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Experimental Studies on Reduction of Coarse Blue Dust for Production of Iron Powder by Hydrogen Reduction

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***Abstract*:- Blue dust occurring in the Iron ore mines of NMDC were found to be underutilized due to lack of technical knowhow to produce value added products. NMDC has developed various methods to use this blue dust for hi-tech applications. Blue dust has been used to produce hard and soft ferrites. This blue dust is also used to produce Ultra pure ferric oxide and high grade ferric oxide. Premium grade sponge iron powder was produced using -212+45 micron size blue dust concentrate using hydrogen gas as a reductant. Whereas the coarse blue dust concentrate (<212 microns) was lying as waste even though it is very rich in iron content. Experiments were conducted using this coarse blue dust concentrate in the size range of -500+212 microns. Preliminary studies were conducted at 850ºC and found to be satisfactory. Based on the satisfactory results a series of tests were conducted. The main variables selected are Bed height (mm), Reduction temperature (degree Celsius) and Reduction time (hours). Bed height of 10mm and 20mm were not found to be encouraging and hence studies were conducted with an optimum bed height of 15mm in the stainless steel boats. The experiments were conducted at 4 hours , 5 hours and 6 hours reduction time where as the reduction temperatures were selected as 600ºC, 700ºC, 850ºC and 900ºC. The products obtained were subjected for chemical analysis and and subsequently physical properties were also studied. The study reveals that coarse blue dust concentrate can also yield a good quality Iron powder.**

Key words: Blue dust, Hydrogen, Reduction, Iron powder, Characterisation and Bailadila

INTRODUCTION

Blue dust is a high grade soft hematite ore fines containing more than 90% Fe_2O_3. This was termed as blue dust due to its characteristic luster and fineness. These iron ore fines are dumped at site due to transportation problem. The particle size of the blue dust lies in the micron range, it cannot be used directly in the blast furnace for iron making [1]. Huge occurances of powdery iron ore have been observed during the study of the Precambrian sedimentary iron ore deposits of Noamundi in Singbhum district of Bihar and Goa [2]. Blue dust is also found to exist in Jilling – Langlata deposits of Eastern India craton [3]. Beneficiation studies were carried out with High silica blue dust from Deposit 5, Bacheli complex, Bailadila Chattisgarh [4]. Various studies were carried out to develop value added products from Bailadila blue dust. Iron oxides were subjected for reduction using hydrogen gas [5], [6]. Blue dust was also used in Iron ore sintering as a sweetener [7].

Huge quantities of Blue dust of various grades is available in the Iron Ore Mines of NMDC Limited. This blue dust as such cannot be used as a feed material for steel production due to its fine nature. This blue dust has to be agglomerated (Sintering and Pelletisation) depending on the size, otherwise this blue dust lies as waste in the mine. Based on the grain size and chemical purity the blue dust resources have been divided in to three types, fine grained (<1.5% silica), Medium grained (1.5% to 3.0% silica) and coarse grained (3.0% to 5.5% silica). Typical composition of the blue dust is presented in Table 1. NMDC is committed for mineral conservation and development of technology for production of value added products from its mine waste. NMDC is engaged in development of value added products from the blue dust. The Silica content of blue dust varies from 1.00% to 6.00% in the mines and size less than 10mm. NMDC had produced various grades of Ferric oxide and Ultra Pure Ferric Oxide from the available blue dust. In the process of value addition to its blue dust available in its mines, recently developed a new product Carbon Free Sponge Iron Powder (CFSIP). This Carbon Free Sponge Iron Powder has many wide Hi -Tech applications like Soft magnetic components which is used in electromagnetic relays, Electromagnetic applications, Powder metallurgical applications, Sintered products, Chemical and Metallurgical applications, Iron Fortification, Breaking systems of automobiles, Surface coatings and welding etc.

Blue dust was processed using plasma to investigate the recovery rate and degree of metallization [8]. Some researchers studied the effect of milling on the reduction behavior of blue dust [9]. Conventionally the iron ore reduction was carriedout using carbon in solid state (direct reduction) and carbon monoxide. Various attempts were done to use hydrogen as reductant to reduce Iron oxide [10], [11] and [12].

A super concentrate has been prepared to reduce the impurity content from the blue dust collected from NMDC Mines [13]. Studies have been proved that a high quality very low impurity iron powder can be produced from super concentrate produced from blue dust [14], [15] and [16]. During this process it was observed that size fraction of -212+45 micron was used, Coarse (+212

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micron) and ultra fine (-45 micron) were not used even though it is very high quality and low impurities [14, 15 and 16]. This paper focuses on the reduction of coarse super concentrate (-500+212 microns) and to produce carbon free coarse iron powder by using hydrogen as reductant.

Table 1. Typical composition of blue dust from Bailadila Sector

Constituent	Assay percent		
	Fine Grained	Medium grained	Coarse grained
Fe	69.00	67.80	65.34
SiO_2	1.00	2.16	5.40
Al_2O_3	0.20	0.60	0.60
LOI	0.18	0.38	0.29
P	0.010	0.015	0.010
S	Traces	Traces	Traces
-150 micron	68.90	54.90	47.90

EXPERIMENTAL SET UP

The furnace used for the tests, consists of pre-heating zone, reduction zone and cooling zone. It has a sample pushing arrangement and the same can be pushed up to a length of 500mm max. The sample is taken in stainless steel (SS) boat and pushed inside the furnace by placing them on a silicon carbide refractory brick. Dummy bricks are used to push and place the sample at desired zones. Initially samples are placed in pre-heating zone for at least 30 min so as to release moisture content if any; Thereafter, samples are sent to reduction zone where temperature can be raised up to 1100°C, depending on the requirement. The parameters such as temperature, quantity of sample, retention time etc; are set based on the type of material reduced.

Furnace has an arrangement of supplying inert gas (N_2) and reducing gas (H_2) through separate pipe lines with flow meters and control valves. Furnace is heated in the presence of Nitrogen gas for at least an hour after achieving a temperature of about 550-600°C before injection of Hydrogen gas at desired reduction temperature. However, Nitrogen gas shall be continued at lower flow rate along with Hydrogen gas.

After completion of the reduction of samples at set parameters, stainless steel boats are pushed in to cooling zone where the samples get cooled to about 60-80°C to prevent re-oxidation. The photograph of air tight furnace used for reduction of coarse blue dust using hydrogen is shown at figure.1. The sample movement in the furnace during and after reduction is shown in figure 2.

The samples are then removed from the furnace and subjected for various characterisation studies like loss in weight, physical, chemical properties, Microscopic examination (SEM) etc..

Fig. 1. Furnace used for reduction of coarse blue dust.

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The sample

The blue dust available in the mines cannot be used as such as feed material for carbon free sponge iron production and it requires very high grade feed material. The feed material should not contain more than 0.30 % Silica (SiO_2) and Fe (T) should be 98.50. Super grade blue dust concentrate has been produced [12]. The concentrate was screened and a size range of -500+212 micron has been separated. This size product was subjected for experimentation to produce carbon free sponge iron powder. The chemistry of sponge iron powder is given at Table 2. Scanning Electron Microscope (SEM) images of coarse blue dust are shown as Fig. 4 (a and b).

Table 2. Chemistry of super concentrate (-500+212 micron)

Constituent	Assay percent
Fe (T)	69.80
Fe_2O_3	98.90
FeO	0.10
SiO_2	0.30
Al_2O_3	0.10
P	Traces
S	Traces
LOI	1.20

EXPERIMENTAL RESULTS

Experiments were conducted in the airtight furnace shown in Fig. 1. All the experiments were conducted with a sample bed height of 15mm. Around 140 grams of sample was taken in a stainless steel boat and experiments were carried out at 600ºC, 700ºC, 850ºC and 900ºC for a time of 4 hours, 5 hours and 6 hours. Experiments were conducted as per the process described at experimental set up. After the completion of experiment the samples were taken out carefully and final weights were recorded. Degree of reduction was calculated as per standards. Apparent density was determined using the standard procedure. All the samples were cooled and characterized for Fe (T), Fe (Metallic), FeO and SiO_2. The experimental results were shown in table 3.

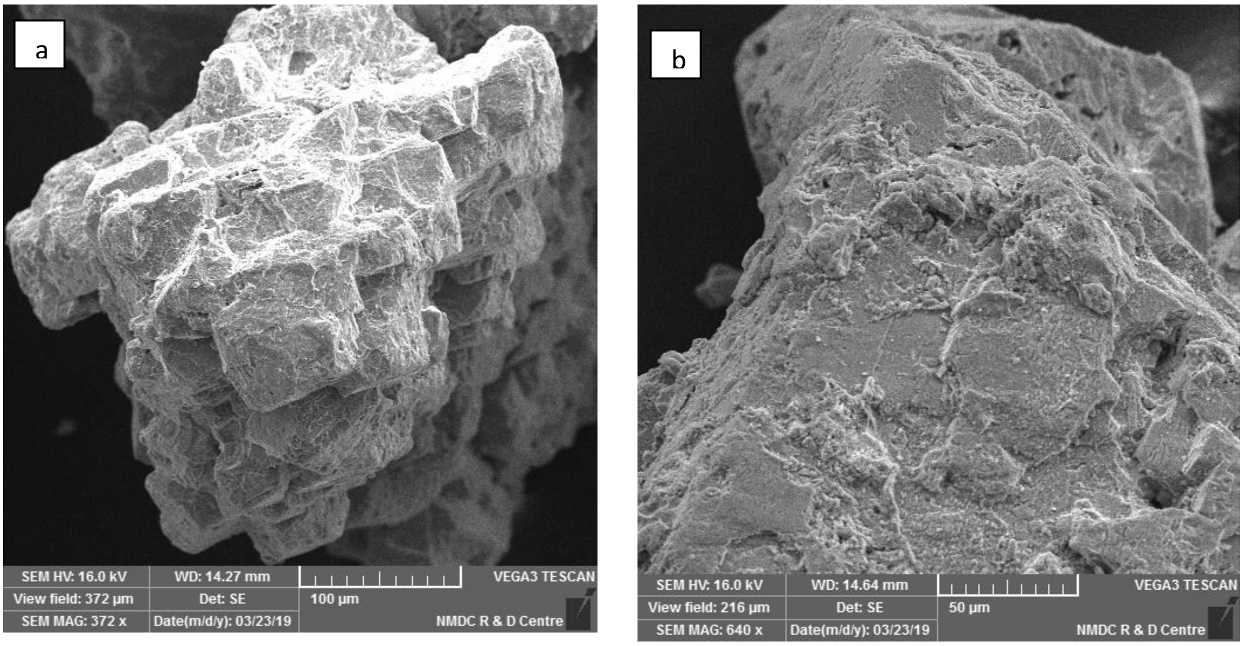

Fig. 3. SEM image of coarse blue dust (a and b) (-500+212 micron)

Table 3. Experimental results

Sl. No	Bed height (mm)	Time (minutes)	Temp (ºC)	%Degree of Reduction	Apparent Density	Fe(T)	Fe(M)	FeO	SiO_2
1	15	300	600	46.53	2.36	76.80	22.80	38.07	0.26
2	15	300	700	58.42	1.88	83.20	27.40	68.42	0.36
3	15	360	700	68.87	1.88	86.40	52.80	42.70	0.20
4	15	240	850	79.10	1.96	89.10	60.70	36.14	0.20
5	15	300	850	83.16	1.94	91.40	70.80	24.69	0.19
6	15	360	850	90.54	1.94	94.40	80.20	23.60	0.27
7	15	240	900	88.86	1.90	93.20	74.60	22.63	0.20
8	15	300	900	94.55	1.96	95.60	85.40	13.12	0.16
9	15	360	900	98.25	1.96	98.25	91.20	3.86	0.29

DISCUSSION

Effect of temperature on degree of reduction:

Effect of temperature on degree of reduction was studied between 700ºC, 800ºC and 900ºC at 5 hours and 6 hours reduction time. A graph was plotted as shown in the fig. 4. In both the cases (5 hours and 6 hours) as the temperature increased the degree of reduction increased. Both the lines are parallel to each other up to 850ºC and then the degree of reduction increased rate increased as the distance between the lines decreased. In this, the highest degree of reduction (98.25%) was attained at 900ºC after 6 hours of reduction, whereas lowest degree of reduction (58.42 %) obtained at 700ºC after 5 hours of reduction.

Effect of reduction time on degree of reduction:

Degree of reduction was studied at three time intervals (4, 5 and 6 hours) and at two temperatures (850ºC and 900ºC). The effect of reduction time on degree of reduction is shown in Fig. 5. This shows that the highest degree of reduction will be obtained at 6 hours reduction time and 900ºC temperature for this particular coarse blue dust sample.

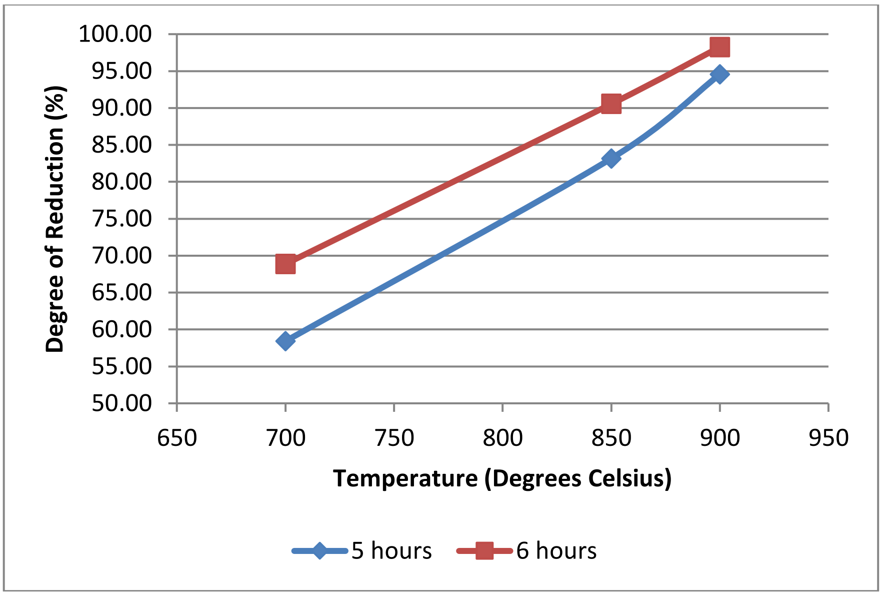

Fig. 4. Effect of temperature on degree of reduction at 5 hours 6 hours reduction time

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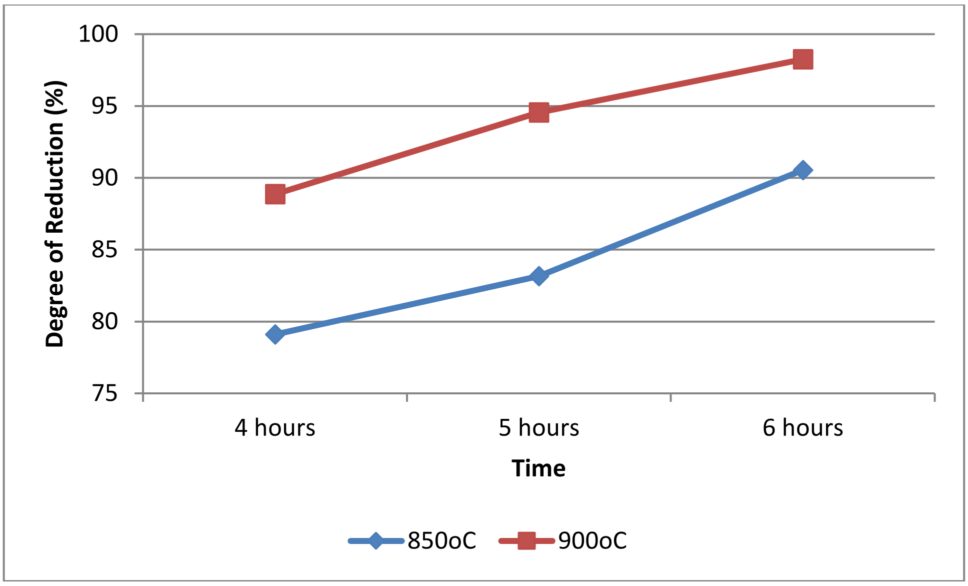

Fig. 5. Effect of reduction time on degree of reduction at 850ºC and 900ºC temperature

Variation of Fe (Total), Fe (Metallic) and FeO:

As the reduction experiments were completed, the sample was subjected for chemical analysis. Fe(Total), Fe(Metallic) and FeO were found using Volumetic method, whereas SiO_2 was determined using gravimetic method and Al_2O_3 by Induction coupled Plasma method. The results were shown in Fig. 6. It was observed that Fe(T) and Fe(Metallic) were increased as the degree of reduction increased whereas FeO increased from 38.07 to 68.42 (around 60% reduction) and then gradually decreased to 3.86 at 98.25% reduction. The Fe (T), Fe (Metallic) and FeO varied between 76.80 to 97.20%, 22.00 to 91.20% and 3.86 to 68.42% respectively. The degree of reduction increased from 44.90 to 98.25%. The apparent density of the sponge iron produced varied between 1.88 to 2.36. From the table 3, it can be observed that the SiO_2 varied between 0.10 to 0.36%.

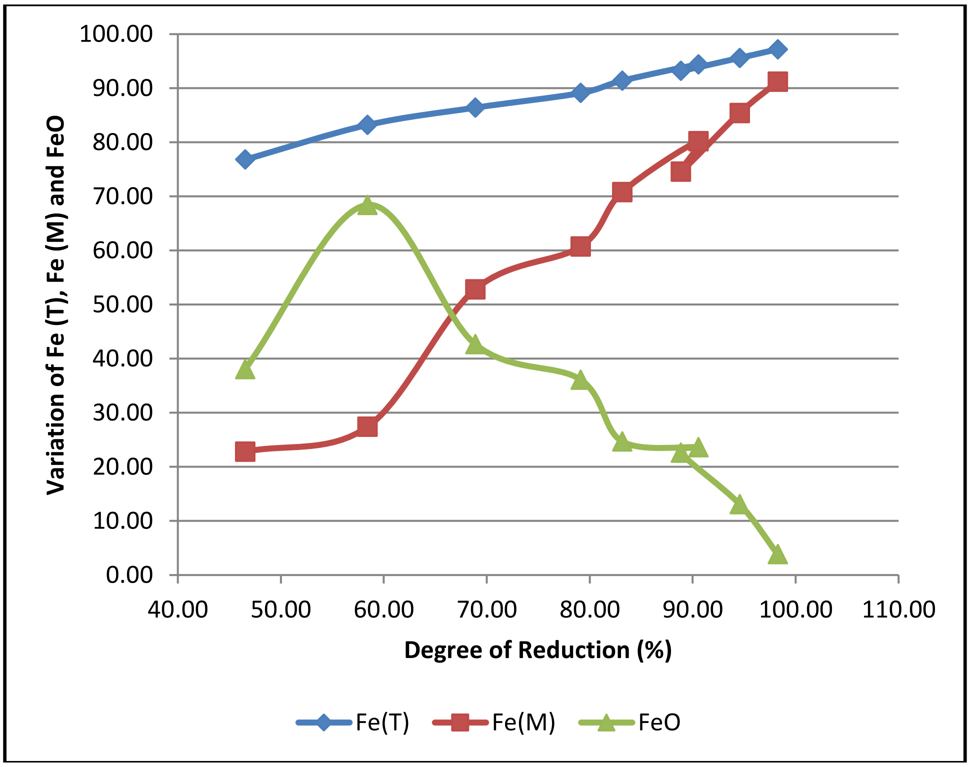

Fig. 6. Variation of Fe (T), Fe (M) and FeO with degree of reduction

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Characterisation of Final Product

The chemical analysis of the final product was determined using standard methods (Volumetic, Gravimetric and Induction coupled Plasma). Iron content is greater than 99.20. The chemistry of the final product is shown in table 5. SEM images of coarse iron powder were taken and presented as Figure 7.

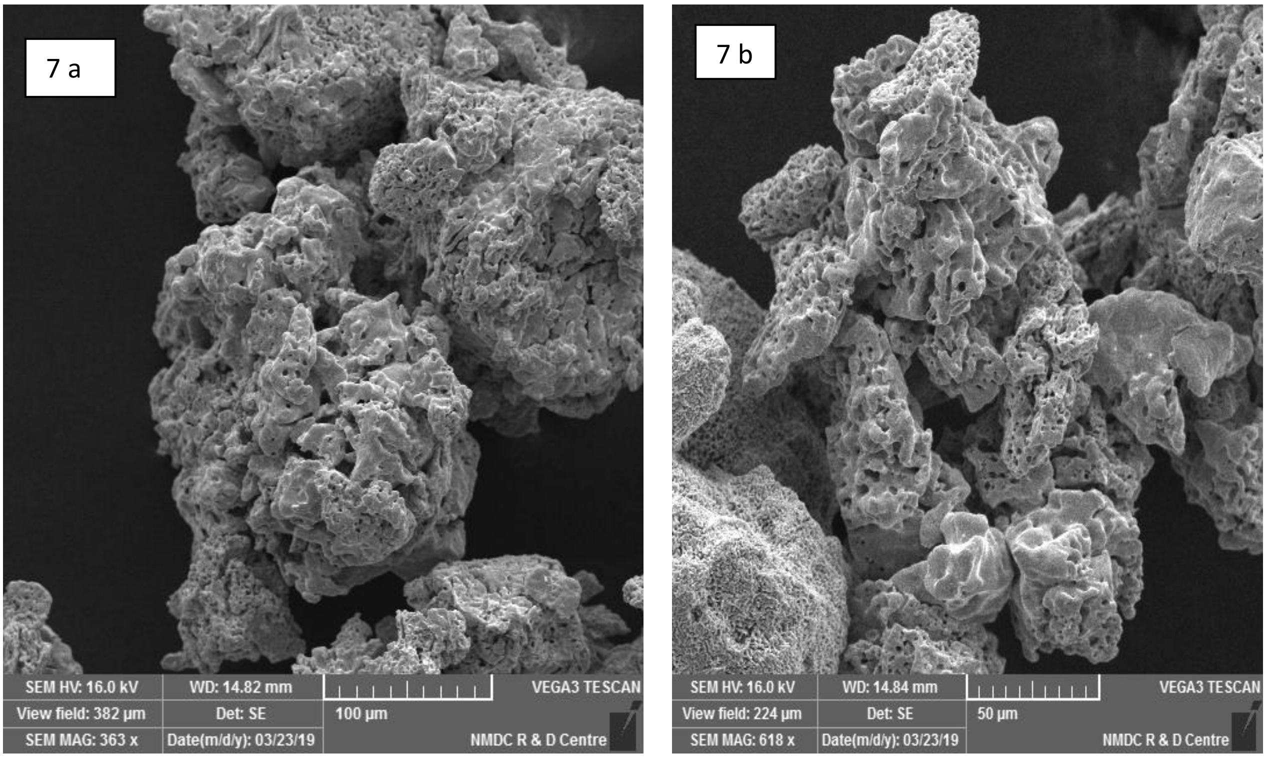

Fig. 7. SEM image of reduced iron powder (-500+212 micron)

Table 5. Chemistry of Carbon free coarse sponge iron powder produced.

Chemistry	Assay percent
Fe	>99.20
SiO_2	0.24
Al_2O_3	0.05
Carbon	<0.0075%
Sulphur	Traces
Phosporus	Traces
Oxygen	0.20%
Acid insolubles	0.40%

The physical properties of the reduced iron powder were found using standard methods. The average particle size and specific surface area found to be 300.7 microns and 722 m2/kg respectively. The apparent density and tap density were found to be 1.70 to 2.40 and 2.70 respectively. The flow rate of the powder is found to be 20 to 35 sec/50grams.

CONCLUSIONS

Super concentrate of coarse blue dust (-500+212 micron) assaying around 69.80% Fe with less than 0.50% impurities (SiO_2+Al_2O_3) was subjected to reduction using hydrogen gas. The bed height of the sample is taken as 15mm as it yielded good results. Experiments were conducted at 600°C, 700°C, 850°C and 900°C temperature and 4 hours, 5 hours and 6 hours reduction time. At low temperature (600°C) and lower reduction time (4 hours) the degree of reduction is very less (44.90%), whereas at higher temperature (900°C) and higher reduction time (6 hours) the degree of reduction is very high (98.25%). The apparent density of the coarse iron powder produced varied between 1.88 to 2.36. The total iron content (Fe_{Total}) is found to be lowest (76.80%) at a temperature of 600°C and at a reduction time of 5 hours. The highest total iron content (98.25%) was observed when the sample was subjected to 900°C and reduction time of 6 hours. The lowest metallic iron (22.80%) was obtained at a temperature of 600°C and at a reduction time of 5 hours. As the degree of reduction increased from 44.90% to 98.25%, the FeO content continuously decreased.

The coarse iron powder produced using hydrogen as reductant analysed greater than 99.20% Fe. The average particle size and specific surface area found to be 300.7 microns and 722 m^2/kg respectively. The apparent density and tap density were found to be 1.70 to 2.40 and 2.70 respectively. The flow rate of the powder is found to be 20 to 35 sec/50grams.

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