A Novel Technique for Making Cold Briquettes for Charging in Blast Furnace

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Abstract. Different metallurgical wastes are generated during pyro processing of iron ore, which is used for making sponge iron or hot metal and for producing steel. Apart from these wastes, coke fines are generated during the coke making, and iron ore fines are generated during mining of iron ore. Although iron ore fines are used for making pellet after beneficiation still, it generates a huge quantity of iron ore waste during beneficiation with comparatively lower iron content. In the present study, briquettes are made by a stiff extrusion process from metallurgical waste like iron ore fines and coke fines with the addition of Portland cement as a binder and clay as a rheology modifier. Physical properties of the briquettes are evaluated, and reducibility of the briquettes is studied in comparison to lumpy iron ore. Phase analysis and microstructural analysis of the briquettes and lumpy iron ore are carried out after firing at different temperatures in the simulated blast furnace condition. Physical and mineralogical properties are correlated with the reducibility of the briquettes and lumpy iron ore. Briquettes made by a stiff extrusion process show a better mechanical strength fired at a different temperature to take the load of burden and better reducibility than lumpy iron ore. The briquettes after self-curing are charged to a 23 mt3 blast furnace which shows encouraging results.

1. Introduction

Large quantities of useful iron bearing wastes are generated at different stages of iron and steel making processes. Apart from these wastes large quantity of iron ore fines are generated during mining, crushing and grinding. Most of them are in fine fraction and difficult to use even for making sinter use as a feed material for the blast furnace. Now a day's pelletization is getting popular which uses iron ore fine and becomes a feed material after induration. But different iron-bearing waste generated could not be used for pallet making due to different metallurgical reasons.

Another method of agglomeration is briquetting, which consists of three basic technologies i.e. vibro-pressing, roller pressing and the stiff extrusion. The stiff-extrusion technique which was mastered by J. C. Steel & Sons (USA), had its 1st industrial application in the year 1993 [1]. In this cold briquetting technology various steel plant by-products can be easily agglomerated using very few amount of binder material and also cost effective than other agglomerating processes, due to their less processing steps.In this advanced technology homogenizing mixture of raw materials with the moisture content of 12-16% passes through the vacuum chamber and then pushed through the die holes by pressure 3-3.5 Mpa [2]. The industrial stiff-extrusion process lines shown in figure 1.

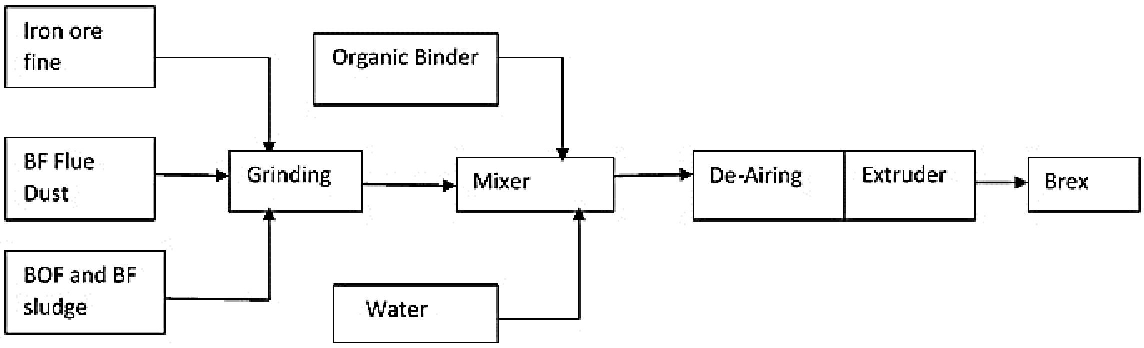

Figure 1. Schematic diagram of the extruded briquette preparation process.

During the passing of homogenizing mixture of raw materials through the vacuum chamber of the extruder, approximately 90% of compressed air is removed [3]. This causes the high mechanical strength of extruded briquette, even after the outlet of the extruder. A Steele 25 extruder used in the present work for briquetting is a combination of Pug sealer and extruder as shown in the figure 2. The Pug Sealer sits on top of the extruder having high pressure screw mixer that mixes the materials intensively. The vacuum inside the vacuum chamber is created by a vacuum pump. The mixture passes through the vacuum chamber and drops down at the blades of the extruder's auger. The vacuum is maintained throughout the working volume of the extruder. Then the material mixture is pressed through the extrusion dies under high pressure and vacuum to generate cold briquettes.

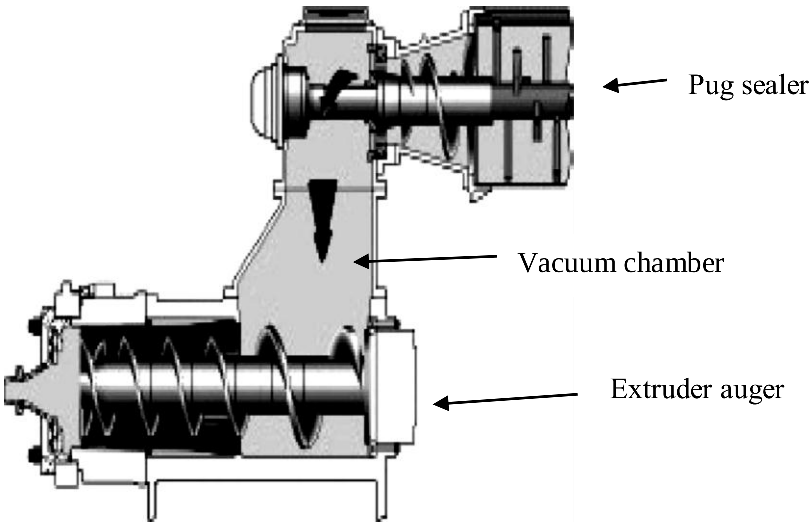

Figure 2. Sectional view of extruder vacuum chamber.

2. Experimental Methodology

2.1 Materials

The materials used in this work are iron ore fines, BF dust, LD sludge, sludge and mill scale. The waste materials were collected from mining area and different steel plants. These materials were evaluated for their chemical properties. Their chemical analyses are given in table 1. Phase analysis is done by X-ray diffractometer at DISIR, Rajgangpur. Phase analysis data are given in table 2. Chemical analysis of Portland cement and bentonite are given in table 3.

Table 1. Chemical analysis (wt. %) of Metallurgical waste.

Constituents	Iron ore fines (wt. %)	BF dust (wt. %)	LD sludge (wt. %)
Fe_2O_3/FeO	78.5	51.5	87.5
SiO_2	5.6	6.3	0.6
CaO	-	4.9	9.5
MgO	-	0.2	1.2
Al_2O_3	5.4	5.1	0.3
TiO_2	0.8	-	-
C	-	30.5	-
Fe_T	53.5	35.6	64

Table 2. Phase analysis by XRD.

Constituents	Major phases	Minor phases
Iron ore fine	Hematite	Goethite, Gibbsite, Kaolin, Pyroxene
BF dust	Magnetite, Hematite, Graphite	Wustite, quartz, Dicalcium aluminate
LD sludge	Magnetite, Wustite	Wollastonite, calcite

Table 3. Chemical analysis (wt. %) of Portland cement and Bentonite.

Constituents	Portland cement	Bentonite
CaO	63.2	0.8
SiO2	20.5	58.4
Al_2O_3	4.5	12.6
Fe_2O_3	4.2	10.6
MgO	2.1	0.3
K_2O+Na_2O	0.6	4.4
SO_3	2.6	-
LOI	1.5	9.4

The raw materials were taken according to their weight percentage, as given in table 4 and mixed properly. The mixture was passed through the homogenizing mixer and the vacuum chamber. Where, vacuum maintained was $0.5x10^{-3}$Bar. The briquette formed was extruded from the die at a pressure of 100 kg/cm^2.After that, the brex were cured in an open air.

Table 4. Mixing percentage of raw materials

Briquette Sample No	LD sludge (Wt. %)	BF dust (Wt. %)	Iron ore fine (Wt. %)	Binder (Wt. %)	Plasticizer (Wt. %)
1	47.28	28.3	18.9	4.7	0.9

The chemical compositions of the raw briquette and lumpy iron ore are shown in table 5. XRD analysis of raw iron ore lump shown in Fig 3 and for raw briquette shown in figure 4.

Table 5. Chemical composition of Briquette and iron ore.

Constituents	Iron ore (Wt. %)	Briquette (Wt. %)
CaO	0.2	8.4
SiO_2	2.21	4.1
Al_2O_3	3.11	2.35
Fe_2O_3	88.53	68.53
MgO	-	1.4
K_2O+Na_2O	0.2	0.2
TiO_2	0.22	0.23
LOI	5.82	15.02

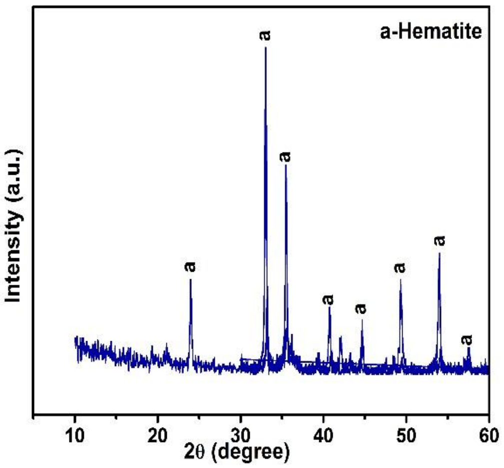

Figure 3. XRD analysis of raw iron ore lump.

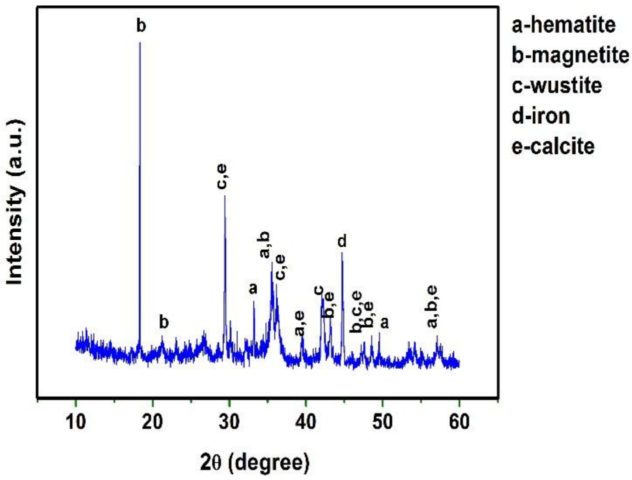

Figure 4. XRD analysis of raw briquette.

2.2 Mechanical strength of briquette and lumpy iron ore

The mechanical properties of briquette and iron ore are given in table 6. The cold compressive strength of briquette after natural curing at different days are shown in figure 5. For getting the mechanical properties which are shown in table 6, the briquette and iron ore lumps were rotated in tumbler drum at

25RPM for 200 revolutions. Then it passed through 6.3 mm sieve and a 0.6 mm sieve. The retained weight percentage of the samples on 6.3 mm size sieve give the tumbler index. Abrasion index is calculated by taking the weight percentage of the fines passed through 0.6 mm size sieve.

Table 6. Mechanical properties.

Sample	Tumbler Index	Abrasion index
Briquette	96	1.8
Iron ore	82	2.9

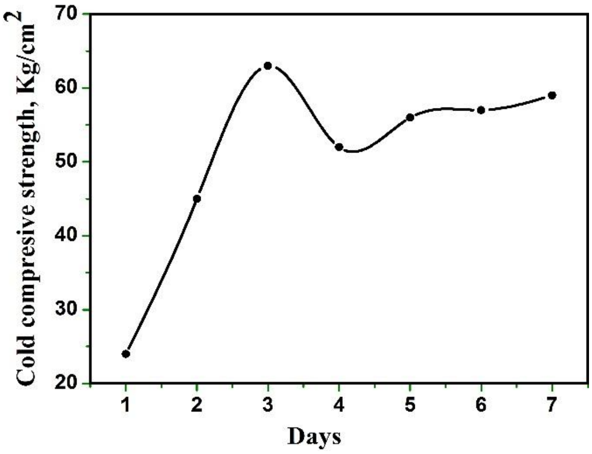

Figure 5. Cold compression strength (CCS) after natural curing**.**

The cold compressive strength of cured briquettes was measured by Tonipact3000. The strength shows a max on completion of third say followed by a decrease on the fourth day and then showed an increasing trend up to the studied range. Such behavior is due to the creation of the coagulation structure in the cement-bentonite-water system, leading to modifications in the properties of the binder. Reduced strength, observed after the third day, can be explained by the destruction of structures.

2.3 Reduction test of Briquette

A hollow alumina tube having a length 1000 mm, outside diameter 85 mm and inside diameter 75mm was taken to carry out the reduction test. The weights of the briquettes and iron ore were taken before and after the test. Briquettes and iron ore were kept in a graphite crucible and the crucible was then placed inside the alumina tube covered by coke breeze having size 1-5mm throughout the height engulfing the graphite crucible completely. The thickness of the coke layer was 25mm. Then it is blocked with insulating blocks. Water cooled metallic clamps were fitted on both the sides of alumina tube. The vacuum pump connected in one of the clamps along with air supply connected with a flow meter. Before heating, the inside chamber of the alumina tube was evacuated up to 0.5 mbar. The furnace was heated at a rate of 10°C/min. When the furnace attained a temperature of 800°C, air supply was started at a rate of 10 liters /min till the completion of the reduction test. The process repeated for all the temperatures from 1000°C to 1500°C. Both briquettes and iron ore were fired from 1000°C to 1500°C with a soaking period of 2 hours. Photograph of the apparatus is given in figure 6 and figure 7.

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Figure 6. Set-up for reduction test

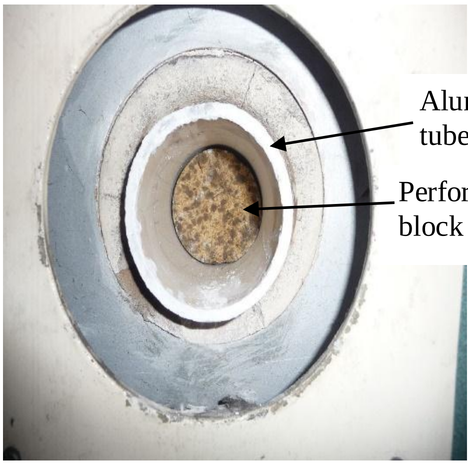


Figure 7. Crucible & alumina tube setup.

3. Results and discussion

3.1 Effect of Temperature on the Reduction of Briquettes and Iron ore lump

The degree of reduction and metallization were calculated. The results of metallic iron, total iron, the degree of reduction and % metallization for iron ore lump are given in table 7. The results of metallic iron, total iron, the degree of reduction and % metallization for briquette are given in table 8. The degree of reduction of briquettes and iron ore lumps was determined by using the formula given in equation (1).

$$\text{Degree of reduction } (\%) = \left(\frac{W_i - W_f}{W_o}\right) \times 100 \qquad (1)$$

Where W_i = the mass of sample before firing. W_f = the mass of the sample after firing. W_0 = the total mass of oxygen present in every individual mixture in the form of Fe_2O_3, Fe_3O_4 & FeO.

The degree of metallization of briquettes and iron ore lumps was determined by using the formula given in equation (2)

$$\text{Degree of metallization } (\%) = \left(\frac{\text{Metallic Fe}}{\text{Total Fe}}\right) \times 100 \qquad (2)$$

Table 7. Degree of reduction & Metallization for Iron ore.

Temperature (^{0}C)	Fe_M (%)	Fe_T (%)	Metalization (%)	Degree of reduction (%)
1000/2 hr	2.16	67.48	3.20	35
1100/2 hr	12.69	69.52	18.25	52
1200/2 hr	17.65	70.25	25.10	61
1300/2 hr	30.33	73.3	41.30	65
1400/2 hr	53.75	80.79	66.53	82
1500/2 hr	84.25	90.4	93.20	93

Table 8. Degree of reduction & Metallization for Briquette.

Temperature	Fe_M	Fe_T	Metalization (%)	Degree of reduction (%)
1000/2 hr	14.20	60.5	23.5	48
1100/2 hr	20.96	61.7	33.97	62
1200/2 hr	30.36	63.7	47.66	72
1300/2 hr	44.74	65.6	68.2	80
1400/2 hr	47.04	67.46	69.7	88
1500/2 hr	79.22	80.87	97.95	99

Table 7 and 8 show that briquettes have a higher degree of reduction than iron ore lump since the reducibility of lumpy iron ore dependent on lump size [4]. The reason for this is evidently related with the presence of the carbon in the BF sludge which is highly reactive and helps for gasification reaction at a comparatively lower temperature where iron ore fines work as a catalyst. From the mineralogical point of view iron ore after heat treatment >1100°C forms iron silicate and iron aluminium silicate which has a melting point around 1200°C. This melting temperature goes down to 1150°C with 1-2% lime. This envelopes the grain and seals the pores resulting in retardation of reducibility. Also, reducibility of iron silicate or iron aluminium silicate is very poor and requires very high temperature. In the case of briquettes, it forms calcium ferrite. Even BF dust also contains some calcium ferrite. The melting temperature of calcium ferrite is high which is >1400°C facilitating the pores which help in better reduction. Apart from this reducibility of calcium ferrite is even better than Magnetite. So to say a reason for higher reducibility of briquette appears to be due to their higher porosity values. Swelling index of briquette at different temperatures obtained are shown in table 9. The swelling indices of briquettes were determined by using the formula given in equation (3).

$$\text{Swelling index (\%)} = \left(\frac{V_f - V_i}{V_i} \right) \times 100 \qquad (3)$$

Where V_f = initial volume of the briquette. V_i = final volume of the briquette.

Table 9. Swelling index at different firing temperatures of briquette

Firing Temperature (^{0}C)	Swelling Index (%)
1000^0	19
1100^0	15
1200^0	13
1300^0	9
1400^0	4

The observed values of swelling index of the briquette are less than 20.This indicating normal swelling at each of the studied firing temperatures. Table 9 also indicates that the value of swelling index decreased drastically with a rise in firing temperature. This is expected to be due to the sintering of iron particles. The sintering effect restricts the growth of fibers responsible for swelling [5].

3.2 X-ray Analysis of Reduced Sample

Phase analysis was done by X-ray diffractometer. X-ray Diffraction patterns of iron ore fired at 1300°C, 1400°C & 1500^{0}C are shown in figure 8.

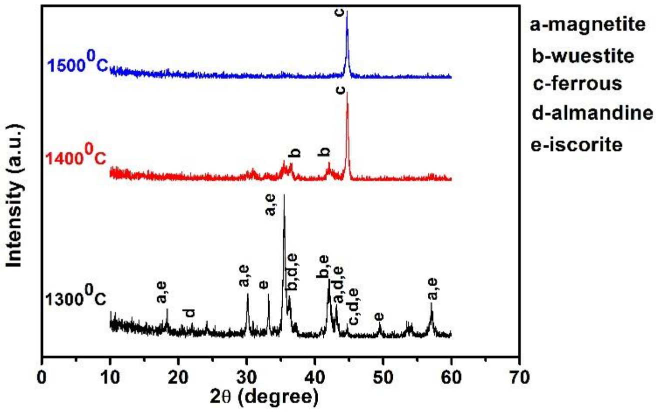


Figure 8. XRD analysis of iron ore fired at 1300°C, 1400 °C, and 1500 °C for 2hr.

At 1300^{0}C, the presence of magnetite and wustite as major phases with ferrous as a minor phase confirms that hematite had been reduced to the next level in the reduction process. At 1400^{0}C, the presence of ferrous as a major phase along with wustite as a minor phase confirms the complete reduction of magnetite to wustite and iron, for which degree of metallization had been enhanced to a greater extent. XRD pattern of reduced iron ore at 1500^{0}C shows that wustite was completely reduced to metallic iron and for which highest degree of metallization was achieved.

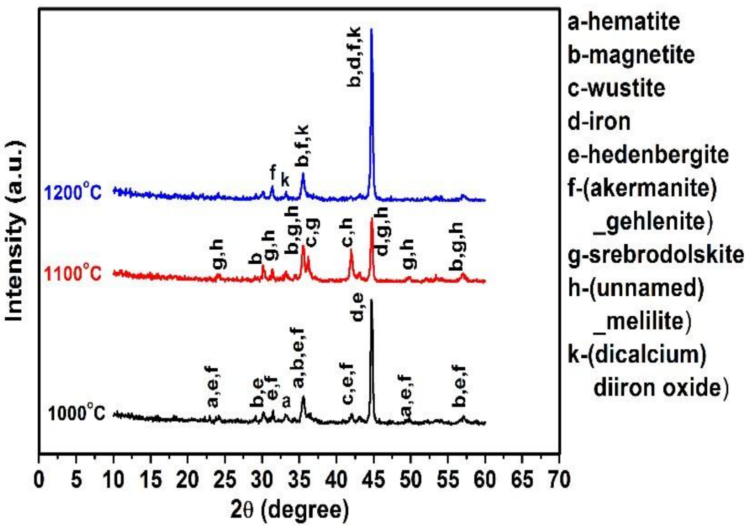


Figure 9. XRD analysis of briquettes fired at 1000 °C, 1100°C & 1200 °C.

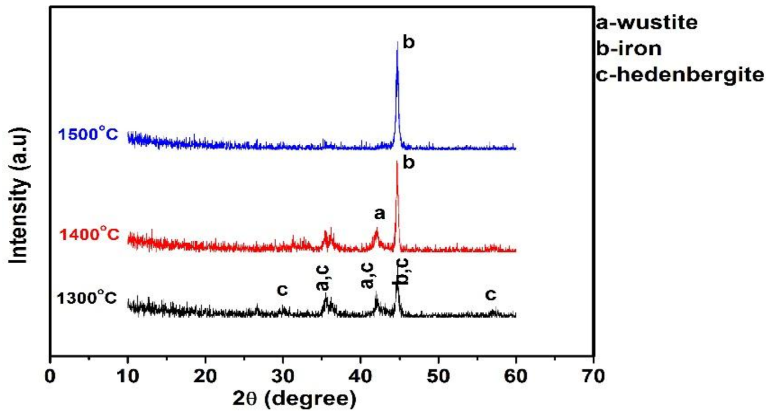
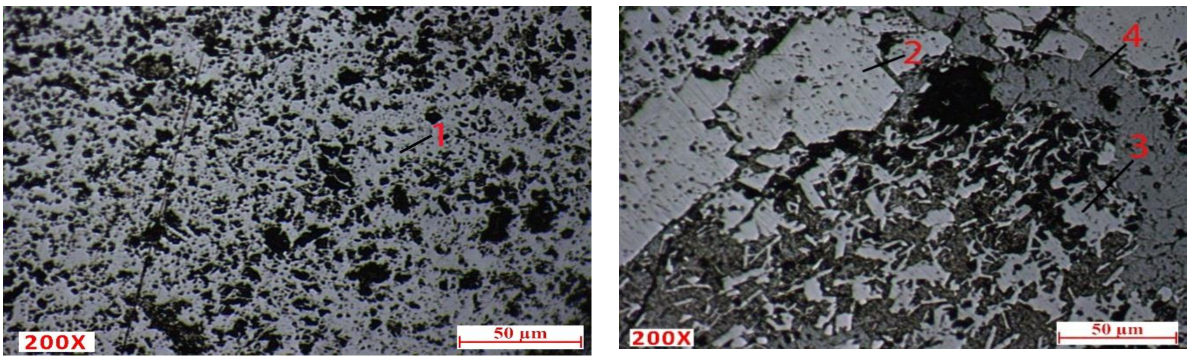


Figure 10. XRD analysis of briquettes fired at 1300 °C, 1400°C & 1500 °C.

XRD pattern of reduced briquettes fired at 1000°C, 1100°C and 1200°C are given in figure 9. XRD pattern of reduced briquettes fired at 1300°C, 1400°C and 1500°C are given in Figure 10. At 1300°C wustite and ferrous phases are present, whereas, only ferrous phase found in the diffraction pattern of briquette reduced at 1500°C. The presence of wustite and iron at 1400° lowered the degree of metallization value as compared to that of briquette reduced at 1500° C.

3.3 Microscopic Analyses

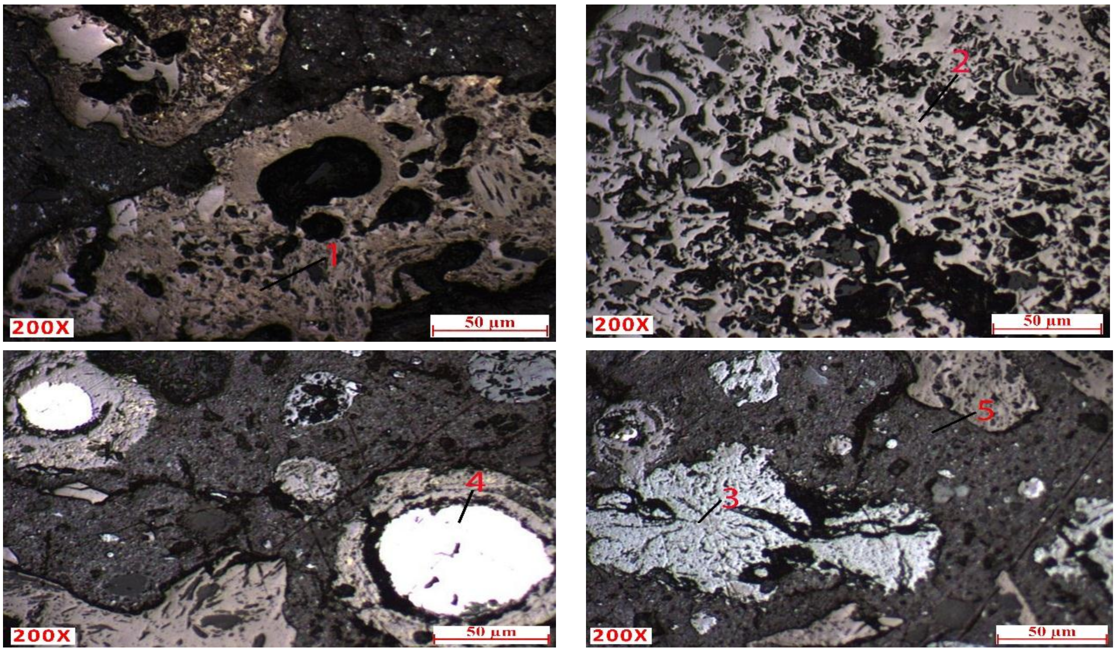
Microscopic evaluation of iron ore and briquette fired at the different temperatures were evaluated on the polished section under the reflected light in a universal microscope. Optical micrograph of iron ore fired at 1200°C is given in figure 11. Photomicrograph of briquette fired at 1200°C is given in figure 12.



1- Hematite, 2- Magnetite, 3- Wustite, 4- Glass

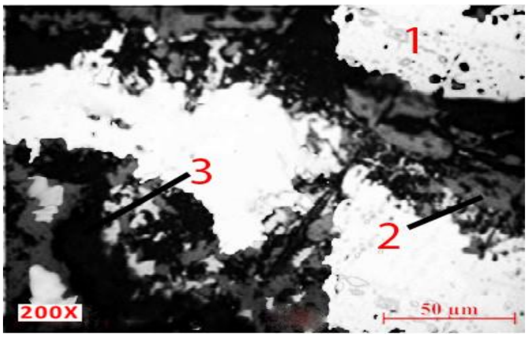
Figure 11. Optical microscopy of the reduced iron ore lump at 1200^{0}C in reducing atmosphere for 2hr.

From below microscopic images of reduced briquette at 1200^{0}C shows the presence of Dicalcium ferrite, which is responsible for faster reduction. At temperature 1400°C in figure 13, one can see the structure of the metal surrounded by the silicates and ferrites. Thus, the metallization of the surface layer of the agglomerated product helps to keep the integrity during its reduction with the creation of the metallic phase supported by the silicate and a ferrite matrix [6]. Blast furnace operating parameters of a 23 M^3 blast furnace, given in table 10 where the charging % of extruded briquette varied w.r.t lumpy iron ore.



1- Hematite, 2- Magnetite, 3- Wustite, 4- Metallic Fe, 5- Dicalcium ferrite

Figure 12. Optical microscopy of the reduced briquette at 1200^{0}C in reducing atmosphere for 2hr.



1- Metal, 2- Ferrite, 3- Silicate

Figure 13. Optical microscopy of the reduced briquette at 1400^{o}C in reducing atmosphere for 2hr.

Table 10. Blast Furnace operation parameters.

BF operation parameters	100% iron ore	80% Briquettes	100% Briquettes
Feed Material	Iron ore, Limestone, Dolomite, sponge iron	Briquettes, Iron ore, Dolomite, Mn ore	Briquettes, Iron ore, Dolomite, Mn ore
Fe content of the charge (%)	58	50.5	46
Blast temp in ^{o}C	950	950	975
Tuyere pressure (kg/cm2)	0.45-0.5	0.38-0.44	0.38-0.44
Hot metal Temp in ^{o}C	1410-1430	1400-1420	1400-1420
Slag temp in ^{o}C	1430-1450	1430-1440	1430-1440
Coke cons/TLM in kg	680	530	490
Slag volume in kg/TLM	360	420	450

Hot metal chemistry (%)			
Si			
Mn			
C	1-1.5	0.7-0.9	0.5-0.7
S	0.3	0.3-0.4	0.3-0.4
P	3.9-4	3.8-4	3.8-4
Fe	0.05-0.06	0.04-0.05	0.03-0.035
	0.09-0.11	0.09-0.11	0.09-0.1
	93-93.5	93-93.5	93-93.5
Slag chemistry (%)			
CaO	34.86	33.12	34.43
SiO_2	31.98	31.13	31.17
Al_2O_3	19.16	17.98	17.3
MgO	9.46	9.65	9.99
TiO_2	0.3	0.25	0.31
	1.2	1.16	0.67
FeO			

4. Conclusions

- Briquettes made by stiff extrusion principle are a novel technique for making briquettes which do not require any heat treatment. Moreover, it is utilized all the waste which is difficult to use at present.
- The briquettes retain its integrity up to the softening till it enters the cohesive zone where it gets melted.
- Briquettes made by this technique shows better reducibility than iron ore. Because major silicate phases after firing iron ore at different temperature are iron silicate and iron aluminium silicate which envelopes the grain and retard reduction. In the case of briquettes formation of ferrite which is easier to reduce than iron silicate or iron aluminium silicate.
- 100% briquettes indicate minimum reductant consumption despite the slag volume is high.
- Burden with 100% briquettes indicates improved hot metal quality as the silicon content is much lower.
- Utilization of waste helps to preserve the environment by utilizing the waste and less generation of greenhouse gas.

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