SCITECH
RESEARCH ORGANISATION

Volume 2, Issue 1
July 12, 2015

Journal of Progressive Research in Chemistry
www.scitecresearch.com

Characterization of Fluxed Iron Ore Pellets as Compared to Feed Material for Blast Furnace

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Abstract:

The effect of lime addition in hematite iron ore pellets on its physico-chemical as well as mechanical properties with respect to blast furnace requirement was studied in a laboratory scale with varying basicity i.e. 0-2.0.Increasing addition lime produced more calcium-alumino-silicate phases (slag) as evidenced by SEM-EDAX analysis. These phases have low melting points which enhances sticking behaviourof pellets, as well as imparting strength to the pellets (resulting increasing compressive strength, tumbler, abrasion and shatter index) and decreasing the porosity. The low basicity pellets were predominantly oxide-bonded, while high basicity pellets were mostly slag bonded. This means that pellets should be fired at sufficiently high enough temperature to generate liquid phases to get the sufficient strength but not so high as to cause pellets to stick. The properties of fluxed pellets were compared with the properties of iron ore lump and pellets, which are being used in blast furnace for production of iron and steel

Keywords: Calcium Aluminium Silicate; Lime Fluxed Pellets; Characterization; Strengthening Behavior; Shatter and Tumbler Test.

1. Introduction

World Blast Furnace Iron (BFI) Production in the year of 2012 was 1100.674 million tons in which India produced 42.258 million tons occupying 4th position in the world, whereas World Direct Reduced Iron (DRI) Production in the year of 2012 was 55.361 million tons in which India produced 19.799 million tons occupying 1st position *(Mandal et al., 2012).*

Iron ore is mined ~1600 million ton in the year of 2010, *(UNCTAD, 2010)* in which almost 98% of the mined iron ore is used in iron & steel making *(USGS, 2013).* Mined iron ore can be used directly as lump ore or converted into sinter, pellets etc. or to be reduced either by direct reduction or in a blast furnace. In 2010, around 25% of the mined iron ore was converted into pellets and its demand is increasing day by day *(UNCTAD, 2010).*

As reported by *Sarkar et al. (2013)* that fines generated in India was 125.1 million tons in 2011 which is almost 60% of total iron ore mined. Hence, agglomeration of iron ore is very essential process to utilize these fines. The agglomerates (pellets) quality plays a vital role in decreasing the consumption of reducing agent (i.e. Coke) and increasing the productivity of blast furnace. In most of the integrated steel plants, the burden mix for the blast furnace is decided as per the availability of the iron ore agglomerates like sinter and pellets (*Dwarapudi et al., 2011).* In India an average pellets used as burden is nearly 15-20% which is 100% acidic pellets *(IBM 2012).* Acid pellets are known for their poor high temperature properties like softening–melting characteristics and reducibility (*Onoda et al., 1980, Dwarapudi et al., 2010*).

The use of various types of binder for making pellets was studied by iron ore industry which was reviewed by *Eisele and Kawatra (2003).* Based on the reviewed article, it was found that the most common binder is bentonite. The addition of 0.5–0.7% are usually made for making pellets which increases the input of acidic oxide inside the pellets *(Forsmo et al.,*

2006) .The acid oxides have adverse effects on the iron–steelmaking economy. Some forms of sodium bentonite contain more than 65% SiO_2 by weight or 85% SiO_2 and Al_2O_3. Besides the adverse effect on pellet chemistry, this additional silica blocks the porosity of the pellet inhibiting the flow of reducing gases into the interior regions of the pellet. This lowers the reducibility of pellets means increases energy requirements in the iron-making process and also increases the costs for handling and disposal of increased slag (*de Souza et al., 1984*). An increase in silica content of pellet charged by 1%, leads to an increase in the unit cost of making the steel by US $ 4–7 /ton (*Chizhikov et al., 2003*). In the case of direct reduced pellets, every percent of acid gangue addition is associated with an increased energy consumption of 30kWh/ton (*Heerema et al., 1989*). Uses of pellets having lower gangue concentrations, substantial improvements in energy and flux consumptions can be realized in iron making processes. Cost savings in energy/flux for each 1.0% reduction in silica can reach $2.50 (U.S.)/ton of hot metal making (*Schmitt, 2005*).

Inorganic material like hydrated lime has a slight advantage over use of bentonite when silica levels are the primary concern of steel makers. However, most of them are incapable of controlling water addition during pelletization, and do little contribution to the cohesive/adhesive forces required to form and maintain green pellet integrity. This results in poor quality green balls that are fragile and easily broken, lowering production rates in iron making facilities. *(Sarkar et al. 2013).*

More attentions have been given in recent years to use of fluxed pellets in blast furnace due to their good strength and improved reducibility, swelling and softening–melting characteristics *(Firth et al., 2008).* Generally, quality of pellets is influenced by the nature of ore or concentrate, associated gangue, type and amount of flux added and their subsequent treatment to produce pellets. These factors in turn result in the variation of physicochemical properties of the coexisting phases and their distribution during pellet indurations. Hence, properties of the pellets are largely governed by the form and degree of bonding achieved between ore particles and the stability of these bonding phases during reduction of iron oxides (*Panigraphy et al., 1990*).

In fluxed pellets, the bonding is achieved through silicate melt formation during indurations. The amount of gangue in the ore concentrate, fluxes and binder, influences the amount and chemistry of silicate melt. CaO fluxes silicate melt as well as reacts with iron oxide to form different calcium ferrites. *Panigraphy et al, (1984)* have presented a detailed report in their technical paper on limestone and dolomite fluxed pellets. They found that, with increasing basicity more amount of liquid phase was formed during induration and hence more strength and less porosity were observed.

Hence, in production of acid, basic or fluxed pellets, the characterization of bonding and crystalline phases are of prime importance in understanding the basis for the production of desired quality pellets. Investigations have been carried out by several researchers on the bonding behavior in fluxed hematite pellets of the basicity 0.5 to 1.00 *(Frazer et al.,1975; Friel and Erickson, 1980; Dwarapudi et al.,2011)* and basicity 0.08 to 1.15 (*Umadevi et al,2011)* . *Friel and Erickson (1980)* had also made good quality dolomite fluxed magnetite pellets with MgO contents vary from 1-2 wt % and CaO/SiO_2 ratios from 0.6 to 1.3. Specifically, compositions within these ranges have good physical properties (tumble >98 %, compressive strength > 2670 N), and they are significantly more reducible and soften considerably at higher temperatures than acid pellets.

Based on the literature, it was cleared that the most of the people have not yet studied the mechanical (shatter and tumbler, abrasion crushing strength etc) as well as physical (porosity, density etc) properties of hardened fluxed hematite pellets properties with respect to blast furnace requirement with varying basicity. The aim of the present study is to deal with the determination of different physical, microstructural as well as mechanical properties of three types of distinct chemical compositional fluxed fired hematite iron ore pellets which will be compared with the property of lumpy hematite iron ore and hence, the properties of fluxed pellets were optimized for their use in the Blast Furnace operation.

2. Experimental

2.1. Materials and Their Characterization

In the present investigation, hematite iron ore was procured from the mines of Barbil, Orissa (India) and limestone from mines of Rajasthan (India) respectively. Lime was made in laboratory by heating limestone at 1050°C @5°C/minute in a muffle furnace and kept for isothermal holding for a period of one hour followed by furnace cooling. The chemical analyses of samples were done using XRF analysis (model-ARL OPTIM'X X-Ray analyzer) as shown in Table 1. X-Ray diffraction analysis was performed by RIGAKU D-MAX IIIB under the following condition, CuKα as emission radiation source (λ = 1.54178Å), Voltage =40kv, Intensity =30 mA, Scan rate 3°/ min at the range of 3-85°.

Table 1: Chemical Analysis of Raw Materials

Raw Material	Fe(T)	SiO_2	Al_2O_3	CaO	MgO	Mn	P	S	Fe_2O_3	Alkali	LOI
Iron Ore	64	1.8	2.6	-	0.08	0.07	0.03	.001	-	0.1	2.5
Lime Stone	-	1.2	1	52	2	-	0.022	-	0.5	0.23	40

The value of iron content in the iron ore was 64% and 52% CaO was in Lime Stone. The analysis of mineralogical composition of iron ore indicated that the gangue content in the iron ore were mainly alumina and silica with a negligible amount of MgO, Mn, P, S and alkali. The details phase analysis of iron ore and lime fines were mentioned in our earlier work *(Sarkar et al, 2013)* which reveals that % Fe_2O_3 in iron ore was around 95%. It means that iron oxides present in iron ore mainly as hematite which is soft in nature and similarly, CaO present as a major phase with little amount of SiO_2 and Al_2O_3, no major foreign particles were observed.

Iron ore were crushed to -1 mm in roll crusher before grinding. Burnt lime and iron ore samples were ground separately to achieve required fineness using 5 kg capacity laboratory scale ball mill to 80 % passing 0.045 mm to get optimum fineness for pellet making as per industrial practice *(Sinha and Sharma, 2014).*

2.2. Preparation of Iron Ore Pellets

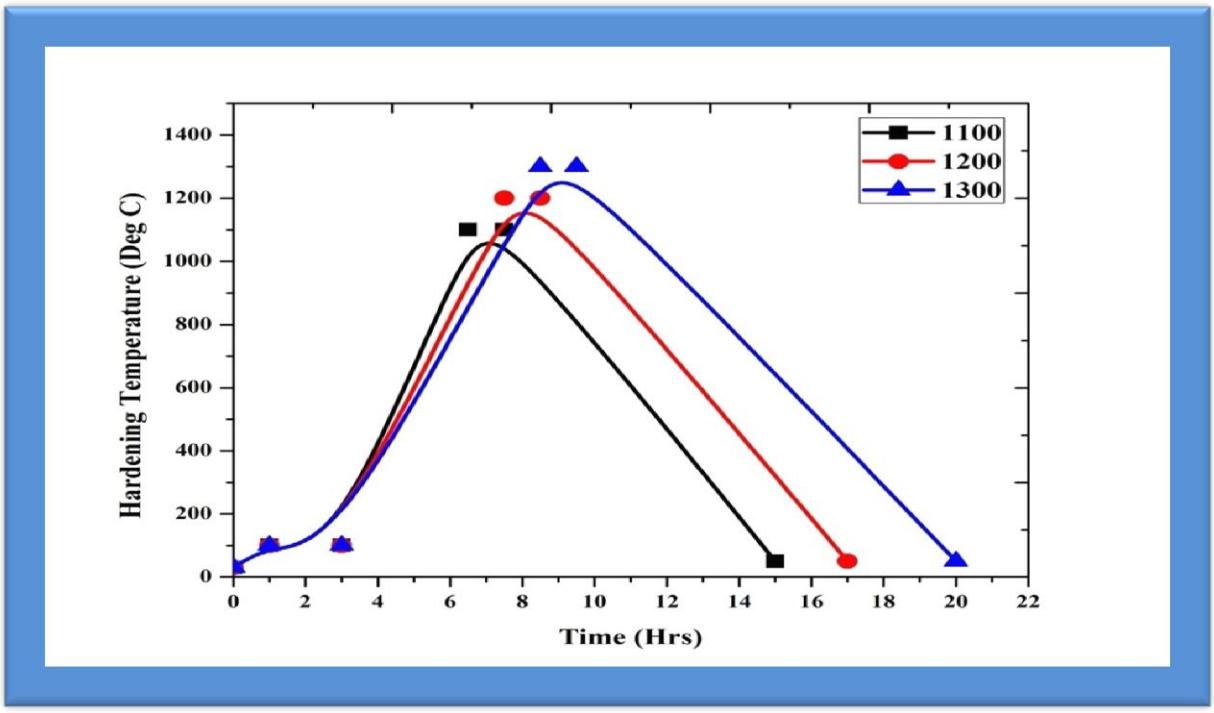
The iron ore fines were thoroughly mixed with different lime fines (0, 2, and 4%) in dry condition, as indicated in Table 2. Pellets (size: 15-16 mm diameter) were made with moistened iron ore fines/ iron ore-lime fines mixture by prolonged hand rolling without bentonite. The optimum moisture content (OMC) and pelletization behavior test of green pellets was performed. A brief description of the procedures adopted for determination of OMC and green pelletization behavior is outlined in our previous article *(Sarkar et al., 2013)* and the results obtained are listed in Table 2.

Table 2: OMC and Composition of Pellets Made

Moisture (%)	Iron ore (%)	Lime Fines addition (%)	SiO_2 input (%)	CaO input (%)	Pellets Basicity (B_1= CaO/ SiO_2)
14	100	0	1.8	0	≈0

Green pellets were air dried for two days followed by oven dried at 110°C for 2 hrs in an electric oven to get moisture free and attained sufficient strength for subsequent handling. Dried pellets were hardened in a resistance furnace at different temperature of 1100°C, 1200°C and 1300°C for 1 hour to attain a workable strength as per heating cycle mention in the Figure 1. Three pellets of each composition from each step were taken out for testing purpose.

Figure 1: Graph of Different Hardening Cycle



2.3. Testing Methodology

Apparent porosity, apparent density, true porosity, true density and sealed porosity of hardened pellets were determined by the Boiling Water method as per *ASTM C-20-00 (2010)*and Water Pycnometer as per *ASTM C135 - 96(2009)* respectively. Results are reported in Table 3.

Table 3: Physical Properties Hardened Pellets Along with Lump Ore

Parameters	Pellets									LUMP ORE
Hardening temp	1100° C			1200° C			1300° C			
Pellets type	Acid	Neutral	Basic	Acid	Neutral	Basic	Acid	Neutral	Basic	
Apparent Porosity (%)	38.44	40.95	56.5	42.78	44.61	42.8	30.31	24.19	9.96	8.1
Apparent Density(g/cc)	2.568	2.313	1.749	2.295	2.289	2.23	3.09	2.7	2.69	3.1
True Density(g/cc)	4.41	4.1	4.08	4.64	4.21	4.09	5.31	4.48	4.39	3.66
True Porosity (%)	41.77	43.58	57.13	50.54	45.64	45.47	41.8	39.73	38.72	15.3
Sealed Porosity (%)	3.33	2.63	0.63	7.76	1.03	2.67	11.49	15.54	28.76	7.2

The cold crushing strength of air dried, oven dried, hardened iron ore pellets as well as oven dried lump iron ore sample have been determined by using a low range UTM (SHIMADZU, Type: SBL, P/N: 340-43120-01, Capacity-5kN) at a very slow speed of 0.05cm/minute.

Tumbler, Abrasion and Shatter indices of hardened pellets as well as oven dried iron ore samples were determined. For tumbler and abrasion indices a standard weight (10 Kg) of oven-dried lump iron ore (-40+10 mm) in a standard drum (dia =1000mm, width =500 mm and lifter height 50 mm) and rotate 200 revolution @25rpm. The percentage weight of material passing through a 0.5 mm screen was represented its abrasion indices, while the percentage weight of material retained on a 6.3 mm screen was taken as its tumbler indices. In shatter test, dried sample of lumpy iron ore of same size and quantity was dropped four times from a height of 2 m on a cast iron floor. The percentage weight of material remaining over 10 mm screen iron ore is represented as the Shatter indices *(Tupkary and Tupkary 2003)*. In case of hardened iron ore pellets 1 kg (nonstandard weight method) sample of pellets were taken for both above experiments *(Gupta, 2010)*. The Crushing strength, Shatter, Tumbler, and Abrasion indices results are summarized in Table 4 and 5 respectively.

Scanning electron micrographs to assess the surface characteristics and structural changes in some of the reduced iron ore pellets were obtained using SEM (FEI Quanta-200FEG) at 20kv on scan rate 10µs with ETD detector, to the failure surface of hardened iron ore pellets after crushing strength measurement. EDAX analysis was performed to determine the different phases present inside the pellets after hardening.

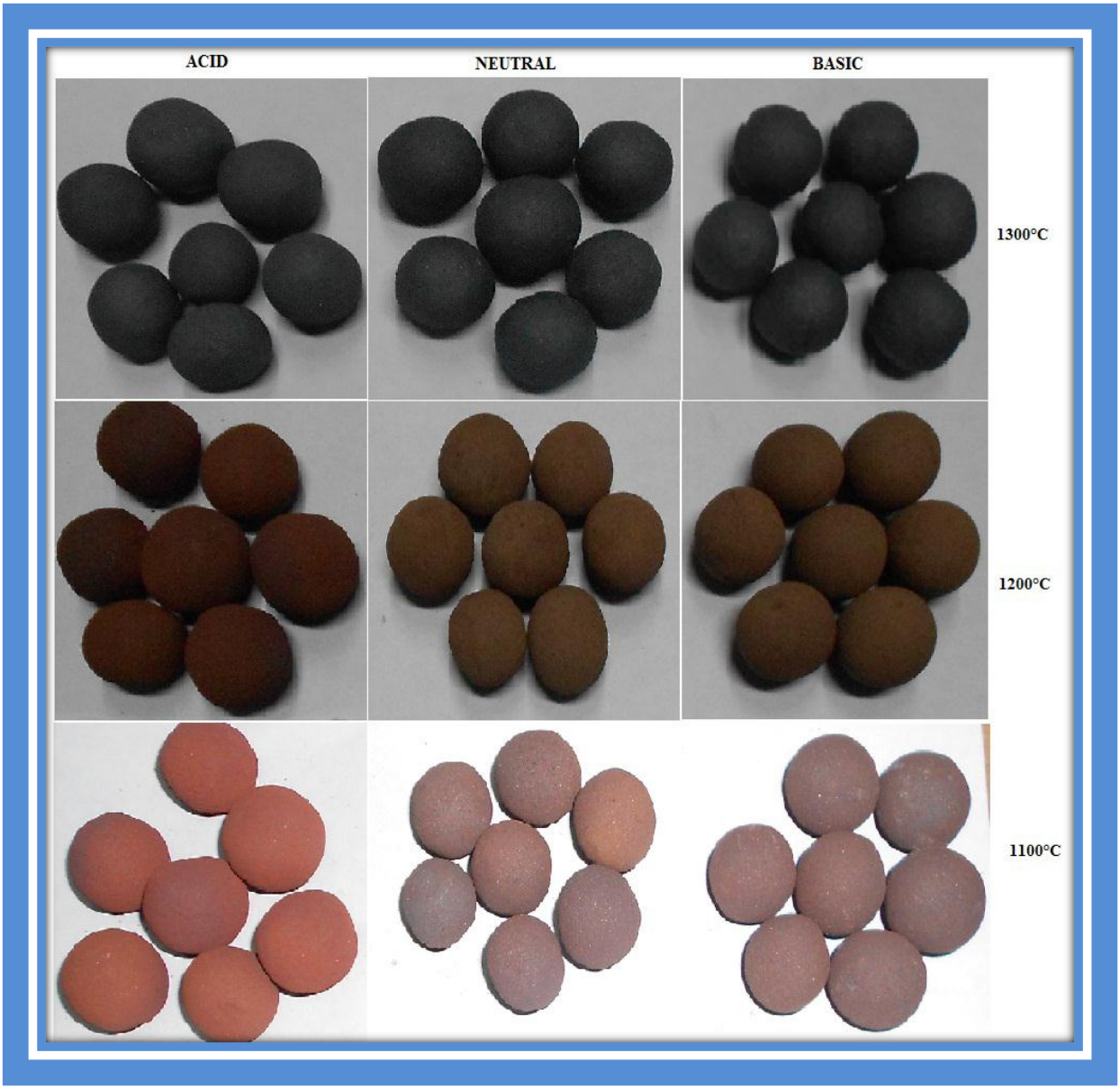
3. Results and Discussions

3.1 Physical Properties

3.1.1 Fusion Behavior and Colour Changes after Hardening

After hardening, colour change of the pellets was observed. It was seen that with increasing lime addition, the colour of the pellets faded from brown to whitish below 1300°C. At the temperature of 1300°C, all pellets were converted into black due to fusion and subsequently few pellets were agglomerated. The color changed at different hardening temperature as well as chemical composition was shown in Figure 2. In increasing temperature sticking tendency due to fusion became increased, therefore hardening temperature restricted to 1300°C.

Figure 2: Color Change in Hardened Pellets



3.1.2 Porosity and Density

With increasing temperature from 1100°C to 1200°C, apparent porosity value of iron ore pellets were observed 38 to 42 %. But at 1300°C hardening temperature, apparent porosity was decreased to 30%, but not upto lumpy iron ore (i.e. 6.7%). Addition of lime resulted in the formation of more amount of calcium silicate melt phase which fills up the pores between solid particles and exerts pressure to pull them together due to interfacial forces thereby reducing the porosity *(Dwarapudi et al, 2011)* .The nature of the trend for true porosity was found similar to the apparent porosity. With increasing lime content, porosity was also increased from temperature 1100°C to 1200°C, but reverse effect was observed in hardened pellets at 1300°C, due to formation of slag which enters into the pores of the pellets; hence it caused the lower porosity but increased % of sealed porosity (Table 3).

3.2. Mechanical Properties

3.2.1. Crushing Strength

Crushing strength of air dried, oven dried and hardened pellets were increased simultaneously as shown in Table 4 which showed that Crushing strength of all types of pellets fired at 1300°C are obtained above the acceptable limit for blast furnace (200kg/pellets). Highest strength of 586 Kg/pellet was observed for basic pellets which are much more than the value of accepted in industrial practice. After increasing hardening temperature, crushing strength was drastically increased from 53.75, 54.96, 56.67 Kg/pellet at 1100°C to 233.75, 486.67 and 586.76 kg/pellets at 1300°C respectively

and which was compared with lump ore i.e. 400Kg/cm^2. This is due to stronger slag bond formation inside the pellets as mentioned above in section 3.1.2. (*Specht et al, 1992, Takashi et al, 2009)* (Note: Pellets having spherical shape so during strength measurement it acts as a point load so strength value shown as Kg/pellts).

Table 4: Crushing Strength of Different Pellets Along with Lump Ore

	Dried (Kg/pellet)		Hardened (Kg/pellet)			Lump (Kg/cm^2)	Conventional pellet (Kg/pellet)
Nature of pellets	Air	Oven	1100°C	1200°C	1300°C	Oven dried	1300-1350°C
Acid	7.75	12.67	42.5	53.75	233.75	400	200
Neutral	8.65	14.21	45.7	54.96	486.67		
Basic	9.75	16.8	48.33	56.67	586.76		

3.2.2. Shatter, Tumbler and Abrasion Indices

Data for the strength properties (tumbler, abrasion, and shatter indices) of hardened iron ore pellets and lumpy iron ores have been presented in Table 5. These are the most popular properties to assess the resistance to degradation during handling before reduction in blast furnace. It was observed that tumbler and abrasion indices of hardened iron ore pellets at temperature 1100-1200°C were decreased as compare to lump ore sample but increased with increasing hardening temperature. In 1300°C hardening temperature, offered high resistance to abrasion and tumbling, most likely due to their hard fine-grained structure .Tumbler and abrasion indices of the harden pellets were found superior than lump ore and also fulfill the industrial requirement of minimum 94 % tumbling index as well as maximum 5 % of abrasion index *(IBM 2012)* .

Furthermore, results obtained (Table 5) indicated a lower value of shatter index (for -5 mm size fraction) in all type of pellets at the temperature range of 1000-1100°C whereas a higher values were observed for hardening temperature of 1300°C as already observed in case of tumbler and abrasion indices. Based on the tumbler, abrasion, and shatter indices results, pellets of hardened at 1300°C appear to be hard and strong. On the other hand, rest pellets to be more porous, soft, and friable and, hence, are liable to produce more deleterious fines (-5mm fraction) during handling or in iron blast furnaces, rotary kilns, etc. As evidenced from Table 3, 4 and 5, there is a definite correlation between porosity and strength properties (tumbler, abrasion, and shatter indices) exists which indicates that with increasing strength, shows decreasing porosity.

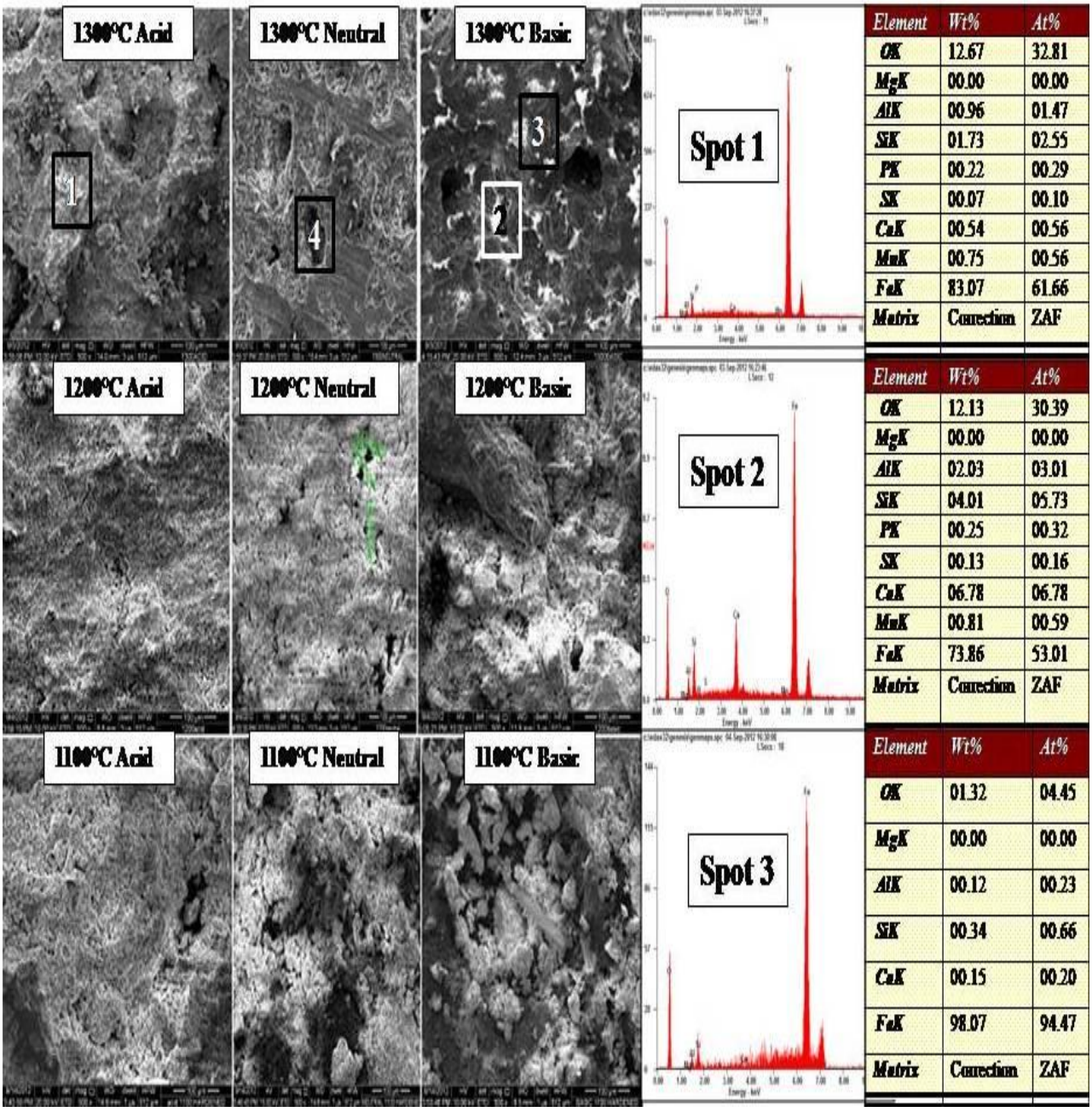
Table 5: Shatter, Tumbler and Abrasion Index of Harden Pellets Along with Lump Ore

Hardening temp	Pellets	Shatter index (%)	Tumbler index (%)	Abrasion index (%)
1100°C	Acid	63.18	94.34	5.66
	Neutral	85.62	98.5	1.5
	Basic	86.54	99.53	0.47
1200°C	Acid	69.71	85	4.45
	Neutral	77.05	86	2.43
	Basic	95.49	90	0.36
1300°C	Acid	100	99.8	0.2
	Neutral	100	100	0
	Basic	100	100	0
LUMP IRON ORE		**94.4**	**90.2**	**6.7**

3.3. Microstructure

SEM images with EDAX analysis of hardened pellets were shown in Figure 3. At lower hardening temperature (1100°C) more porosity and free particles were observed which indicates that there is no formation of slag constituents. But at 1300°C hardening temperature, particles were fused and agglomerated resulting less porosity and more strength which was also observed physically as discussed in section 3.1.1. In Figure 4, it was clearly shown that at high hardening temperature, slag bridge (mainly responsible for high strength) tightly bonded to get the strength. With increasing lime content silicate slag phase transformed to calcium silicate slag, which will generate high strength at high temperature**.** This behavior was also confirmed by many other authors *(Pal et al, 2009)*.

Figure 4: SEM micrographs of hardened pellets of 0wt% lime, 2wt% lime and 4wt% lime (Spot 1: Aluminium silicate slag phases at lower basicity ; Spot 2: Calcium –Aluminium Silicate slag Phases at higher basicity; Spot 3: Metallic phases and Spot 4: porosity)



Element	Wt%	At%
OK	12.67	32.81
MgK	00.00	00.00
AlK	00.96	01.47
SiK	01.73	02.55
PK	00.22	00.29
SK	00.07	00.10
CaK	00.54	00.56
MnK	00.75	00.56
FeK	83.07	61.66
Matrix	Correction	ZAF

Element	Wt%	At%
OK	12.13	30.39
MgK	00.00	00.00
AlK	02.03	03.01
SiK	04.01	05.73
PK	00.25	00.32
SK	00.13	00.16
CaK	06.78	06.78
MnK	00.81	00.59
FeK	73.86	53.01
Matrix	Correction	ZAF

Element	Wt%	At%
OK	01.32	04.45
MgK	00.00	00.00
AlK	00.12	00.23
SiK	00.34	00.66
CaK	00.15	00.20
FeK	98.07	94.47
Matrix	Correction	ZAF

4. Conclusions

Following conclusions were drawn from the present study:-

1. It has been possible to use fluxed iron ore pellets using iron ore and lime fines as a raw material to minimize flux input in Blast Furnace as a limestone and avoid bentonite addition as binder in conventionally prepared pellets.
2. The preparation of fluxed iron ore pellets with high basicity (1.17) has been possible without sticking pellets upto 1300°C hardening temperature.
3. The use of lime contents up to 4 wt% (pellet basicity 1.17), in the pellets at 1300°C temperature for one hour hardening period, shown higher crushing strength(586.76Kg/pellet) and lower apparent density (2.69 gm/cc) with respect to lump ore(400 Kg/cm^2 and 3.1gm/cc). The formation of more calcium silicate in basic pellets was imparted higher strength to the pellets in comparison to acid pellet.
4. Shatter, tumbler and abrasion resistance of fluxed hardened pellets were found superior at 1300°C (i.e. 100%, 100%, 100% ; 99.8%, 100%, 100% and 0.2%, 0%, 0% for acid, neutral and basic pellets respectively) compared with lumpy iron ore (94.4%, 90.2% and 6.7%).
5. Apparent and true porosity of hardened fluxed pellets were improved than lump ore. For example, apparent porosity 38.44%, 40.95%, 56.5% for 1100°C; 42.78%, 44.61%, 42.8% for 1200°C and 30.31%, 24.19%, 9.96% for 1300°C for acid, neutral and basic pellets respectively in comparison with lump iron ore i.e. 8.1%.
6. With increasing hardening temperature, apparent porosity value was increased tremendously upto 44.61 % at 1200°C. But at 1300°C, slag inside the pellets fused resulting less porosity as lower as 9.96 % which enhanced crushing strength (586.76 kg/pellet) as compared to lumpy ore (400 Kg/c^{m2}).

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