

Production of Pre-Reduction of Iron Ore Nuggets with Lesser Sulphur Intake by Devolatilisation of Boiler Grade Coal

Chanchal Biswas, Anrin Bhattacharyya, Gopes Chandra Das, Mahua Ghosh Chaudhuri, Rajib Dey

Abstract—Boiler coals with low fixed carbon and higher ash content have always challenged the metallurgists to develop a suitable method for their utilization. In the present study, an attempt is made to establish an energy effective method for the reduction of iron ore fines in the form of nuggets by using 'Syngas'. By devolatilisation (expulsion of volatile matter by applying heat) of boiler coal, gaseous product (enriched with reducing agents like CO, CO₂, H₂, and CH₄ gases) is generated. Iron ore nuggets are reduced by this syngas. For that reason, there is no direct contact between iron ore nuggets and coal ash. It helps to control the minimization of the sulphur intake of the reduced nuggets. A laboratory scale devolatilisation furnace designed with reduction facility is evaluated after in-depth studies and exhaustive experimentations including thermo-gravimetric (TG-DTA) analysis to find out the volatile fraction present in boiler grade coal, gas chromatography (GC) to find out syngas composition in different temperature and furnace temperature gradient measurements to minimize the furnace cost by applying one heating coil. The nuggets are reduced in the devolatilisation furnace at three different temperatures and three different times. The pre-reduced nuggets are subjected to analytical weight loss calculations to evaluate the extent of reduction. The phase and surface morphology analysis of pre-reduced samples are characterized using X-ray diffractometry (XRD), energy dispersive x-ray spectrometry (EDX), scanning electron microscopy (SEM), carbon sulphur analyzer and chemical analysis method. Degree of metallization of the reduced nuggets is 78.9% by using boiler grade coal. The pre-reduced nuggets with lesser sulphur content could be used in the blast furnace as raw materials or coolant which would reduce the high quality of coke rate of the furnace due to its pre-reduced character. These can be used in Basic Oxygen Furnace (BOF) as coolant also.

Keywords—Alternative ironmaking, coal devolatilisation, extent of reduction, nugget making, syngas based DRI, solid state reduction.

I. INTRODUCTION

THE availability of coking coal is decreasing rapidly worldwide. [1] The steel industries are trying to incorporate non coking coal as an alternative reductant. [2] Direct reduction of iron ore oxide using non coking coal increases the amount of sulphur percentage in the product due to high percentage of ash present in non coking coal. [3] The high sulphur content in the reduced iron is difficult to remove

Chanchal Biswas, Gopes Chandra Das, and Rajib Dey are with the Department of Metallurgical and Material Engineering, Jadavpur University, Kolkata, India (phone: +919733587845, e-mail: chanchal18@mail.com).

Anrin Bhattacharyya is the Chair of Ferrous Metallurgy, Montanuniversität Leoben, Leoben, Austria.

Mahua Ghosh Chaudhuri is with the School of Materials Science and Nano Technology, Jadavpur University, Kolkata, India.

in secondary steel making process. [4] In ambient temperature the solubility of sulphur in α -iron is up to 0.002% and in γ iron at high temperature (1000 °C) it is up to 0.013%. So it is very difficult to remove sulphur from liquid steel. During solidification of liquid steel, in presence of sulphur, a low melting phase of iron sulphide is formed. This is deposited between grain boundaries of steel. The formation of iron sulphide decreases the steel quality [5]. The sulphur is removed in desulphurization process. Sulphur exhibits negative deviation from Henry's law in molten iron. Sulphur bonds strongly with iron [6]-[8]. During the solidification of liquid steel, sulphur and iron form FeS eutectic which is segregated at the iron grain boundaries and it decreases the steel properties [9], [10]. The presence of C, Si and P increases with increase of activity coefficient of sulphur. The desulphurization process is time and cost intensive. Modern iron and steel industries require improvement in quality, efficient production and techno-economic feasibility [11]. As per modern industrial needs, minimizing the sulphur content during the direct reduction process is of importance.

The purpose of the present study is to minimize the sulphur percentage in the reduced sample by coal devolatilisation process by avoiding direct contact between iron ore nuggets and coal (which is the major carrier of sulphur). In this process, the iron ore oxide is pre-reduced by volatile matter content of the boiler grade coals. The advantage of pre-reduction of iron ore briquette by coal gasification is to lower sulphur content due to absence of direct contact between coal and iron ore. The pre-reduced nuggets could be used in the blast furnace itself either as a coolant or simply as a raw material which would minimize the high quality of coke rate of the furnace because of its pre-reduced by boiler grade coal [12]-[16]. Thereby the quality and productivity of steel are not compromised even by using lean grade coal. Other problem of iron making is that the raw material fines have considerable adverse impact on environment and for that reason their proper utilization to achieve sustainability of the environment has always been a challenge for the metallurgists. Nearly, 10-12% of the fines are generated while converting lumps into calibrated lump ore (CLO). On an average, it has been found that nearly 2.5 ton of iron ore lumps are required to generate 1 ton of CLO [17], [18].

Nuggets are made from the agglomerated inferior quality iron ore fines by using binder with the applied pressure. Gasifier cum vertical shaft furnace is used to perform the gasification of the coal and reduction of iron ore briquettes.

Tetsuo Horie and Makoto Shimizu investigated a process control with gasification of coal and the reduction of iron oxide took place simultaneously in a reactor. Fluidized bed is selected for the type of reactor from initial study on reduction time at 800 °C. They achieved higher degree of metallization using coking coal. [19]

James Burgess investigated underground coal gasification technology (UCG) effectively to convert coal fields into gas fields. This gas is directly used for reduction of iron ore into pig iron production at lower cost. [20] In the Midrex plant, the cold syngas is first depressurized to about 3 barg. After that, low pressure syngas is mixed with recycled gas to produce the required reducing gas mixture [21]-[23]. This mixed syngas is then heated to more than 900 °C. The hot reducing gas mixture enters the shaft furnace where it reacts with the iron ore to produce DRI.

II. MATERIALS AND METHODS

A. Materials Used

- **Iron Ore:** Hematite iron ore fines are supplied by NMDC from Bachel & Kirandol Mines of Baliadila Complex at Chhattisgarh, India.
- **Boiler Coal:** Boiler coal is obtained from Talcher & IB Valley sources of Mahanadi Coal Field (MCL), India.
- **Molasses:** Molasses are obtained from Tata steel plant, Jamshedpur, Jharkhand, India.
- **Bentonite:** Bentonite is collected from JSW Steel, toranagallu, Bellary, Karnataka, India.

B. Sample Preparation

In present work, below 10 mm size fractions of hematite are crushed by jaw crusher, roll crusher and ball mill. They are then subjected to sieve analysis to get of particles with different sizes. Three ranges of size fractions are selected for nuggets, coarse (between 0.5 mm and 0.178 mm), semi coarse (between 0.178 mm and 0.104 mm) and fines (between 0.104 mm and 0.044 mm) by BSS standard mesh size. Boiler grade coal is crushed by jaw crusher. The size fraction of charged boiler grade coal is 6.3–19 mm. The details of the different samples are summarized in Table I.

TABLE I
DETAILS OF THE DIFFERENT SIZE FRACTION SAMPLES

Sample	Size fraction of the samples		
	Fines	Semi Coarse	Coarse
SE1	60	20	20
SE2	20	60	20
SE3	20	20	60

C. Making Nuggets

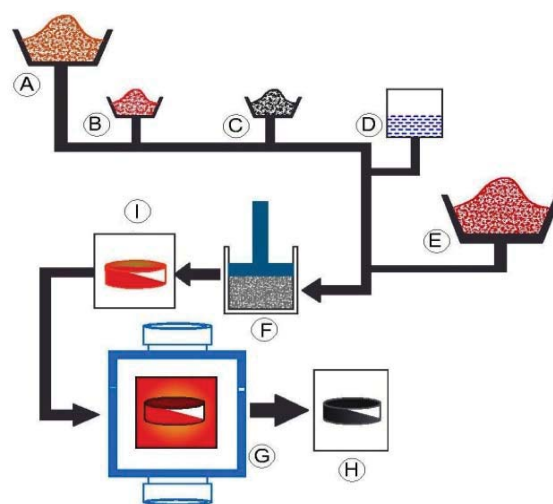
Nugget basically contains 4% bentonite, 4% molasses, 4% moisture and the rest iron oxides. All ingredients are taken in their proper weight percentage. Then, they are mixed thoroughly to get a homogenous mixture as far as possible and are cured overnight before making nuggets. 12.5 grams of that mixture is taken into the cylindrical die. The die along with sample is placed in the hydraulic press (hand operated). The

pressure applied to the die is 350 kg. The die is kept in rest for a while (1 minute). Then, the sample is removed and separated. Cylindrical shape is formed. These nuggets are charged in coal devolatilisation furnace for reduction.

D. Characterization of Raw Materials and Reduced Samples

Proximate analysis of boiler grade coal is also carried out and the results are compared with the TGA/DTA results. Raw ore and reduced specimens are characterised using XRD, (Rigaku Ultima-III, Cu K α , 40 kV, 30 mV). The positions and intensities of peaks are compared by PCPDF-WIN software with JCPDS International data. The Wavelength Dispersive X-ray Fluorescence (WDXRF) is used to quantify raw ore composition by MagiX2424, PANalytical Super Q. The surface topography, composition of raw ore and reduced samples are analysed by SEM and EDX (SEM and EDX model no-JEOL JSM-8360 and EDX oxford instrument). Boiler grade coal and coke dust fines are characterised by TGA/DTA (Perkin-Elmer instrument, model-Pyris diamond TG-DTA) in the range of 37 to 900 °C at the rate of 10 °C/min in N₂ atmosphere. GC analysis model no CHEMETO-8610 is used for gas analyses which are generated from coal volatilization of boiler grade coal. Shatter and abrasion index of the nuggets are obtained in the standard shatter and abrasion lab scale apparatus before and after the reduction. The extents of reduction are calculated from the weight loss method. Amount of sulphur and carbon are obtained during pyrolysis process for reduced samples using Horiba EMIE 320 V, made in Japan. The chemical analysis of the reduced samples is evaluated by the standard procedure of the Bureau of Indian Standards (BIS 10812:1992).

The flow diagram of the total process to find out the reduction of nuggets is given in Fig. 1.



(A) Iron Ore (B) Bentonite (C) Boiler Grade Coal (D) Moisture (E) Mixture of all Ingredients (F) Die and Piston (I) Nuggets (G) Coal Devolatilization Process (H) Pre-Reduced Nuggets

Fig. 1 Flow diagram of inter the process

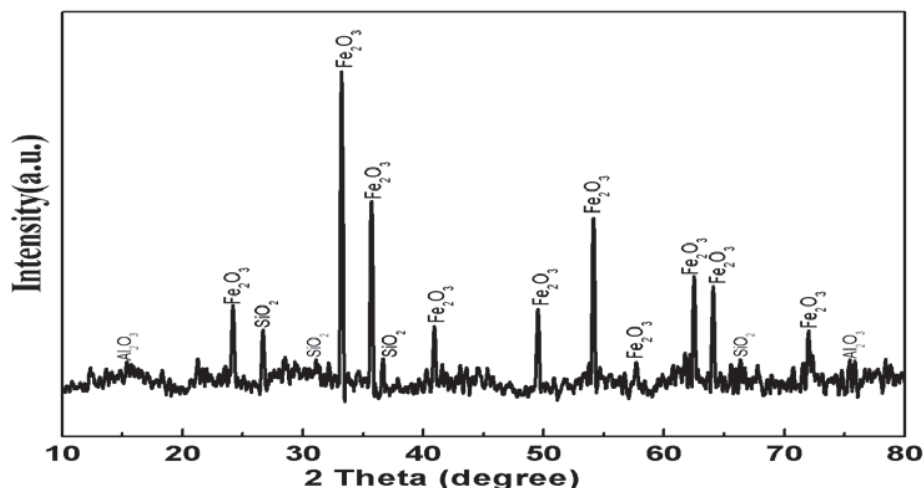


Fig. 2 XRD pattern of the raw ore sample

TABLE II
WDXRF RESULT OF IRON ORE

Fe (T)	SiO ₂	Al ₂ O ₃	MnO	MgO	CaO	Na ₂ O	K ₂ O	TiO ₂	S	P ₂ O ₅
64.05	4.03	2.29	0.05	0.28	0.36	0.02	<0.01	0.24	0.1	0.06

III. RESULT AND DISCUSSION

A. XRD Analysis of Iron Ore

XRD pattern of the raw iron ore sample is given in Fig. 2. It is observed that the major peak is of hematite (Fe₂O₃) along with minor peaks of alumina (Al₂O₃) and silica (SiO₂). The d values of the phases are matched with the standard JCPDS d values. [JCPDS – 85-0987, 84-1435, 86-1630, 86-1410].

TABLE III
PROXIMATE ANALYSIS OF BOILER GRADE COAL

Sample	Fixed carbon	Volatile matter	Moisture	Ash
Boiler grade coal	28.11 (%)	28.28 (%)	7.4 (%)	36.21 (%)

TABLE IV
ULTIMATE ANALYSIS OF COAL

Sl No	Parameters	Sample tagged as boiler grade coal
1	Total moisture % (as received basis)	2
2	Analytical moisture	7.3
3	Volatile matter % (air dry)	29.13
4	Ash % (Air dry)	33.05
5	Fixed Carbon % (Air Dry)	30.52
6	Sulphur % (Dry Air)	0.561
7	Ultimate analysis (Dry basis)	
	% Carbon	50.9
	% Hydrogen	3.47
	% Nitrogen	0.803

B. WDXRF Analysis of Iron Ore

From WDXRF data elemental analysis of hematite ore fines, total Fe content of the ore and major oxide concentrations are measured and is given in Table II in weight percentage (%).

C. Proximate and Ultimate Analysis of Boiler Grade Coal

The amount of fixed carbon, volatile matter, moisture content and ash percentage for boiler grade coal are calculated by proximate analysis and is given in Table III.

The data of proximate analysis are further refined by ultimate analysis (Leco Tru Spec) of boiler coal. The results of ultimate analysis of coal are given in Table IV.

D. TGA/DTA Analysis

Volatilisation properties of boiler grade coal obtained by TGA/DTA analysis are given in Fig. 3. The nature of the weight loss confirms the data from the proximate analysis of the boiler coal. The graph shows a weight loss up to 110 °C, which is caused due to the expulsion of the moisture (7-8%) in the coal. The major weight loss from 400 to 550 °C is due to the removal of the volatile fraction (25-30%) of the coal. This is the most important step during the devolatilisation. After 550 °C, the weight loss is very slow. This may occur due to soot formation or mild oxidation by the trace amount of oxygen present in nitrogen which is used as a shielding gas in the experiment. The derivative weight loss and derivative heat flow curve is shown Fig. 3. The loss around 375 °C may have arisen because of the burning of organic matter in the coal.

E. GC Analysis of Boiler Coal

GC Analysis of boiler grade coal is carried out to find the composition of the presence CO, CO₂, H₂, and CH₄ in the gas mixture. GC analysis of boiler grade coal is given in Fig. 4 at 650 °C. Syngas gas compositions at different temperature are given in Fig. 5. So, it can be inferred that hydrogen, carbon monoxide, carbon dioxide, and methane percentage of the reducing gas increases with temperature.

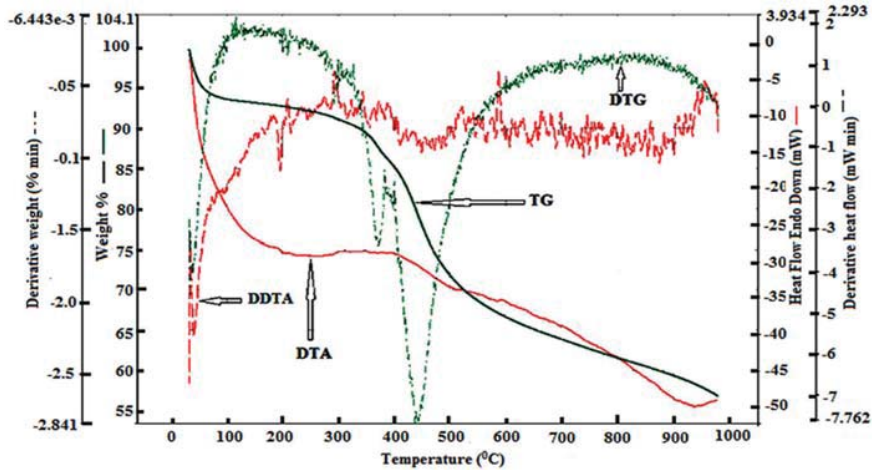


Fig. 3 TG/DTA plot of the boiler grade coal

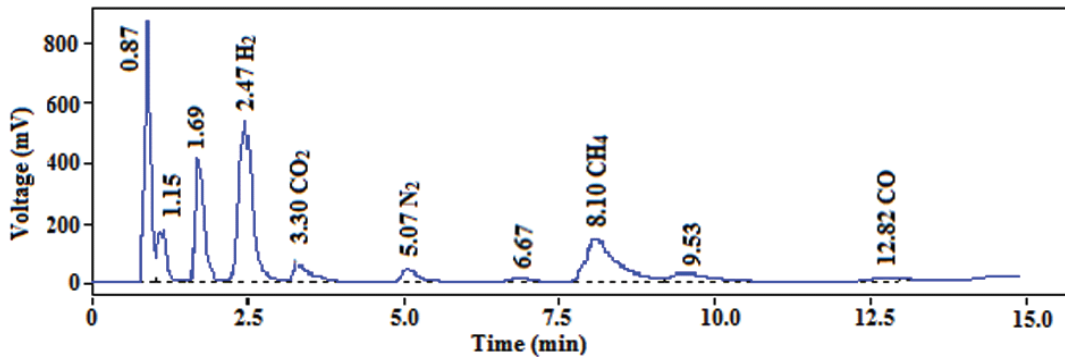


Fig. 4 GC analysis of boiler grade coal at 650°C

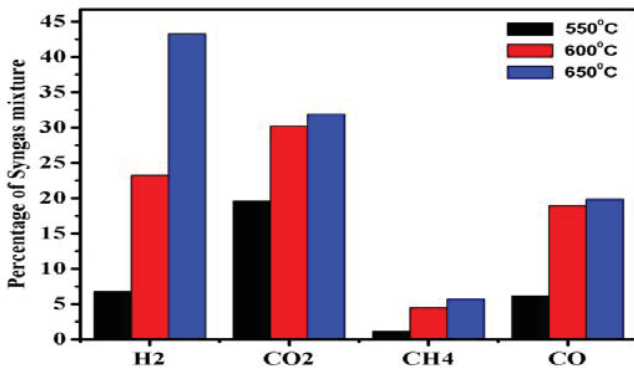


Fig. 5 Syngas mixture of boiler grade coal at different temperatures

B. Mechanical Properties of the Nuggets

All sets of nuggets are studied to assess their mechanical properties. Mechanical properties such as shatter index and abrasion index of each set of nuggets are given in Fig. 6. It can be concluded from Fig. 6 that the predominant fine particles combine to give lower shatter index and abrasion index. For Sample SE3, better results are obtained because of uniform distribution of fine particles in coarse particles. The reduction in void spaces leads to the betterment of mechanical properties.

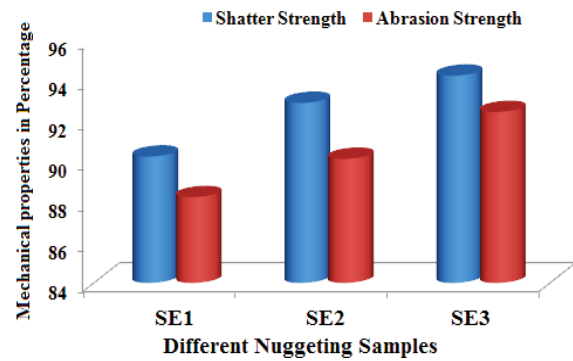


Fig. 6 Mechanical properties of the nuggets sample

C. Analysis of Carbon Soot

The carbon soot is generated in the course of pyrolysis of boiler grade coal is analyzed using XRD and FESEM techniques.

XRD and FESEM Analysis

XRD plot of carbon soot generated from the pyrolysis of boiler grade coal when heated at 650 °C is given Fig. 7. The plot does not show a sharp intensity peak for graphite. Instead, it shows a hump around 25° recommending the presence of fine amorphous carbon.

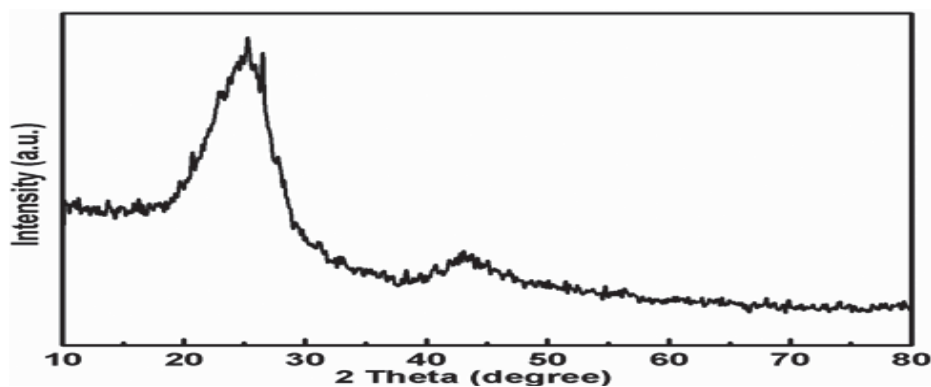


Fig. 7 XRD pattern of the carbon soot generated from the pyrolysis of boiler grade coal at 650°C

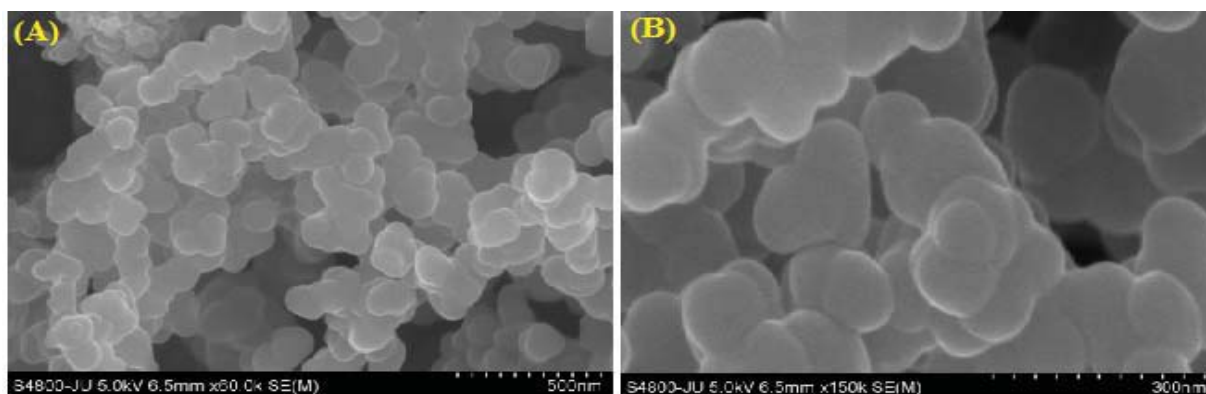


Fig. 8 FESEM micro photograph of the carbon soot at different magnifications (A) 500 nm (B) 300 nm

The FESEM image of the carbon soot is shown in Fig. 8. The images show agglomerated amorphous carbon particles with very narrow size distribution. This fine carbon is highly reactive for better reduction.

D. Coal Devolatilisation and Reduction of Iron Ore Nuggets

Gasifiers cum vertical shaft furnace is used to perform the devolatilisation and reduction together and is capable of gasifying boiler grade coal and reduce agglomerated iron ore at the same time using mixed reductant (hydrogen, carbon mono oxide and Nano-sized carbon soot particles) generated during thermal heating of the boiler grade coal. The furnace consists of only one heating element (at the reduction zone). The distance of the coal level is calculated from the heating element (using thermocouples). When the heating element attains the reducing temperature, the coal bed automatically reaches volatilisatation temperature at 650 °C without using another heating element, resulting in energy saving. By measuring the temperature gradient inside the shaft or tube furnace the required height of the furnace along with the position of heating elements and iron ore nuggets & coal are obtained. Nuggets are reduced at three different temperatures (1100, 1150 and 1200 °C) and three different times (60, 90 and 120 minutes). A new batch of coal is charged when the previous batch becomes fully devolatilized. In each batch an average of 60-65 g boiler grade coal is charged. This is the maximum amount of boiler grade coal that the pyrolysis zone can hold at 650 °C of the furnace. The size fraction of charged

coal is around 6.3-19 mm. After complete pyrolysis, the charred coal is taken out by the drop-bottom and a new batch is put into the furnace. The coal is charged five times during the 2 hour experiments (at an average interval of 24 minutes). The furnace is heated with the iron ore nuggets (kept on the wire mesh) up to 1200 °C (maximum) under atmospheric conditions. As soon as the temperature of the coal bed reaches 650 °C by the radiated heat from the reduction zone to maintain the temperature, the first batch of coal is charged. The furnace was designed for coal devolatilisation process, published in Journal [24]

E. Extent of Reduction of the Reduced Samples

The extents of reduction of pre-reduced samples are calculated from mass loss (calculated in percentage as the ratio of actual mass loss [M_1] to the mass loss in case of theoretically complete reduction [M_2]). This is calculated on the basis of weighing the sample after reaction. A theoretical calculation of mass loss for complete reduction is calculated beforehand, also taking account of other possible cases of mass loss (e.g. loss on ignition) to reduce their effect on final calculation. The ratio M_1/M_2 , when is calculated in percentage, gives an approximate quantitative idea about the extent to which the oxygen has been removed from the sample. It means that 100 % extent of reduction signifies a complete transformation from hematite to metallic iron. The detail of extent of reduction of coal devolatilization reduced samples is given in Table V and graphical presentations are

given in Fig. 9. The details calculation on extent of reduction is given Appendix 1.

TABLE V
EXTENT OF REDUCTION OF THE REDUCED SAMPLES OF DIFFERENT SIZE FRACTIONS AND REDUCTION TIMES IN 60, 90 AND 120 MINUTES AT 1200°C

Samples	Extent of Reduction (60 Minutes)	Extent of Reduction (90 Minutes)	Extent of Reduction (120 Minutes)
SE1	86.28	90.51	94.65
SE2	84.56	89.32	93.17
SE3	82.61	88.14	91.41

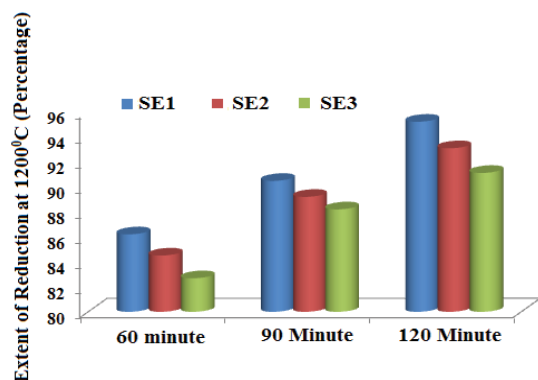


Fig. 9 Extent of reduction of nuggets of different size fraction at 1200 °C for reduced in 60, 90 and 120 minutes

TABLE VI
EXTENT OF REDUCTION AT 1150 °C FOR DIFFERENT TIMES AND SIZE FRACTIONS OF THE REDUCED SAMPLES

Samples	Extent of Reduction (60 Minutes)	Extent of Reduction (90 Minutes)	Extent of Reduction (120 Minutes)
SE1	83.65	88.17	92.09
SE2	81.32	86.81	90.24
SE3	80.07	83.91	89.54

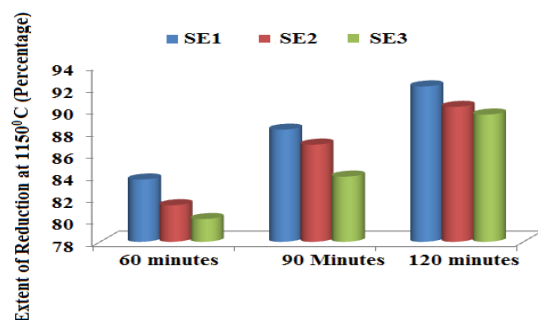


Fig. 10 Extent of reduction of nuggets of different size fraction at 1150 °C for reduced in 60, 90 and 120 minutes

From Tables V-VII and Fig. 9-11, it is observed that extent of reduction increases with increase reduction time and temperature. It also depends upon the size fraction of iron ore are used to from nuggets. Higher size fraction of fine particles gives higher extent of reduction as total reaction surface area increases.

TABLE VII
EXTENT OF REDUCTION AT 1100 °C FOR DIFFERENT TIMES AND SIZE FRACTIONS OF THE REDUCED SAMPLES

Samples	Extent of Reduction (60 Minutes)	Extent of Reduction (90 Minutes)	Extent of Reduction (120 Minutes)
SE1	81.29	85.67	89.09
SE2	79.73	83.28	87.82
SE3	78.01	81.87	85.87

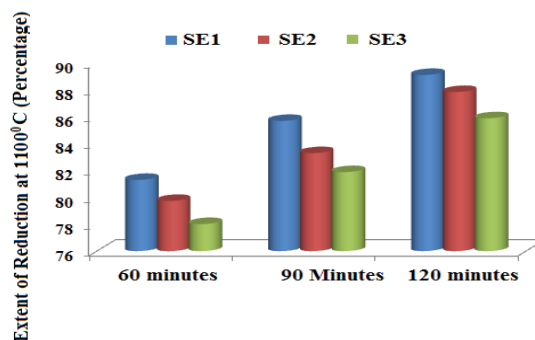


Fig. 11 Extent of reduction of nuggets of different size fraction at 1100 °C for reduced in 60, 90 and 120 minutes

F. Analysis of the Reduced Samples at 1200 °C for 60, 90 and 120 Minutes

Fig. 12 represents the XRD plots of the SE1 set of reduced nuggets heat-treated at 1200 °C for 60, 90 and 120 minutes. From Fig. 12, it is clearly seen that after 60 minutes no formation of metallic peak of Fe is present, only major peaks of FeO is present with Fe₃O₄. However, after 90 minutes, the presence of Fe peak in the XRD plot indicates that transformation has started and FeO is the major phase. After 120 minutes of reduction, the major fraction is iron with small fraction of FeO is present.

During the coal devolatilisation process, carbon shoot, H₂ and CO gases are generated which are mainly responsible for pre-reduction of agglomerated nuggets. From the XRD analysis, it can be conjectured that the hematite phase followed its normal path of stepwise reduction from firstly magnetite, subsequently wustite, and finally to metallic Fe.

G. SEM and EDX Analysis of the Reduced Samples

Figs. 13 (A)-(C) show the SEM micro photographs of the SE1 samples heated at 1200 °C for 120, 90 and 60 minutes respectively. The image shows agglomerated spongy honey comb structure. It is observed that increasing the reduction time has the highest influence on the morphology of iron.

EDX plot of the reduced samples are given in Fig. 14. It shows the presence of high value of Fe and small value of Al, Si, and O and the details are given in Table VIII which represents the weight percentage and atomic percentage of the elements in the sample after heat treatment.

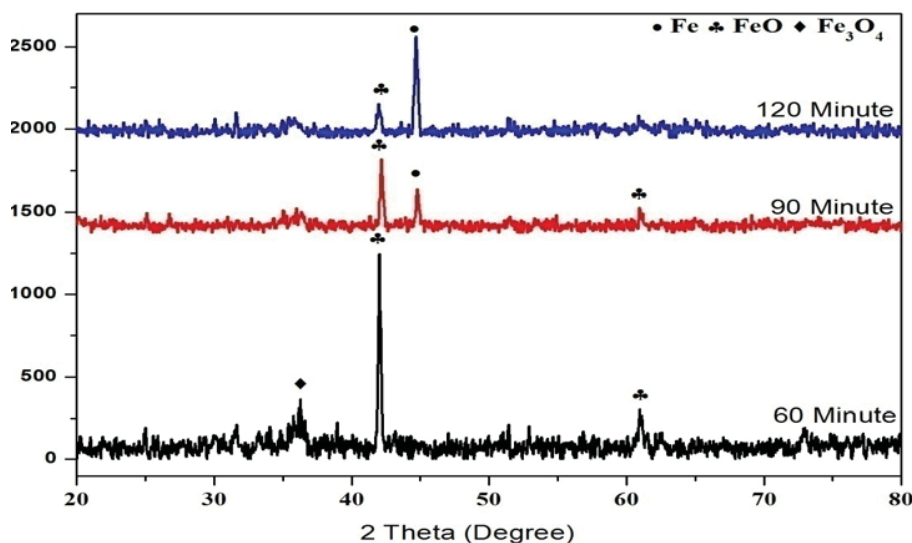


Fig. 12 XRD analysis of the SE1 reduced samples heat treated at 1200°C for 60 minutes, 90 minutes and 120 minutes

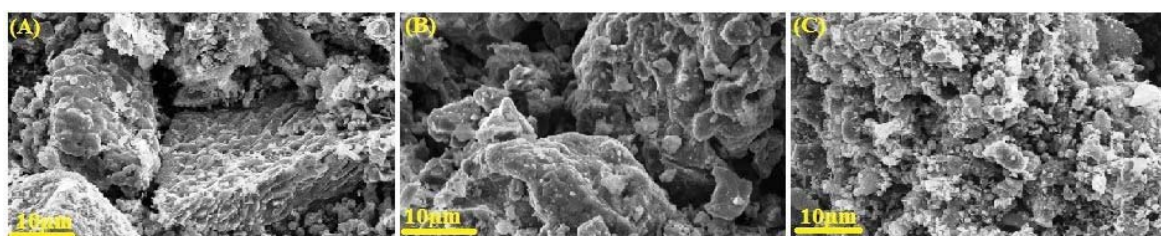


Fig. 13 (A) 120 Minute (B) 90 Minutes (C) 60 minute: SEM micro photographs of the SE1 samples heat treated at 1200 °C

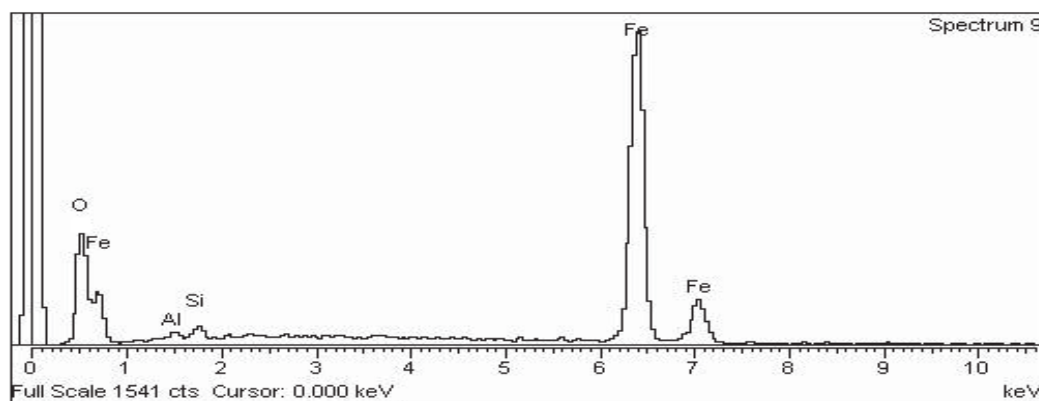


Fig. 14 EDX plot of the SE1 reduced samples heat-treated at 1200°C for 120 minutes

TABLE VIII
EDX RESULTS OF THE SE1 REDUCED SAMPLES HEAT-TREATED AT 1200 °C
FOR 120 MINUTES

Element	Weight%	Atomic%
O K	9.06	27.01
Al K	0.48	0.74
Si K	0.99	1.47
Fe K	89.48	70.78
Totals	100.00	100.00

H. Degree of Metallization of the Reduced Samples

Chemical analysis of the reduced samples is performed by the standard procedure of the Bureau of Indian Standards [25]

In brief, a 0.2 g sample has been taken in a dry conical flask and a 100 ml freshly prepared ferric chloride solution (10 g in 100 ml distilled water) is added in it. Then it is stirred for 30 min. A prepared acid mixture (25 ml hydrochloric acid and 25 ml orthophosphoric acid) are added to the conical flask and cooled. After cooling, the solution is titrated against 0.1 N potassium dichromate solution using BDS indicator. The burette reading is then recorded and calculated to get the total metallic iron content of the reduced sample. The results of degree of metallization are given in Table IX.

TABLE X
DEGREE OF METALLIZATION OF THE REDUCED SAMPLES AT DIFFERENT SIZE FRACTIONS, TEMPERATURES AND TIMES

Samples	Degree of Metallization (%) (90 Minutes)		Degree of Metallization (%) (120 Minutes)	
	1150°C	1200°C	1150°C	1200°C
	SE1	38.87	54.14	64.98
SE2	34.12	48.32	59.17	72.23
SE3	31.21	42.74	54.23	68.63

I. Sulphur Test of the Reduced Samples

Amount of sulphur and carbon are obtained during pyrolysis process for reduced samples are calculated by using Horiba ...EMIE 320 V, made in Japan. Sulphur contents for coal volatilized reduced samples are calculated with different size fractions, temperatures and times. Results are shown in given in Tables X, XI.

TABLE X
AMOUNT OF SULPHUR INTAKE OF THE REDUCED SAMPLES, HEAT TREATED AT DIFFERENT SIZE FRACTIONS, TEMPERATURES AND TIMES

Sam ples	Amount of sulphur (%) (90 Minutes)			Amount of sulphur (%) (90 Minutes)			Amount of sulphur (%) (120 Minutes)		
	1100	1150	1200	1100	1150	1200	1100	1150	1200
	°C	°C	°C	°C	°C	°C	°C	°C	°C
SE1	0.070	0.079	0.087	0.091	0.096	0.10	0.098	0.103	0.106
SE2	0.067	0.071	0.081	0.086	0.091	0.097	0.091	0.098	0.102
SE3	0.064	0.067	0.078	0.082	0.089	0.0.93	0.087	0.094	0.099

TABLE XI
AMOUNT OF SULPHUR INTAKE OF THE REDUCED SAMPLES, HEAT TREATED AT DIFFERENT SIZE FRACTIONS, TEMPERATURES AND TIMES

Samp les	Amount of Carbon (%) (60 Minutes)			Amount of Carbon (%) (90 Minutes)			Amount of Carbon (%) (120 Minutes)		
	1100	1150	1200	1100	1150	1200	1100	1150	1200
	°C	°C	°C	°C	°C	°C	°C	°C	°C
SE1	0.373	0.423	0.501	0.424	0.524	0.674	0.897	0.981	1.12
SE2	0.341	0.396	0.412	0.387	0.498	0.647	0.841	0.927	1.08
SE3	0.312	0.347	0.404	0.394	0.487	0.617	0.814	0.903	1.05

In direct reduction process, the reduced nugget of iron ore fines & reductant (stoichiometric amount of 1:1 ratio mixture of boiler grade coal and coke dust) contains 2.08% sulphur at around which is relatively very high in comparison to the pyrolysis process. The results of sulphur test analysis of the devolatilisation reduced sample, it is proved that the SYNGAS reduction process gives fruitful results using boiler grade coal.

J. Mechanical Properties of the Reduced Samples

The reduced nugget resembles a very strong and stable agglomerated metallic aggregate. Shatter and abrasion tests demonstrate negligible loss of material due to impact, which will make it suitable for charging in a blast furnace which in course will minimize the coke rate. Quality of steel is improved. Productivity of the steel industries will be increased.

IV. CONCLUSION

❖ Iron ore nuggets are reduced successfully at different temperatures using the pyrolysis products of lean grade coal in the laboratory scale. The pyrolysis of lean grade coal is carried out at around 600–650 °C.

- ❖ Amorphous carbon soot is generated in the course of devolatilization of coal also helped in better reduction.
- ❖ Extent of reduction depends on reduction times, temperatures and size fractions of the iron ore. It is observed that metallization increases with increase in reduction temperature, time and fraction of finer size iron ore particle in nugget. Maximum extent of reduction (94.65%) is achieved when reduction carried out at 1200 °C for 120 minutes.
- ❖ The metallic iron content of the reduced hematite sample is approximately 78.87%. The chemical analysis of the reduced sample shows that the concerned reduction process provided a good degree of reduction.
- ❖ Hence, by performing the sulphur test analysis of the reduced sample, it is proven that the SYNGAS reduction process gives fruitful results using lean grade coal. It is found that sulphur and carbon percentage increase with the reduction time and temperature where as it decreases with higher particle size fraction.
- ❖ In the actual process, the pre-reduced nuggets can be charged directly in blast furnace with higher mechanical strength which in course will minimize the coke rate and in turn will reduce sulphur content of iron.

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APPENDIX 1

- A nugget contains 4% molasses + 4% bentonite + 2% moisture + rest hematite iron ore oxide
- Experimental weight before reduction = 0.43 g molasses + 0.43 g bentonite + 0.21 g moisture + 9.51 g iron ore
- Total weight of the Nuggets = 10.58
- Loss on heating = 95% 4 g molasses + 18% 4 g bentonite + oxygen contained in hematite + 100% 2 g moisture
- 100 g ore contains 91.5 g hematite
- 9.51 g ore contains 8.7 g hematite
- 91.5 g hematite contains 64 g iron (From WDXRF and Chemical Analysis)
- 8.70 g hematite presents in 6.08 g iron content
- Oxygen content = 8.70 - 6.08 = 2.62 g
- Weight loss from the nuggets in case of 100% reduction = (0.40 + 0.07 + 0.31 + 2.62) = 3.30 g
- Remaining weight after reduction = (10.58 - 3.30) = 7.28 g
- Experimental weight after reduction = 7.365 g
- Experimental weight loss of the nuggets after reduction = (10.58 - 7.42) = 3.18 g

- Fixed weight loss of the nuggets after reduction = $(0.40 + 0.07 + 0.21) = 0.68\text{gm}$
- Total loss of oxygen after reduction = $3.16 - 0.68 = 2.48\text{ g}$
- Extent of reduction = (weight loss of oxygen after reduction / weight of oxygen before reduction)
- Percentage of extent of reduction = $(2.48/2.62)*100 = 94.65\%$

REFERENCES

- [1] Haque, E. Md.: Indian coal production and ways to increase coal supplies, *International Journal of Scientific and Research Publications*, 3 (2013), no. 2, pp 1–3.
- [2] Srinivasan N. S.: Reduction of iron oxides by carbon in a circulating fluidized bed reactor, *Powder Technology*, 124 (2002), pp 28–39.
- [3] Kumar, M.; Patel, S. K.: Characteristics of Indian non-coking coal and iron ore reduction by their chars for directly reduced iron production, *Mineral Processing and Extractive Metallurgy Review*, 29 (2008), no. 3, pp 258–273.
- [4] S C Koria, Dynamic variation of lance distance impinging jet steel making processes *Steel research* vol.59 (1988), No.6 p.257-262.
- [5] J. Kijac, M. Borgon, Desulphurization of Steel and Pig Iron, *Metalurgija* 47 (2008) 4, 347-350.
- [6] Chen-Lin Chou, Sulfur in coals: A review of geochemistry and origins, *International Journal of Coal Geology* 100 (2012) pp 1–13.
- [7] A. Ghosh and A. Chatterjee: *Ironmaking and Steelmaking: Theory and Practice*, PHI Learning Private limited, 2008, 472 p.
- [8] L. Socha, J. Bažan, K. Gryc, P. Machovčák, J. Morávka, P. Stymal, Evaluation of Fluxing Agents Effect on Desulphurization in Secondary Metallurgy Under Plant Conditions, *Metalurgija* 52 (2013) 4, 485-488.
- [9] V. Posch, P. Miceli, W. Pluschkell and H. Lachmund, Desulphurization of liquid steel with refining top slags, *Technical steel research*, Publications of the European Communities, 2002, ISBN 9289442794 pp 13-20.
- [10] M. Andersson, M. Hallberg, L. Jonsson, and P. Jonsson, Slag–metal reactions during ladle treatment with focus on desulphurization, *Ironmaking & Steelmaking: Processes, Products and Applications*, Volume 29, Issue 3, 2002 pp 224-232.
- [11] Hiroyasu Shigemori, Member, IAENG, Quality and Operation Management System for Steel Products through Multivariate Statistical Process Control Proceedings of the World Congress on Engineering 2013 Vol I, WCE 2013, July 3 - 5, 2013, London, U.K. ISBN: 978-988-19251-0-7.
- [12] Ranjan Sen, Siddharth Mukherjee and Rajib Dey, 'Effect of grading of Chromite ores on the quality of Briquettes' *JISI*, volume 50, pp.200-206, (2010).
- [13] Cheeley, R.; Leu, M.: Coal gasification for DRI production—An Indian solution, *Steel times International*, April 2010.
- [14] Pawlik, C.; Schuster, S.; Eder, N.; Winter, F.; Mali, H.; Fischer, H.; Schenk, J.: Reduction of Iron ore fines with CO-rich gases under pressurized fluid bed conditions, *ISIJ International*, 47 (2007), no. 2, pp 217–225.
- [15] Bell, D. A.; Towler, B. F.; Fan, M.: *Coal gasification and its applications*, Elsevier, 2011.
- [16] Sato, M.; Hideaki, S.; Kanji, T.: Development of the process for producing pre-reduced agglomerates, *JEF Technical Report*, 13 (2009), pp 7–13.
- [17] Geological survey of India Production of iron ore lumps fine concentrates in India (source MNI January 2015).
- [18] M. Kumar, S. Patel, *Mineral Processing & Extractive Metallurgy*, "Assessment of Reduction Behaviour of Hematite Iron ore Pellets in Coal Fines for Application in Sponge Ironmaking" Rev-30, Pp 240-259, 2009.
- [19] Tetsuo Horie and Makoto Shimizu, Development of Coal Gasification-iron ore Reduction Process, by Fluidized Bed, *ISIJ International*, 73 (1987), no. 7, pp 46–53.
- [20] James Burgess, South Africa to Use Large-Scale Underground Coal Gasification, 28 April 2013.
- [21] Y.Q. Sun, Z.T. Zhang, L.L. Liu, X.D. Wang, Integrated carbon dioxide/sludge gasification using waste heat from hot slags: syngas production and sulfur dioxide fixation, *Bioresour. Technol.* 181 (2015) 174–182.
- [22] J. Nakano, L.L. Liu, X.D. Wang, Z.T. Zhang Achieving waste heat to energy through sewage sludge gasification using hot slags: syngas production. *Scientific Reports* 2015.
- [23] Y.Q. Sun, Z.T. Zhang, S. Seetharaman, L.L. Liu, X.D. Wang, Characteristics of low temperature biomass gasification and syngas release behavior using hot slag, *RSC Adv.* 4 (2014) 62105–62114.
- [24] Chanchal Biswas, Anrin Bhattacharyya, Gopes Chandra Das, Mahua Ghosh, Choudhuri & Rajib Dey, A Novel Devolatilization Technique of Pre-reduction of Iron Ore Using Lean Grade Coal, *Berg- und Hüttenmännische Monatshefte (BHM)* DOI 10.1007/s00501-015-0401-2 (2016) Vol. 161 (3): 95–101
- [25] Bureau of Indian Standards BIS 10812:1992, total metallic iron calculation, 1997 pp 1-6