CHAPTER-3:

EXPERIMENTAL PROCEDURE
3. EXPERIMENTAL PROCEDURE

This chapter presents the experimental set up used to carryout characterization of the samples, granulometry studies and pellet firing studies. Experimental procedures followed to measure the pellet quality and parameter levels for different variables are also presented in this chapter.

3.1 Experimental plan

To investigate the green pelletizing characteristics of the Noamundi region iron ore fines, the effect of different fluxes on the formation of phases and microstructure during the pellet induration and their subsequent effect on the pellet quality, detailed experimental plan was prepared. The study also investigates the effect of iron ore fineness on the green pellet formation and quality. It was planned to apply the findings of the experimental results to establish right pellet chemistry for the recently commissioned 6 MTPA capacity iron ore pelletizing plant at Tata Steel, Jamshedpur in India.

3.1.1 Iron ore fines sample from mines

Two tonnes of representative pelletizing grade iron ore fines (0 to 10 mm size) sample was received from Noamundi captive iron ore mines. After coning and quartering, as shown in Fig.28, 250 kg composite sample was obtained from the bulk sample.

3.1.2 Laboratory experiments

Characterization of iron ore fines was carried out using QEMSCAN microscope to estimate the proportion of different minerals and their liberation size. XRD and TGA analysis of iron ore and different fluxes, used for the pelletizing, was carried out to qualitatively determine different phases and their behaviour during induration.

Grinding of iron ore fines to get the desired fineness for pelletizing was carried out in 70 litre capacity laboratory ball mill. Turbo mixer was used to
prepare the pelletizing mixture and green pellets were prepared in the prepared using a laboratory balling disc with a diameter of 600 mm. Green pellet firing was carried out in laboratory rotary hearth furnace of 3 meters diameter. Fired pellets were tested for cold compression strength (CCS), reduction degradation index (RDI), reducibility index (RI), swelling index and softening-melting characteristics.

Microstructural studies were carried out using optical microscope with image analyser software and electron microscope to identify different phases formed. Semi quantitative EDX analysis of different phases in the microstructure was carried out to estimate their chemical analysis. X-ray mapping technique was also used to know the distribution of different elements in the microstructure.
Fig. 28 Iron ore sample preparation
3.1.3 Experimental set-up and procedure

Characterization of the iron ore fines was carried out using state-of-the-art QEMSCAN microscope. QEMSCAN is an automated instrument based on Scanning Electron Microscope principles attached with EDS (Energy Dispersive Spectrometer) and BSE (Back Scattered Electron) detector. A suit of state-of-the-art software takes care of data acquisition and report generation. The operational parameters are acceleration voltage: 25kV, electron beam current: 5.00nA and electron beam diameter: ≈2micron.

As received iron ore sample was screened to separate +3 mm fines from the minus 3 mm fines. The +3mm was crushed in a roller crusher at 3 mm gap to generate -3mm fines. These fines were ground in the laboratory ball mill of 70 litres capacity. The ball to charge ratio was kept 1:4 in all the tests. Each batch for grinding consists of 4 kgs of ore fines and 16 kgs of grinding media. To study the effect of grinding time and to generate ground ore with different fineness, grinding time was varied from 2 to 5 hours in the interval of 1 hour.

Ground ore was mixed with the binder, solid fuel and flux in laboratory turbo mixer in dry form. Mixed powder was wetted with water up to about 6% wt.% and passed through 3 mm screen to obtain uniformly mixed granules. Green pellets were prepared using the laboratory balling disc, with a diameter of 600 mm, an edge height of 200mm and a tilting angle of 45 degrees at 27 rpm. During balling, green pellets were screened with 10 mm and 12.5 mm screens to get 10-12.5 mm pellets. In all the pelletizing studies, bentonite content was fixed at 0.8% with respect to dry pelletizing mixture. Green pellets were tested for their drop strength, green compression strength (GCS) and moisture content. Before firing, the pellets were dried in the hot air oven at 150°C for 5-6 hours to ensure that all the moisture is removed.

Firing of the pellets was carried out using electrically heated rotary hearth furnace (RHF) as shown in Fig.29. RHF consists of five heating zones and one cooling zone. All the zones are provided with air inlet nozzles to inject
air for creating oxidizing atmosphere during induration. The counter current reactions between the hot pellets and air facilitate the complete oxidation of admixed coal in the green pellets. Temperature profiles in each zone were maintained to simulate the firing conditions in the industrial pellet indurating machine. Ceramic wool insulated stainless containers and also Inconel wire trays were used for firing the pellets in the RHF. Photographs of pellets before firing, during firing and after firing are shown in Fig.30.

Cold compression strength (CCS) of the fired pellets was tested as per ISO 4700. For CCS testing, 60 pellets of 10 to 12.mm size are tested by loading the single pellet under compression force between surface hardened platens at the rate of 15mm/min. CCS was recorded as maximum load at which pellet breaks completely and reported as mean value for the 60 pellets tested.

Reduction degradation index (RDI) was tested as per ISO 4696-2. 500 grams of pellets in the size range of 10-15 mm were isothermally reduced in a vertical retort furnace, as shown in the schematic diagram in Fig.31, at 550°C for 60 minutes in the gas containing 30% CO and 70% N₂ supplied at 15 litres per minute. After cooling the reduced sample, the same was tumbled in a drum of 200mm length of 130 mm diameter for 30 minutes at 30 rpm. After tumbling, the sample was screened at 3.15 mm and %of -3.15mm is reported as RDI.
Fig. 29 Rotary hearth furnace used for firing the pellets

Fig. 30 Photographs of pellets before, during and after firing in RHF
Fig. 31 Schematic diagram of RDI apparatus for pellet testing
Reducibility index (RI) of the pellets was tested as per ISO 7215. 500 grams of pellets in the size range of 10-15 mm were isothermally reduced at 900°C for 180 minutes in the gas containing 30% CO and 70% N₂ supplied at 15 litres per minute. RI is calculated as per the following formula:

\[
\text{Reducibility Index, (RI)}\% = \frac{\text{Wt. Loss,} \%}{(\text{Fe-Fe in FeO}) \times 48 + \text{FeO} \times 16} - \frac{112}{72}
\]

Swelling index (SI) of pellets was measured as per ISO 4698. 18 pellets of 10-12.5 mm size are measured for their volume to record the volume before reduction \( (V_i) \). Then they are placed in Inconel basket in 3 layers and heated in inert atmosphere up to 900°C. At this temperature, pellets were reduced for 60 minutes in the gas comprising 30% CO and 70% N₂ supplied at 15 litres per minute. After cooling the reduced pellets in inert atmosphere, volume was measured to record the volume after reduction \( (V_f) \). The SI is calculated as following:

\[
\text{Swelling Index} = \frac{V_f - V_i}{V_i} \times 100
\]

**Figure 32** shows a schematic diagram of apparatus used for softening-melting test with the description of test procedure.
Fig. 32 Schematic diagram of Softening-Melting testing apparatus

**Softening-Melting test procedure**

<table>
<thead>
<tr>
<th>Parameter</th>
<th>Specification</th>
</tr>
</thead>
<tbody>
<tr>
<td>Sample size</td>
<td>20-12.5 mm</td>
</tr>
<tr>
<td>Sample bed diameter</td>
<td>45 mm</td>
</tr>
<tr>
<td>Bed height</td>
<td>100 mm</td>
</tr>
<tr>
<td>Applied load, kN/m²</td>
<td>98 (from 500°C to end of test)</td>
</tr>
<tr>
<td>Gas flow rate</td>
<td>6 L/min</td>
</tr>
<tr>
<td>Gas composition</td>
<td>100% N₂ up to 200°C, 70% N₂ and 30% CO from 200°C to 1600°C</td>
</tr>
<tr>
<td>Maximum test temperature</td>
<td>1600°C</td>
</tr>
<tr>
<td>Heating rate</td>
<td>20°C/min up to 900°C, 3°C/min from 900°C to 1200°C, 5°C/min from 1200°C to 1600°C</td>
</tr>
<tr>
<td>Reporting of test results</td>
<td></td>
</tr>
<tr>
<td>1. Temp. at which gas pressure starts to increase (T₁)</td>
<td>Softening start temp. at 100 mmHg</td>
</tr>
<tr>
<td>2. Temp. at which ΔP reached maximum (T₂, max)</td>
<td></td>
</tr>
<tr>
<td>3. End of melt down temperature (T₃)</td>
<td></td>
</tr>
<tr>
<td>4. Softening Melting range (T₁-T₃)</td>
<td></td>
</tr>
<tr>
<td>5. Maximum pressure drop (ΔP₃)</td>
<td></td>
</tr>
<tr>
<td>6. Residual obtained after test</td>
<td></td>
</tr>
</tbody>
</table>
Advanced RDI tests on optimized pellet samples were conducted to simulate the conditions in the stack zone of the blast furnace according to the operating line of the blast furnace. A 500 gram sample was heated according to a predetermined temperature/time profile with corresponding reduction gas composition up to 900°C. The gas composition was changed according to a RIST operating line and was thus dependent on the progress of reduction. After reduction the sample was subjected to a tumbling test. The test results were reported as; a) time required for indirect reduction b) disintegration percentage <3.15 mm after tumbling.

Advanced swelling and softening tests on optimized pellets were carried out to understand the behaviour of pellets near the cohesive zone of the blast furnace. A 500 gram sample was heated in a nitrogen atmosphere to a temperature of 950°C. The sample was then isothermally reduced at 950°C in a gas atmosphere of 40% CO and 60% N₂ until the O/Fe level is 0.375. In the next stage the sample was softened under nitrogen by linearly raising the temperature to 1200°C. The sample temperature, the pressure drop across the bed, the bed shrinkage and the weight of the sample were continuously measured. The test results are reported as; a) reduction time b) delpa P across the bed at the end of reduction c) Softening temperature, T₁₀₀, i.e. temperature at pressure drop is 100 mm WC.

Advanced swelling tests were carried out on the optimized pellet samples to understand the swelling tendency of pellets at high temperature. 10 pellets were reduced for 30 minutes and 90 minutes at 1050°C in a reducing gas of 100% CO. The test results are reported as; i) ΔV after 30 min (%) ii) ΔV after 90 min (%).

For micro structural studies three fired pellets from each batch were collected representatively. Pellets with more irregular and non-uniform shape were avoided for the microstructure study as they experience uneven heat treatment across their cross section. Microstructural study of the these pellets
was carried out using optical microscope (Axioplan2) with image analyser software and electron microscope (JEOL JXA-6400). Semi quantitative analysis of different phases in the microstructure was carried out using KEVEX super dry, EDX detector. X-ray mapping technique was also used to know the distribution of different elements in the microstructure.

Image analysis technique was used to estimate the amount of phases formed in the fired pellets. Image analysis is a technique that is used to provide an objective measurement of different phases in microstructure. Pellet samples were cut into half and hot mounted at 175°C temperature and 90 daN load for 14 minutes using a conductive resin. Once sample was mounted and polished, as shown in Fig.33, it was placed under the Zeiss- Axioplan2 microscope for examination. A black and white CCD digital camera with a maximum resolution of 756 X 581 pixels was mounted behind the lens of the microscope to capture the light reflected from the sample. A 10 X eye piece and 20X objective lens on the microscope has been selected for the current study. At this level of magnification, the view frame on the sample surface is approximately 710 X 530 μm.

The signal from the camera was provided to a personal computer through a gain correction amplifier to correct the signal for optimal display. The computer software used for interpreting the camera signal into digital image was the Axiovision 4.7 Imaging System supplied by Carl Zeiss Vision. Basically, the digital image captured from the black and white camera is represented by pixels having 256 shades of grey values i.e. 0 to 255 [44]. The lower range of grey values represents pores and the oxide grains represent higher range values. A digitized black and white photograph is transformed into a segmented image with the specified range of grey values to different phases. Once the image has been processed, image analysis tools from the Axiovision software were used to measure the area fraction of different phases in the microstructure.
Fig. 33 Mounted and polished pellet samples for microstructural study
3.1.4 Parameter levels for laboratory investigation

3.1.4.1 Characterization of iron ore samples

As received iron ore fines were screened for into different size fractions and QEMSCAN mineralogical analysis was carried out for the following fractions to estimate the mineral mass and grain size; +12.5 mm, +10 mm, +8 mm, +6 mm, +3 mm, +1 mm, +0.5 mm, +0.15 mm, +0.045 mm and minus 0.045 mm. Liberation pattern for hematite & goethite and deportment of Fe and Al was measured for all the above mentioned fractions.

Ground iron ore fines were screened for into different size fractions and QEMSCAN mineralogical analysis was carried out for the following fractions to estimate the mineral mass and grain size; +2.36 mm, +1.44 mm, +1.0 mm, +0.25 mm, +0.15 mm, +0.075 mm, +0.063 mm, +0.045 mm, +0.037 mm and minus 0.037 mm. Liberation pattern for hematite & goethite and deportment of Fe and Al was also measured for all the above mentioned fractions.

3.1.4.2 Grinding & granulometry studies of iron ore samples

Effect of grinding time on the particle size distribution (PSD), mean particle size (MPS) and \( P_{80} \) of iron ore samples was carried out in the laboratory ball mill. Grinding time was varied from 1 to 5 hours in the interval of 1 hour. These studies were helpful to generate required fineness for the subsequent green pelletizing studies.

3.1.4.3 Green pelletizing studies

Pelletizing behaviour of iron ore with different fineness was modelled by constructing self-preserving curves. Fineness was varied in terms of minus 45 microns ~ 60%, 65%, 70%, 75% and 80%. Green pellet quality was estimated for different fineness from 60 to 80% passing 45 microns, in terms of drop number, green compression strength and moisture content.
3.1.4.4 Pellet firing studies

Green pellets were fired at three different temperatures, viz., 1250°C, 1275°C and 1300°C to find out the optimum firing temperature. To find out the relation between firing temperature and fineness of the pelletizing feed, pellets made from varying fineness, viz., 26 microns, 38 microns, 55 microns and 70 micron MPS were fired at the above mentioned firing temperatures.

3.1.4.5 Effect of fluxes on the pellet quality

To study the effect of limestone on the pellet quality, green pellets were prepared at varying basicity (CaO/SiO$_2$) ranging from 0, 0.2, 0.4, 0.6 and 0.8. Limestone content in the green pellets was varied from 0.5 to 3.0 wt%. In all the pellets, fixed carbon was maintained at 1.0 % (as maintained in the commercial pellets) by adding anthracite coal.

To study the combined effect of CaO & MgO on the pellet quality, pellets were prepared with 1.5% MgO and basicity ranging from 0, 0.2, 0.4, 0.6 and 0.8. Dolomite content in the green pellets was varied from 2 to 6 wt.%.

To study the effect of magnesite on pellet quality, pellets were prepared with varying MgO contents ranging from 0.5% to 3.0% in the intervals of 0.5%. Magnesite content in the green pellets was varied from 1 to 7 wt.%.

To study the effect of pyroxenite on pellet quality, pellets were prepared with varying MgO contents ranging from 0 % to 1.5% in the interval of 0.5%. Pyroxenite content in the green pellets was varied from 0 to 5 wt.%. To further fine tune the MgO content, pyroxenite fluxed pellets were prepared with MgO content ranging from 0.3, 0.6, 0.9, 1.2, 1.5 and 2.0% and these pellets were tested for their advanced metallurgical properties.