling unit included	0.1^{0}C/min —50^{0}C/min

Samples were scanned between room-temperature to max 1200^{0}C with heating rate of 10^{0}C/min to 30°C/min,~~and~~ one run up to 1260°C was also performed to check the formation of any melt. The experiments were conducted in air, nitrogen, argon and oxygen atmosphere with the desired flow rate (100-300 ml/min). Around 8-10 mg sample was used in each run. Alpha-alumina of equal weight was used as reference material. The results were further analyzed for desired temperature range, as per thermal events exhibited using instrument software for temperature, ΔT, weight change and its rate. Figure 3.2 shows the photograph of the thermal analyser system (Sieko 5100-32).

Fig. 3.1 Rigaku-D.Max x-ray diffractometer unit

Fig. 3.2 Sieko 5100-32 thermal analysis unit

3.2.5 Evolved gas testing:

Analysis of the evolved gases is desirable in understanding the decomposition reactions better. As this facility is not available with present thermal analysis system, gases evolved were tested, especially for its chlorine gas and chloride vapours, by heating in a tube furnace of 0.02 m diameter and 0.6 m length at 950^{0}C in a ceramic boat. About 4 gram of sample was used. The gas coming at other end of tube is bubbled through distilled water. Argon, oxygen, nitrogen and air was used as carrier gas with a flow rate just enough to create reasonable bubbling at other end. The bubbled distilled water was tested for its chlorine content by silver nitrate test for appearance of white precipitate. Figure 3.3 shows the set-up for evolved gas analysis.

3 2.6 True density measurement:

The true density of fine powder and products of heating, at different temperature was measured by specific gravity bottle using kerosene as medium as per standard. (IS: 5483 -1969)

fine [illegible]

3.3 Preparation of Pellets:

3.3.1 Hand rolled pellet::

The synthesized powder was mixed with binder (1% dextrin) and appropriate quantity of water. Pellets of different sizes were hand rolled. Care was taken to get uniform How porosity, good strength and smooth surface by prolonged rolling of the pellets with finger tips and then on a glass plate. Hand rolling is preferred to get good spherical shape and for the limited quantity of material available. These

Fig. 3.3 Set-up for evolved gas testing/analysis

pellets were dried and then fired at 910°C for 90mins and allowed to cool in the furnace itself. The radial hole of 0.1cm diameter was drilled up to the centre of the pellet.

3.3.2 Pelletization of ore :

The ore powder was pelletized in a disc pelletizer of 0.6m diameter and 0.15m lip height. One percent dextrin was used as binder. The overall rolling time was about 90 min. The green pellets of size 10-16 mm and spherical shape were picked, dried at 150-200°C for 120min and then fired at 950°C for 60 min to attain workable strength of pellet. These pellets were furnace cooled. A radial hole of 0.1cm diameter was drilled up to the centre of pellets. The pellets were subsequently fired at temperatures of 1030, 1120,1200,1215,1250 and 1275°C for 1 hr in a silicon carbide tube furnace and in platinum wound tube furnace for higher temperatures.

3.3.3 Pressed pellet:

It was difficult to prepare pellets having porosity below 20-22% by conventional method, even by firing at high temperature. Thus they were made by following method:

1. Briquette the ore powder to cylindrical shape with equal length and diameter using punch and die. The pressure applied was 560 Kg/cm^2 to 5600 Kg/cm^2
2. Slow heating to 900^{0}C and then cool.
3. Carving the pellet to spherical shape
4. A radial hole of 0.1cm diameter was drilled up to centre.

5. The pellets re-fired at desired temperature of 950°C to 1200°C.

Sharma and Gupta[12] used a similar technique to prepare low porosity pellets in their swelling index studies. Attempts were also made to stabilise the ore powder before pressing by preheating it to decompose the associated minerals and gangue, However the briquettes could not be made from this powder as the powder lost its compactibility.

3.4 Pellet Characterization:

3.4.1 Size measurement:

Size of the pellet was measured by making random measurement of pellet diameter using digital micrometer. Average of 15 to 20 such reading is taken as the size of pellet.

3.4.2 Density measurement:

The bulk density of pellets was measured by weight and calculated volume of pellets. A few pellets were tested for density by immersion test. in Shah ? The result obtained was comparable.

3.4.3 Porosity measurement:

The bulk porosity of fired pellet was determined by

$$\% \text{ Porosity} = \frac{\text{True density of fine powder} - \text{Bulk density of pellets}}{\text{True density of fine powder}} \times (100)$$

3.4.4 Measurement of thermal diffusivity and conductivity

The method used is similar to one suggested by Watt's et al, but with modifications. and a different analysis procedure. The method is selected for its suitability to spherical geometry and radial heat transfer. Watt et al measured the temperature at half radius and at the centre of the pellet and applied transient state conduction equation to evaluate diffusivity. Positioning of a thermocouple at centre and at surface of pellet is more reliable and easier for its accurate positioning as compared to placing at any radial distance. It was reported [48] that a ten percent error in positioning of thermocouple at half radial distance gives about twenty percent error in thermal diffusivity values due to sharper temperature profiles. On the other hand, the temperature profile near centre is almost ~~flatter~~ flat, ~~to~~ minimising such errors. Moreover, it is not desirable to insert two thermocouples in the pellet body specially when the effect of bulk density and porosity needs to be investigated. In view of the above facts, the surface and centre temperature were measured and results were analysed by applying principles of heat balance rather than non-isothermal heat equation suggested by Watts

Why?

Radiation effects

In the present set up the pellet is fixed between three chromel-alumel thermocouples so that one thermocouple penetrates the pellet through the drilled hole with its bead at the centre. The other two thermocouples are diagonally opposite to the pellet diameter with their beads touching the surface. The insulators are inserted to the wires keeping certain distance from pellet surface. The short circuit between wires is avoided by coating the wire with a thin layer of iron oxide (slurry/dried) and maintaining a safe distance between two wires. Loose iron ore powder was filled in the centre hole to avoid any air gap between thermocouple and space of hole. Both the outer thermocouple were tied to a thin walled stainless steel tube containing centre thermocouple and clamped. Figure 3.4 indicate the set up for thermal diffusivity measurement.

An independently controlled, preheated to about 700 ^{0}C temperature, tube furnace ~~was lowered on the~~ pellet and the rise in surface and centre temperature of the pellet were recorded by two pen strip chart recorder at a chart speed of 10 cm/min. Relationship between surface temperature and centre temperature was also recorded using an omniscribe X-Y recorder with accurate scale (1cm = 1mv). As tube furnace does not produce an isothermal reservoir but establishes, a temperature plateau some where at the centre of the tube, hence its design was modified by using reflectors above and below the pellet location. These reflectors are made of stainless steel and of almost the same diameter as the furnace tube. Their use decreases the convective air inspiration, from the bottom of the furnace and also provides configuration similar to an enclosed chamber. It also increases the length of the hot zone and variation of temperature within it is decreased. This is also helpful in maintaining the pellet position at the centre of furnace tube as the reflector acts as a guide. The direct radiation from furnace wall to thermocouple bead was avoided by placing a dome shaped metallic shield on the lower reflector to envelop the pellet. This decreases the lateral temperature gradient and hence allows to measure surface temperature more accurately, The heat flux passing through the thermocouple wire is likely to cause some error in evaluation of thermal

[illegible]

[illegible]

Fig. 3.4 Set-up for measurement of thermal diffusivity

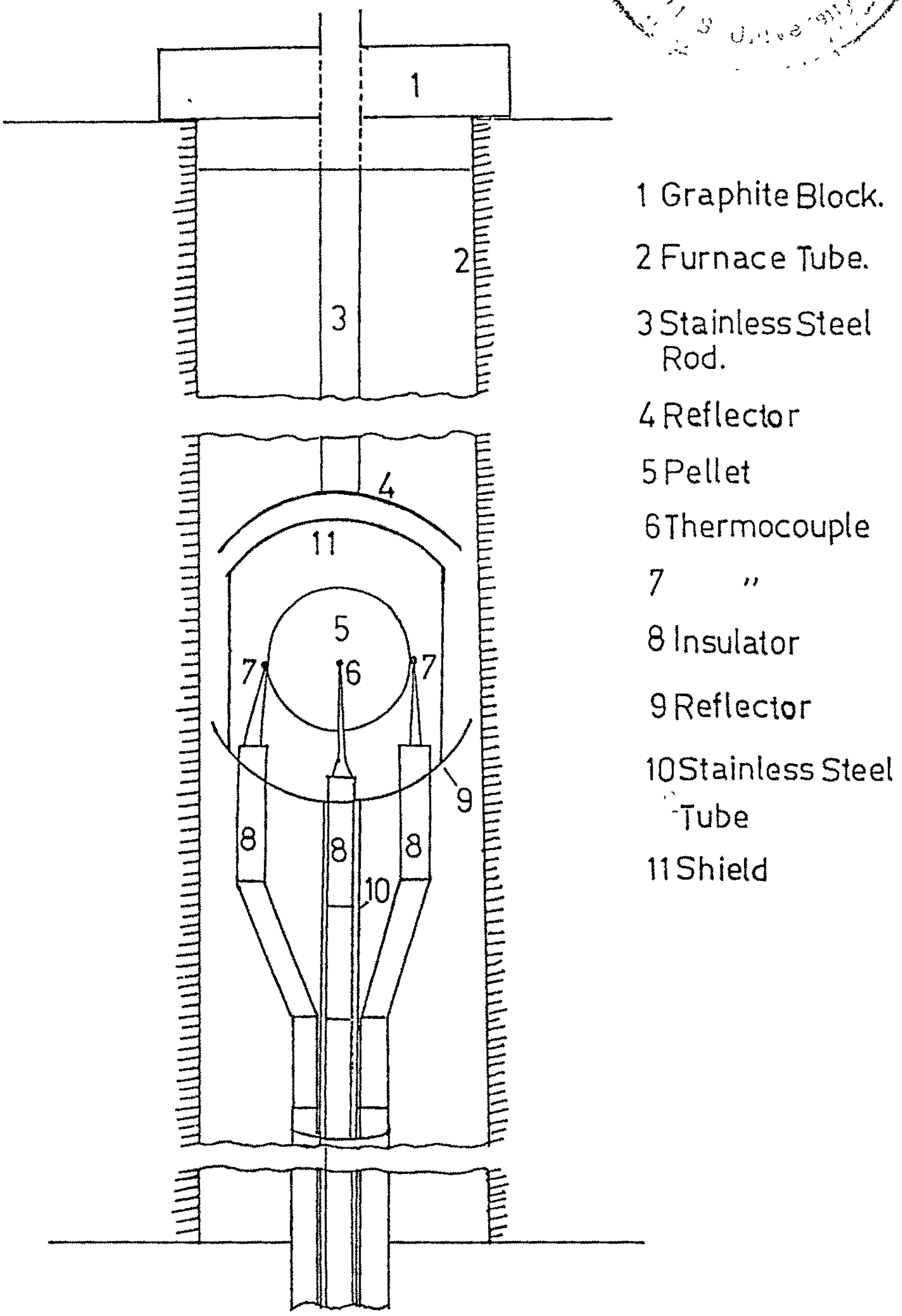

Fig. 3.5 Pellet configuration and location of thermocouples

diffusivity. Assuming the thermal contact between the pellet and wire to be sufficiently good to produce similar temperature gradient, it was reported that ~~an~~ errors of about 2.5% in thermal diffusivity value [47,48]. Same order of an error may be anticipated in the present case. Figure 3.5 indicate the location of pellet and thermocuples.