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The Reducibility Studies On Itakpe And Agbaja Resultant Pellets

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Abstract: ***The reducibility study on resultant pellets using the available facilities to execute this research work. The chemical compositions and structural analyses of the pellets took place. The sample studied were examined with the use of Thermogravimetry(TG), Differential Thermal Analyzer (DTA), X-Ray Fluorescence (XRF), X-Ray Diffraction (XRD), Optical and Electron Microscopy and Scanning Electron Microscopy (SEM) with Energy Dispersive Spectroscopy (EDS). The processes were used to examine the morphologies of the pellets. The reducibility studies were performed in a muffle furnace with model number. The pellets were fired in the container with diameter 6.1 cm x 5.9cm made of stainless steel materials Metallurgical coking coal gotten from the Ajaokuta Steel Company Limited was used as reductant. The processes were monitored and the highest reducibility value obtained for the Itakpe pellet was at 99.7% with corresponding temperature at 1000^{o}C for a heating period of 120 minutes. On the other stage, 95.7% was the reducibility value obtained with Agbjaja pellets with corresponding temperature at 1000^{o}C at a heating period of 120 minutes. The samples were analysed before the reducibility tests and similar tests were also performed after the reducibility tests. The SEM and EDS tests performed on some selected the pellets show that reducibility took place in these order at 800^{o}C for 120 minutes, Fe Content was 4.5 wt%, at 920^{o}C for 120minutes, Fe content was 84.5wt% and at 1000^{o}C and 120 minutes, Fe content was 6.4wt% while the for Agbaja pellets had the reducibility in these order at 800^{o}C for 120 minutes the Fe content was 40.7wt%, at 920^{o}C for 120minutes Fe content was 60.2wt% and at 1000^{o}C for 120 minutes.***

Keywords: Reducibility, Study, Resultant, Pellets, Itakpe, Agbaja Iron Ore

I. INTRODUCTION

Iron ore deposit was discovered in Nigeria as far back as 1904 since then several deposits have been discovered. The deposits are hematite, magnetite, goethite or siderite – goethite grades. The reserve is estimated at over 3 billion metric tonnes and their utilization deposits in iron and steel plants will reduce the cost of importation thereby saving foreign exchange, improve Nigerian's technology transfer i.e. agriculture, military defence and provide employment and revenue generation [1].

The Itakpe iron ore deposit is located north-east of Okene in the eastern part of Kogi state, which is presently the most elaborately investigated ferrous deposit in Nigeria, this is been developed for utilization in the Blast Furnace at Ajaokuta Steel Company Limited situated at Ajaokuta Steel township and for steel production operation at the Delta Steel Company, Aladja situated in Delta state for the production of Direct Reduced Iron (DRI).

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The topography of the region is a plateau rising gently to the east, down to the river Niger. The plateau is bestrewn with scattered hills, which are made of Precambrian gneisses, and granites that overlook the surrounding by about 200m to 300m. The Itakpe deposits is part of these hills. Its estimated reserve is over 300 million tonnes while its proven reserve is 200 million tones [1].

Mineralogical studies of Itakpe deposits which has been well documented in the last few years yield the following features;

Magnetite – hematite quartzite; 19.7%

Hematite - magnetite quartzite; 37.5% Hematite quartzite; 25.9%. In these various forms, the average chemical or instrumental assay of vital elements or compounds indicates 36.8%Fe, 42.6%SiO_2, 1.0% Al_2O_3 and 0.05% P and 0.05% S. It is observed that the Fe grade is low to be used in either the blast furnace which requires about 60-62% Fe grade while the Direct Reduced Iron (DRI) requires about 65-68%Fe grade. The Itakpe iron ore has tolerable percentage of both sulphur and phosphorous in iron and steel making. The percentage acid gangue should be less than 3.5 %.

In this iron ore deposit, materials deleterious to iron and steel production (such as sulphur, phosphorous and non-ferrous metals) are absent from the mineral matrix. The particle size of the constituent iron minerals varies from coarse and fine-grained, (with total liberation from gangue at 600µm to 800µm which renders them suitable for conventional beneficiation techniques (like gravity and magnetic separation), unlike the extremely fine-grained Agbaja deposits of <5µm liberation size which respond poorly to these processing methods.

Mineralogy of Agbaja iron ore is Oolithic too in nature, limonite which occurs in mammilated or stalactite forms having fibrous structure resembling hematite [3]. The Iron ore is typically classified as high grade (+65% Fe), Medium grade (+62 – 65% Fe) and low grade (-62% Fe). Typically, the Integrated Steel Plants(ISPs) use medium/high-grade iron ore whereas the sponge iron plants require only high-grade iron ore, preferably, with +67% Fe.

The Agbaja iron ore is traded in lumps (i.e. sized ore) or in fines. Production/availability of lumps is limited by virtue of the natural occurrence and also because of the generation of a lot of fines during the crushing of large lumps present in the run-off –mines (ROM). Natural pellet is a term coined by producers in some Asian counties, to designate sized iron ore used directly in Sponge Iron production. Blue Dust is the name given to naturally occurring, extremely friable, high-grade hematite iron ore powder.

Very low-grade iron ore cannot be used in metallurgical plants and needs to be upgraded to increase the iron content and reduce the gangue content. A process adopted to upgrade ore is called beneficiation. Iron ore is upgraded to higher iron content through concentration. Iron ore is being beneficiated all round the world to meet the quality requirement of Iron and Steel industries. However, each source of iron ore has its own peculiar mineralogical characteristics and requires the specific beneficiation and metallurgical treatment to get the best product out of it. The choice of the beneficiation treatment depends on the nature of the gangue present and its association with the ore structure. Several techniques such as washing, jigging, magnetic separation, advanced gravity separation and flotation are being employed to enhance the quality of the iron ore.

Due to the high density of hematite relative to silicates, beneficiation usually involves a combination of crushing and milling as well as heavy liquid separation. This is achieved by passing the finely crushed ore over a bath of solution containing bentonite or another agent which increases the density of the solution. When the density of the solution is properly calibrated, the hematite will sink and the silicate mineral fragments will float and can be removed.

REDUCIBILITY: This is a name for the velocity of iron oxide transformation to metal by effect of reduction gas, or also a time needed for a complete iron oxide reduction. The value of the reduction rate is the metallic charge weight loss per time unit. The weight loss is caused by the charge oxygen transformation into gas.

The reducibility test method is one of the several procedures used to evaluate the behaviour of natural and processed iron ores and agglomerates such as pellets and pellets under specific conditions. On the other hand, Iron ore can be upgraded to a higher iron ore content through beneficiation. This process generates iron ore filter cake which needs to be pelletized and used in the steel making process. Also during the processing of high-grade iron ores which does not need beneficiated, fines which are generated can be pelletized and used instead of been disposed of iron ore pellets formed from beneficiated or run off mine iron fines. The iron is usually ground to a very fine level and mixed with limestone or dolomite as a fluxing agent and bentonite or organic binders as a binding agent. If the ore is a hematite ore, coke or anthracite coal can be added to the mix to work as an internal fuel to help fire the pellets.

This mixture is blended together in a mixer and fed to balling discs or drums to produce green pellets of size typically about 9-16mm. The green pellets are then fed to the induration machine. Both straight grates and grate kilns dry the pellets out in a drying section, then bring the pellets up to a temperature of about 800-900 °C in a preheat zone, then finish the induration process at roughly 1200-1350°C. The pellets are then cooled to a suitable temperature for transporting to a load-out facility. Both processes recycle the heat from the pellet back through the process to aid in energy efficiency and decrease fuel usage.

Both processes can be used to generate almost any type of desired pellet chemistry, from direct reduction pellets (DR pellets) to Blast Furnace pellets. By adjusting the amount of fluxing agent or limestone added, pellets can be made that are anywhere from acid (or non-fluxed) pellets to heavily fluxed pellets. In view of these Nigeria is blessed with abundant iron ore deposits located in some state of the Federation like Kogi, Enugu and Abuja etc It should be noted that Iron ore deposit was discovered in Nigeria as far back as 1904 since then several deposits have been discovered. Table 1.0 shows the Chemical Composition and Estimated Reserve of some Nigerian Iron ores. The deposits are hematite, magnetite, goethite or siderite – goethite grades. The effective harnessing of the estimated reserve deposits which runs to over 3.1 billion metric will generate employment opportunities, wealth creation, revenue generation and foreign exchange earnings as

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these will further improve technological base i.e. in the agricultural sector, military defence and provision of infrastructures and reviving of the moribund iron and steel industries across the nation. The pellets produced are to be used in the operation of the Blast furnace and Direct Reduced Iron (DRI) typical example of such furnaces are located at Ajaokuta Steel Company Limited, Ajaokuta Kogi State, while the other type of furnace is used at the Delta Steel Company Limited, Aldaja, Delta State.

II. MATERIALS

ITAKPE IRON ORE

The topography of the region is a plateau rising gently to the northeast of Okene in the eastern part of Kogi State, down to the river Niger. The plateau is bestrewn with scattered hills which are made of Precambrian gneisses and granites that overlook the surrounding by about 200m to 300m. The Itakpe iron ore deposits is part of these hills. Its estimated reserve is over 300 million tonnes while its proven reserve is 200 million tones. The deposit has an average iron ore content of 36%. This has to be beneficiated at the rate of 8 million tonnes per year to produce 64% Fe concentrate as sinter materials, for the Ajaokuta Steel Company Limited Blast furnace and 60% Fe concentrate as pellet feed for the Direct Reduction Plant (DRP) at the Delta Steel Company Limited, Aldaja, and Delta State. The iron ore is suitable as a feedstock to one of the Direct Reduction Methods of Ironmaking. The ore is typical of one formed by magnetic segregation. This iron ore deposit is the most elaborately investigated ferrous deposit in Nigeria, which is being developed for the utilization in the blast furnace. The picture below shows the sample of the Itakpe Iron ore sourced at the National Iron Ore Mining Company (NIOMCO) Itakpe, Kogi State, Nigeria. The Itakpe iron ore specimen is known to be a compacted, crystalline-like banded iron ore which has various colours like dark grey, brown and black. The Itakpe iron ore slightly magnetic in nature.

AGBAJA IRON ORES

The Agbaja Iron ore is an acidic pisolitic/ oolitic ore consisting of goethite, magnetic and major amounts of aluminous and siliceous materials. It cannot be used directly in the Blast Furnace process or another reduction process without further treatment e.g. pelletization or briquette. The ore is a lean ore and sedimentary origin. It is, therefore, necessary to harness the opportunities created to work upon the ore in order to add economic value to our national economy. The ore is also known to be oolittitic in nature, limonite which occur in mannmilated or stalactite forms having fibrous structure resembling hematite. The Agbaja Iron ore is made of brown compacted fine-grained materials which consist of extremely lager particles which show the tendency to be friable. Agbaja iron ore is strongly magnetic. The ore particles were further processed by crushing them for specific experimental procedure. The Agbaja iron ore sample is compacted ground fine particles which significantly exhibits the characteristics of being friable and also magnetically strong. The picture below shows the iron ore as being sourced at the Agbaja Plateau in Kogi State.

CHEMICAL ANALYSIS OF ITAKPE AND AGBAJA IRON ORE

Table 1 and 2 shows the result of chemical analyses of both Itakpe and Agbaja iron ore using an X-Ray Fluorescence (XRF) methods the experiments were performed at the laboratory of Tshwane University of Technology, South Africa.

Component	Unit	Result
Na_2O	mass%	0.354
MgO	mass%	0.385
Al_2O_3	mass%	12.21
SiO_2	mass%	20.53
P_2O_5	mass%	1.556
SO_3	mass%	0.138
K_2O	mass%	0.614
CaO	mass%	0.11
TiO_2	mass%	1.398
V_2O_5	mass%	0.085
Cr_2O_3	mass%	0.064
MnO	mass%	0.164
Fe_2O_3	mass%	52.54
NiO	mass%	0.025
CuO	mass%	0.033
ZnO	mass%	0.02
Rb_2O	mass%	0.013
SrO	mass%	0.014
ZrO_2	mass%	0.052
BaO	mass%	0.202

Table 1: Show the Chemical composition of Itakpe Iron Ore using XRF Method using XRF Method

Component	Unit	Result
Na2O	mass%	0.1183
MgO	mass%	0.2464
Al2O3	mass%	6.0470
SiO2	mass%	25.2321
P2O5	mass%	0.0227
SO3	mass%	0.1899
Cl	mass%	0.0955
K_2O	mass%	0.4294
Cao	mass%	0.4284
TiO_2	mass%	0.8505
Cr_2O_3	mass%	0.0895
MnO	mass%	0.4983
Fe_2O_3	mass%	43.7579
NiO	mass%	0.0530
CuO	mass%	0.0107
ZnO	mass%	0.0555
ZrO_2	mass%	0.0206
P_2O_5	mass%	trace

Table 2: Show the Chemical composition of Agbaja Iron ore

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X-RAY DIFFRACTION (XRD) ON ITAKPE IRON ORE

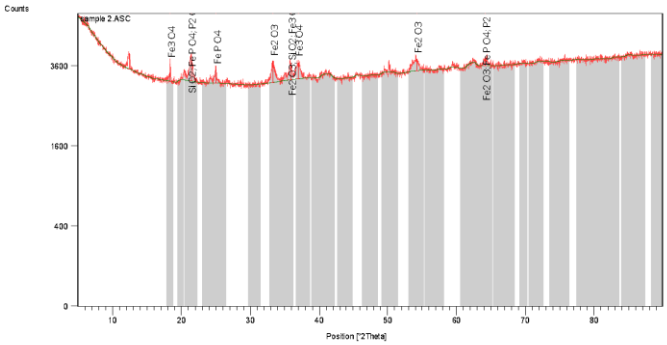

Figure 1: X-ray Diffraction on Itakpe Iron ore

X-RAY DIFFRACTION (XRD) ON AGBAJA IRON ORE

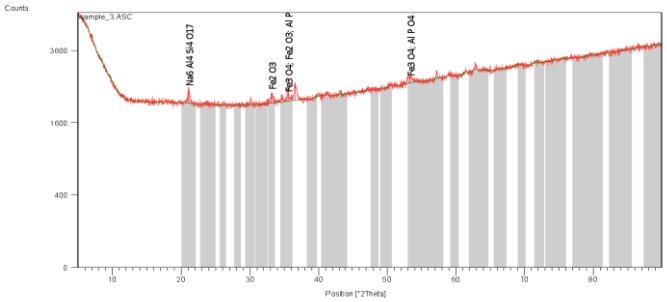

Figure 2: X-ray Diffraction on Agbaja Iron ore

METALLURGICAL COKING COAL FROM AJAOKUTA STEEL COMPANY LIMITED

The coal used for this experiment was selected from among the imported metallurgical Coking Coal at the Ajaokuta Steel Company Limited. The proximate analysis of the coal was done by the vendor before importing them to the steel company and the chemical compositions of the coal are shown in the table below:

S/No	Chemical Composition	Percentage (%)
1	Fixed Carbon	85
2	Volatile Matter	2.95
3	Ash Content	9.5
4	Moisture	2
5	Sulphur	0.5
6	Phosphorus	0.05

Source: Ajaokuta Steel Company Limited, Ajaokuta

Table 3: Chemical Analysis of Metallurgical Coking Coal

Equipment and Methods

The following equipment was used for the preparation of the two ore samples. Some of the experiments were carried out at the National Metallurgical Development Centre (NMDC), Jos Plateau State, other at the Laboratories in the Department of Metallurgical and Materials Engineering. The laboratories are located at the Nigerian Liquefied Natural Gas (NLNG) in the Department of the Metallurgical and Materials Engineering of the University of Nigeria (UNN), Nsukka, Enugu State and the Laboratories of Extractive Metallurgy at Tshwane University of Technology, South Africa.

Ball Milling Machine: The samples were crushed and ground and the experiments were performed at the National Metallurgical Development Centre, (NMDC) Jos was a Ball milling machine made by Bico Sprecher and Schn (2287) were used. The samples were further prepared by using sieve sizes of 0.5mm and 0.63mm respectively for the screening of the iron ore sizes to international standard. Pelletizing Machine: A palletization machines was used to produce the pellets. The machine used is with a model name Form and Test Seidner Strength testing machine, while an equipment with model name D7940, Salter Scale 50kg type was used for the weighing of the milled iron ore.

ELECTRONIC DIGITAL WEIGHING BALANCE: The Electronic Digital Weighing Balance with model name C&G GmbH Gielerister 65-69 (41460) Neuss, Germany was used for weighing of the produced pellets at the laboratory of the Department of Metallurgical and Materials Engineering, University of Nigeria, Nsukka.

BEAKER: Some beakers were filled with Benzene which was used for the determination of the Microporosity value after the iron ore samples had been heated to 900oC.

SAMPLE HANDLER: The produced pellets were carefully handled with a view to handling them properly without breakage

MUFFLE FURNACE: A Muffle furnace was employed for the heating of the produced pellets. The pellets were also heated for the purpose of determining the indurating compressive strength and Microporosity value before immersing the samples into the benzene solution. The furnace was also used for reducibility tests on the produced pellets.

THE LABORATORY DRY OVEN: The Laboratory Dry Oven with model number DHG 9101 was used for heating of the pellets at low temperature particularly with respect to determining the moisture contents of all the produced pellets.

TESTOMETRIC TESTING MACHINE: Testing of samples were carried with the use of the Testometric Testing Machine with model name M500-250CT manufactured by a British company. Indurating Compressive Strength, Green and Dry compressive strength of the pellets were determined using the Testometric Testing Machine located at the laboratory of the Department of Metallurgical and Materials Engineering situated at the Nigerian Liquefied Natural Gas (NLNG) building.

INVERTED METALLURGICAL MICROSCOPE: The inverted Metallurgical Microscope with model name XJL-17 was used for the examination of the microstructures of the iron ore lump samples before and after the reducibility tests. The machine is also located at the laboratory of the Department of Metallurgical and Materials Engineering situated at the Nigerian Liquefied Natural Gas (NLNG) building.

III. METHODS

The Iron ores obtained consist of the chemical compositions given in Tables 1 to 2 The Itakpe iron ore concentrate has phosphorus in traces while the Agbaja Iron ore contain amount of phosphorus.

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EXPERIMENTAL PROCEDURE OF REDUCIBILITY

Reducibility is a summary of raw materials properties which determine the rate of conversion of iron oxides to metals by treatment with reluctant. A measure of reducibility is represented by a weight loss of an ore sample per time unit caused by the transition of oxygen into gas.

PROCEDURE FOR REDUCTION STUDIES

The produced pellets were used for these experiments, while a metallurgical coking coal obtained from the Ajaokuta Steel Company Limited was also used as reluctant. The collected iron ores lumps were crushed in 15-20 mm sizes. The collected Metallurgical coking coal was crushed to -5+ 15 size. The crushed iron ore lumps were dried in the laboratory dry oven with a view to eliminating moisture content that was present in the ores as they were subjected to a temperature 120oC.

The produced pellets were taken inside a stainless steel container (size: 6.1cm height × 5.9 cm inside diameter) with a mouth tightly closed by an airtight cover having an outlet for exit gas. Then the lumps and pellets were surrounded with coking coal, which serves as reducing agent in the experiments at various period and time.

The experimental procedures were strictly followed. The muffle furnace was used to heat up the samples to the required temperatures of 800oC, 840oC, 860oC, 880oC, 920oC, 960oC and 1000oC, at 8OC per minutes rate and the samples were all allowed to soak at various temperatures as indicated above by varying the soaking period in the range of 20 -120 minutes.

The experimental process was performed to determine the degree of reduction (%) at the stipulated temperatures. Each of the containers were properly labelled for specific experiments. The samples were brought out from the muffle furnace at the designated interval of 20 minutes up to 40 minutes residence, and the same process was performed for all the rest samples at an interval of 40 minutes up to 120 minutes of residence in the furnace. The containers containing the samples that were brought out from the muffle furnace were all allowed to cool at the room temperature in the normal air atmosphere and thereafter he weight losses analysis of the iron ore lumps and pellets were determined and recorded. The degrees of reduction pellets were calculated using this following formula.

$$\text{Degree of Reduction} = \frac{\text{Weight Loss}}{\text{Total weight of removable oxygen in Iron oxide}} \times 100\%$$

MICROSCOPE EXAMINATION

The Inverted Metallurgical Microscope was used to examine the iron ore lumps. Some important distinct phases were identified in the Itakpe ore by optical microscopy, the iron ore shows some grey like structures, some whitish mottled and blackish/whitish location. These structures and features were observed through the use of the inverted Metallurgical Microscope as shown in figure 3. The micrographs of the sample before and after the reducibility experiments were performed. The Abuja iron ore has some quality like texture with characteristic indicating that it contains pisolitic structure on the surface as indicated in figure 4. The iron ore shows a concentrated material in the pisolite nature while the matrix present consists of major impurities.

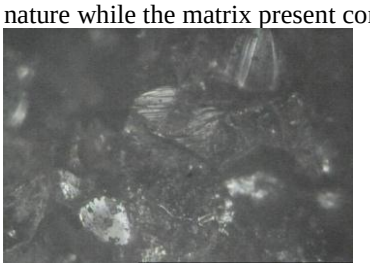

Figure 3: shows the microscope of the Itakpe Iron ore Before the reducibility experiment

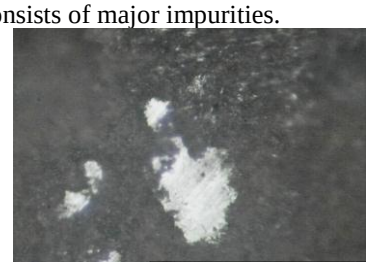

Figure 4: shows the microscope of the Agbaja Iron ore before the reducibility experiment

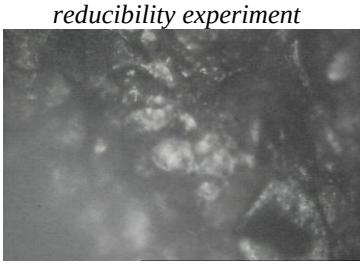

Figure 5: The Micrograph of Itakpe Iron Ore reduced @ 960^{o}C (Fully reduced)

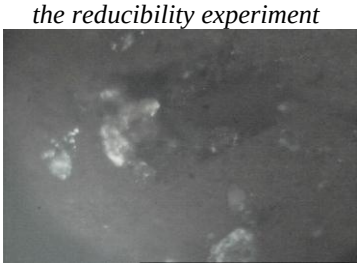

Figure 6: The Micrograph of Agbaja Iron ore reduced @ 960^{o}C (Fully reduced)

SAMPLE PREPARATION (PELLET PREPARATION)

15 kg of each iron ore of Itakpe and Agbaja were charged at different times into ball milling machine made by Bico Sprecher and Schn (2287) Industrial control, United State of America. Then one thousand six hundred (1600) balls of varying diameters ranging from 15 mm to 40 mm were charged into the ball mill (15 mm balls – 320 pieces, 20mm balls-320 pieces, 25mm balls-320 pieces, 30 mm balls -320 pieces and 40mm-320 pieces).

The samples were allowed to mill for six (6) hours after which they were discharged and sieved using 0.63mm sieve size. The oversize materials were recycled until they all passed through the 0.63mm sieve. At this point, the samples prepared were worked upon:

1.15 kg Itakpe iron ore pulverized to – 0.63 mm sieve size
2.15kg Agbaja iron ore pulverized to – 0.63mm sieve size.

The figures below show some processes of the prepared samples

1500g blended Iron ore was weighed with Itakpe iron ore in the blend-1425g. (95%) and Agbaja iron ore in the blend-75g.(5%) were weighed using Salter Digital weighing balance with trademark – Mettler Pm 2000. The weighed samples were charged into a clean and moisture-free Erich 2287 Palletizing disc machine of 35cm diameter wide palletizing disc. 4% lime was also added, while the Machine rotated at the speed of 25 rpm. The samples were properly mixed after which 1000 ml of water by volume was measured and added to the iron ore mix in the rotating pelletizing disc which works gradually; while the charge were been scrapped on a continuous basis to avoid sticking to the disc. As the experiment progresses the pellets of varying diameters ranging from 10mm to 20mm were formed. Rotation of the Pelletizing disc continued in a reduced speed of 15rpm; after satisfactory

formation of pellets impacted further strength on the pellets formed.

Figure 7: shows some produced pellets from Itakpe Iron ore

Figure 8: shows some produced pellets from Agbaja Iron ore

THERMOGRAVIMETRIC ANALYSIS (TGA)

THE THERMOGRAVIMETRIC ANALYSIS (TGA) OF ITAKPE IRON ORE

The line with blue colour runs on 30^{o}C and moved upwards until it got to the peak value of Deriv. weight (%C) of 0.0048. This value declined and raised until it achieved a stable value until it finally attained a value of 0.0007at the Deviation weight (%C). On the other hand, the line with light green colour indicates weight (%) versus temperature. The weight (%) started from 100 and continue to decline until it got to 1000^{o}C with corresponding value at 99.2. Figure 2.19 shows the Isothermal behaviour of the thermal decomposition of the Itakpe iron ore.

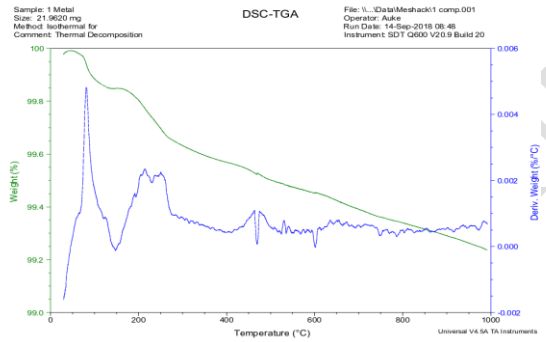

Figure.9: Show the Thermogravimetry Analysis (TGA) Performed on Itakpe Iron ore

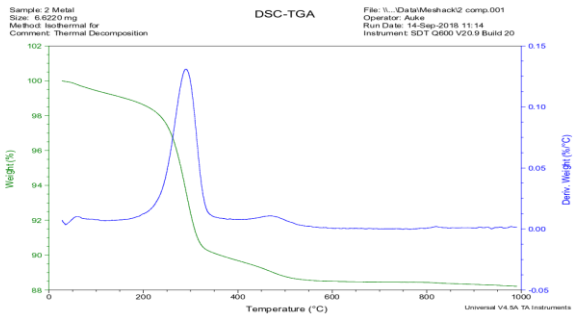

Figure 10: Show the Thermogravimetry Analysis (TGA) performed on Agbaja Iron ore

THERMOGRAVIMETRIC ANALYSIS (TGA) OF AGBAJA IRON ORE

The line blue colour runs on 30oC, got to 200oC and moved upwards it got to the peak value of Derivation weight (%C) at 0.13 with corresponding temperature value at 300oC. This value declined and continued at a steady, movement and dovetailed at 0.00 Derivation weight (%C). While the light green line started from 100 weight (%) and continue to decline and dovetailed at 88.2 (weight%) with corresponding value at 1000oC. Figure 2.20 shows the Isothermal behaviour of the thermal decomposition of the Agbaja iron ore.

SCANNING ELECTRON MICROSCOPE (SEM) OF ITAKPE AND AGBAJA IRON ORE

Examination by Scanning Electron Microscopy (SEM) /Energy-Dispersive Spectroscopy (EDS) shows that there are grey phase was quartz, the white phase hematite, and the mottled areas intergrowths of hematite and magnetite. Figure11 shows the Scanning Electron Microscopy there for showing the morphology while figure12 shows the Energy-Dispersive Analysis of the Intake Iron ore, where the elements are distributed with weight (%) concentration ranging from Fe, O Al, Si and C and also with their corresponding density values.

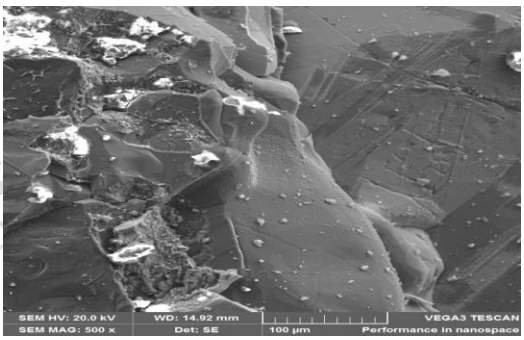

Figure 11: Shows the Scanning Electron Microscopy SEM of the Itakpe Iron ore at 100µm

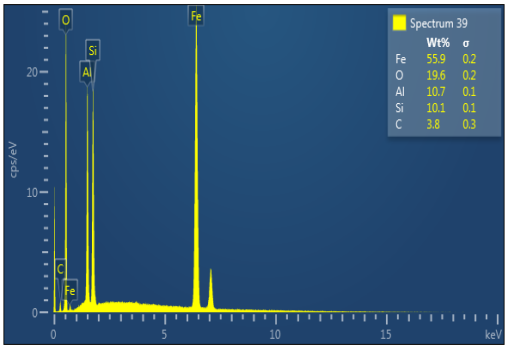

Figure 12: Shows Energy-Dispersive Analysis of the Itakpe Iron ore

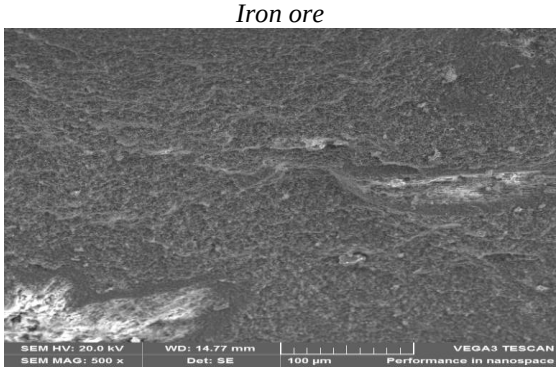

Figure 13: shows the Scanning Electron (SEM) of Microscopy the Agbaja Iron ore at 100µm

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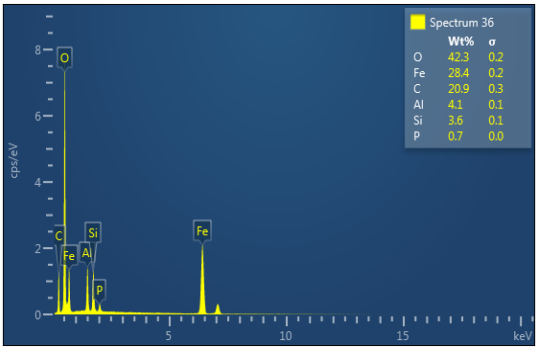

Figure 14: Shows Energy-Dispersive Spectroscopy of the Agbaja Iron ore

IV. RESULTS AND DISCUSSION

RESULTS

Table 4 and figures 15 and 16 show the degree of reduction (%) of produced pellets in Itakpe and Agbaja iron ore as a function of furnace holding time for the reduction of produced pellets of Itakpe iron ore at a temperature between 800°C – 1000°C

Temperature	Time (in min)	Reducibility Itakpe Iron Ore	Reducibility Agbaja Iron ore
800°C	20	36.8	36.8
	40	38.6	38.6
	60	49.5	49.5
	80	51.3	51.3
	100	52.6	52.6
	120	55	55.0
Temperature	Time(in min)	Reducibility	Reducibility
840°C	20	40.6	40.6
	40	44	44/0
	60	51	51/0
	80	52.9	52.9
	100	54.5	54.5
	120	56	56/0
Temperature	Time(in min)	Reducibility	Reducibility
880°C	20	41.4	41.4
	40	42.8	42.8
	60	52.5	52.5
	80	54.2	54.2
	100	56	56/0
	120	57.3	57.3
Temperature	Time(in min)	Reducibility	Reducibility
920°C	20	44.4	44.4
	40	45.8	45.8
	60	55	55.0
	80	56.7	56.7
	100	58.3	58.3
	120	64	64.0
Temperature	Time(in min)	Reducibility	Reducibility
960°C	20	49.6	49.6
	40	61.4	61.4
	60	68.5	68.5
	80	73.9	73.9
	100	75.2	75.2
	120	80.7	80.7
Temperature	Time(in min)	Reducibility	Reducibility
1000°C	20	57.9	57.9
	40	66.3	66.3
	60	71	71.0
	80	83.6	83.6
	100	94.4	94.4
	120	99.7	99.7

Table 4: Produced Pellets from Itakpe and Agbaja Iron Ore

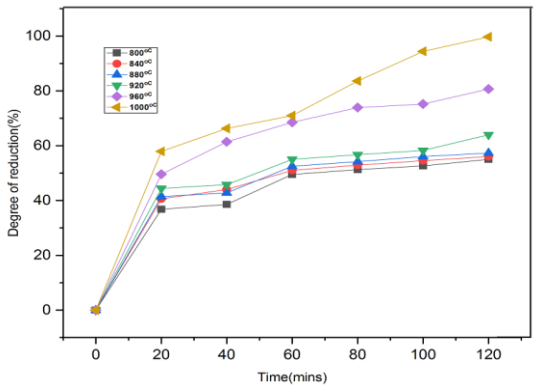

Figure 15: Reducibility of produced Pellets from Itakpe iron ore Versus Time (in mins) At temperature between 800°C to 1000°C

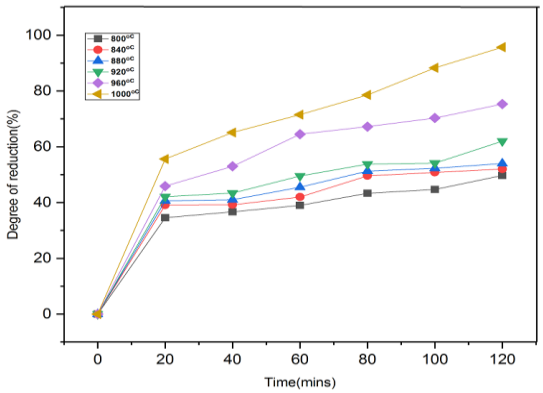

Figure 16: Reducibility (%) of produced pellets From Agbaja iron ore Versus Time (in mins) at temperature between 800°C to 1000°C

DISCUSSION

REDUCIBILITY STUDIES ON THE RESULTANT PELLETS

In this study, reducibility behaviour of pellets produced from the Itakpe and Agbaja iron ore fired in a muffle furnace.

The reducibility experiments were performed using the obtained metallurgical coking coal obtained from the Ajaokuta Steel Company Limited, Ajaokuta, Kogi State as reluctant. The pellets were subjected under an identical slow heating

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temperature where the heating rate was at $8^oC\ min^{-1}$ was maintained.

The samples were heated up to a temperature of various temperatures ranging from 800°C - 1000°C and allowed the samples to soak for a period ranging from 20minutes to 120minutes.

The corresponding value were collected and recorded and these values obtained were used to plot graphs as a function of furnace holding time for the reducibility at temperature between 800°C – 1000°C.

The results obtained from the experiments showed that there were higher degree of reduction (%) to the corresponding produced fired pellets. It was noticeably observed that they have porosity values in the produced fired pellets.

The produced fired pellets showed higher level of reducibility. It was noticeably observed that they produced pellets of the Itakpe iron ore was still intact at 920°C for 100mins while the Agbaja iron ore pellets got scattered at the temperature and time. These observations are shown in figures 17a and 17b respectively. Similar trend was observed as from figures 18a- and 18b. It was also observed that both produced pellets scattered as shown in figures 17a and 17b at the same temperature of 920°C for 120 minutes. But at a temperature of 960°C for the period of 120minutes it was discovered that the pellet produced from the Itakpe iron ore did not scatter, but that of Agbaja iron ore scattered these observations are indicated on figures 18 a and 18b respectively. The same trend also occurred where both pellets were subjected to a temperature of 1000°C for 120mins and got scattered as the observations were also indicated in figures 19a and 19b respectively.

Figure 17 a: Pellet Itakpe @ 920°C for 120 mins

Figure 17 b: Pellet Agbaja @ 920°C for 120 mins

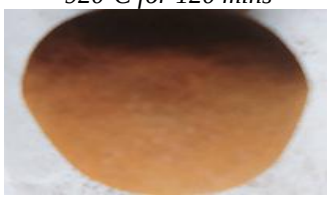

Figure 18a: Pellet Itakpe @ 960°C for120 mins

Figure 18b: Pellet Agbaja @ 960°C for120 mins

Figure 19 a: Pellet Itakpe @ 1000°C for 120 mins

Figure 19b: pellets @ @ 1000°C for 120mins

EFFECT OF TIME ON THE DEGREE OF REDUCTION

The research work carried out shows that the rapid heating of the pellets shows that the heating time has an approximately significant effect on the reduction behaviour on pellets. As shown in table 4 on produced pellets from Itakpe and Agbaja iron ore have the highest degree of reduction(%) with increasing reduction time up to the range studied (i.e. 120 minutes).

It was also observed that all the fired pellets were almost completely reduced (more than 90% reduction) in about 120 minutes. This indicates that the utilization of these pellets in sponge iron making is likely to allow usage in the Blast Furnace and the Direct Reduced Iron.

For effectiveness and efficiency of the furnaces, these results, therefore, will translate to a greater amount of energy savings and will extend the lifespan of the furnaces. The excessively high degree of reduction in the first 40 minutes was mainly associated with the release of volatiles from the metallurgical coking coal used due to their reformation into H_2, CO, etc.

The major participation of these reducing gases in the reduction of iron oxide (i.e. an appreciable presence of H_2 and CO in the reduction chamber gives a boost in the reduction rate). The decrease in reduction rate with increasing time above 60 minutes was undoubtedly due to the combined effects of an increase in product metallic layer thickness and diminished evolution of volatile matter from the coal. An increase in the thickness of the product iron layer offers greater resistance in the diffusion of carbon and reducing gas to the surface of unreduced iron oxide.

EFFECT OF HEATING MODE ON THE REDUCIBILITY ON THE PELLETS

In this research work, the effect of heating mode on the samples were studied with a view to determining its effects on the reducibility on the produced pellets. the pellets were reduced using metallurgical coking coal at a temperature range between 800°C - 1000°C (the soaking times was varied from 20minutes -120minutes at an interval of 20mins), these experiments were performed under rapid and slow heating conditions with the use of muffle furnace.

It is fairly clear that in comparison to rapid heating, the slow heating to reduction temperature gives appreciably higher degree of reduction. It is more likely that the rapid heating from 920°C to 1000°C causes a higher rate of volatile matter escaping from the metallurgical coking coal, thereby providing less time for H_2 and CO (reducing gases) to be in contact with the pellets.

The results in this work thus indicate that there was a higher degree of reduction in rapid heating in the pellets during lower heating operation; the volatile matters were released from coal at a slower rate. The more deposition of highly reactive pyrolytic carbon, and increased time of contact of carbon and reducing gases (H_2 and CO) with the pellets appearing to be the obvious reasons for the higher degree of reduction (%).

Heating of hematite pellets from room temperature to the required reduction temperature (1000°C) in reducing

atmosphere, to some extent, is also responsible' for higher degree of reduction(%) under slow heating condition.

SCANNING ELECTRON MICROSCOPY AND EDS

- ✓ Resultant Pellets from Itakpe Iron ore @800^{o}C 120 minutes after the reducibility Test: Examination by Scanning Electron Microscopy (SEM) showing the morphology of the ore structure at 50µpm and the distribution of Fe content and other elements in the iron ore.

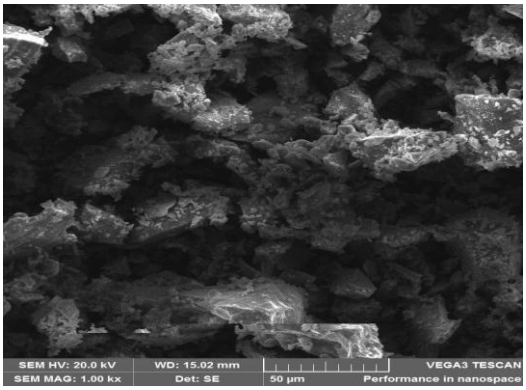

Figure 20(a): show the examination of Pellets Itakpe Iron ore @ 800^{o}C pellets from iron

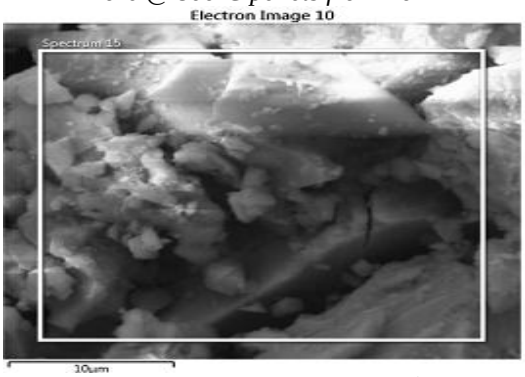

Figure 20(b): show electron Image 10 800^{o}C by using the Scanning Electron

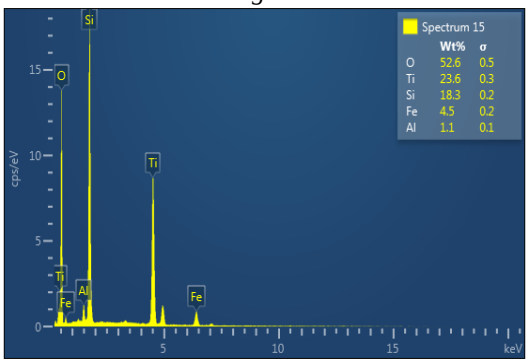

Figure 20 (c): shows the EDS ore @800^{o}Cafter reducibility Test

- ✓ Produced Pellets from Agbaja Iron ore @800^{o}C 120 minutes after the reducibility Test: Examination by Scanning Electron Microscopy (SEM) showing the morphology of the ore structure at 100µpm and the distribution of Fe content and other elements in the iron ore.

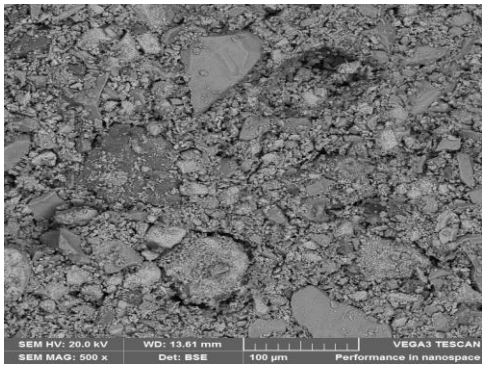

Figure 22(a): show the examination of Produced Pellets Itakpe Iron ore @ 920^{o}C by using the Scanning Electron Microscopy (SEM)

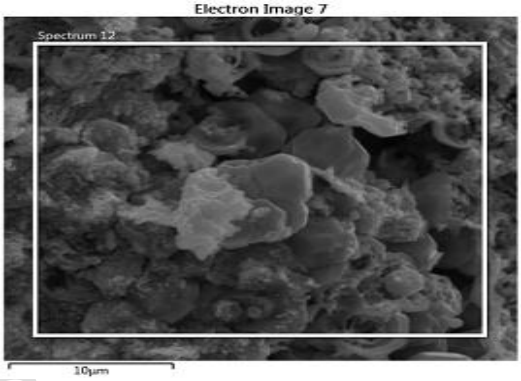

Figure 22 (b): show electron Image 7 @ 920^{o}C

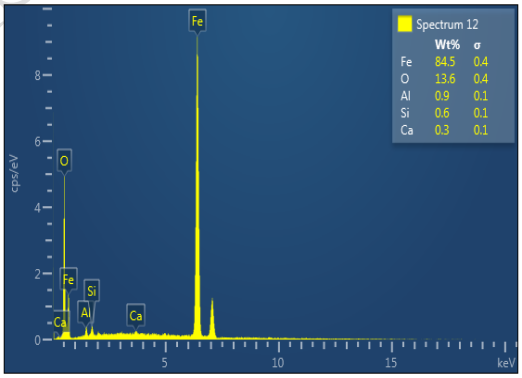

Figure 22(c): shows the EDS where pellets from iron ore @920^{o}C after Reducibility test

- ✓ Produced Pellets from Itakpe Iron ore @920^{o}C for 120mins after the reducibility Test: Examination by Scanning Electron Microscopy (SEM) showing the morphology of the ore structure at 100µpm

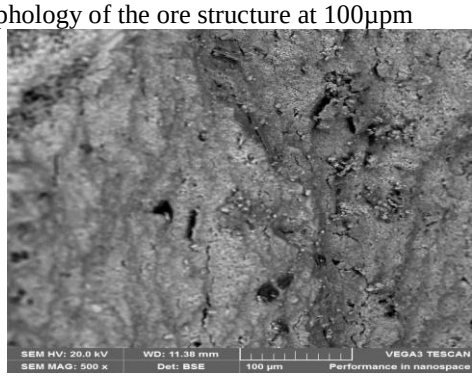

Figure 23 (a): show the examination of Pellets Electron Microscopy (SEM) reducibility test Pellets from iron ore after @ 920^{o}C

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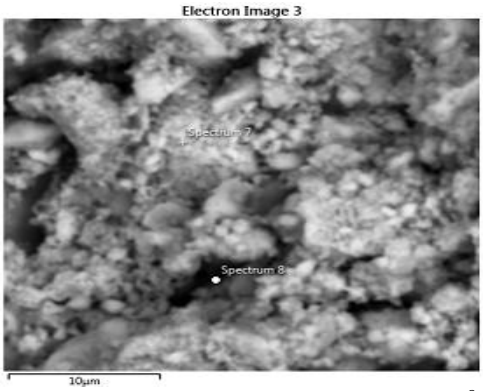

Figure 23 (b): show electron Image 8 @920°C

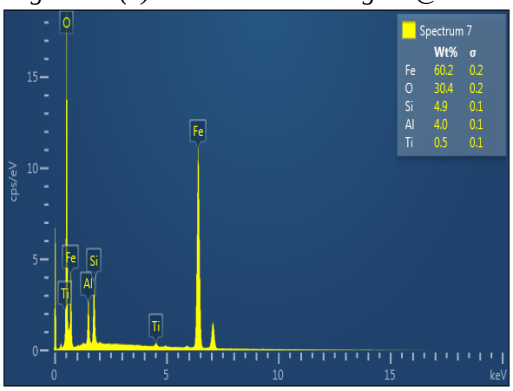

Figure 23(c): shows the EDS where Resultant pellets @ 920°C by using the Scanning

- ✓ Produced Pellets from Itakpe Iron ore @1000°C for 120minutes after the reducibility Test: Examination by Scanning Electron Microscopy (SEM) showing the morphology of the ore structure at 100µm

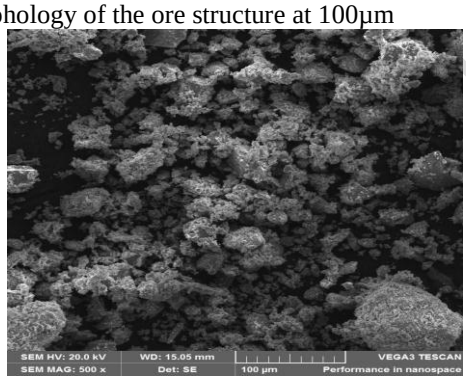

Figure 24(a): show the examination of Pellets from Itakpe Iron ore @ 1000°C after reducibility test

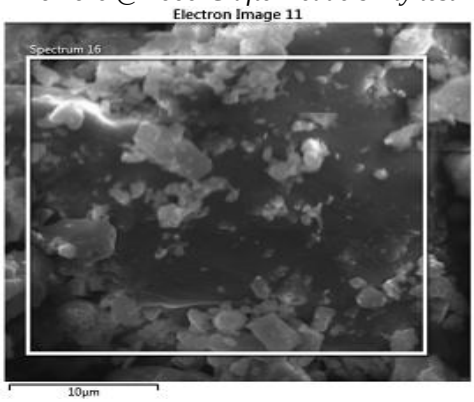

Figure 24(b): show electron pellets from iron Image 11 @ 1000°C

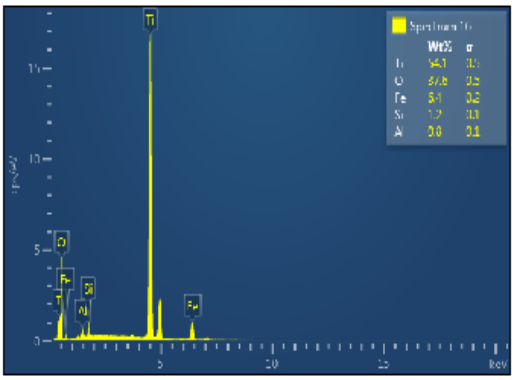

Figure 24(c): shows the EDS where ore @1000°C after the reducibility

- ✓ Resultant Pellets from Agbaja Iron ore @ 1000°C for 120 minutes after the reducibility Test: Examination by Scanning Electron Microscopy (SEM) showing the morphology of the ore structure at 100µm

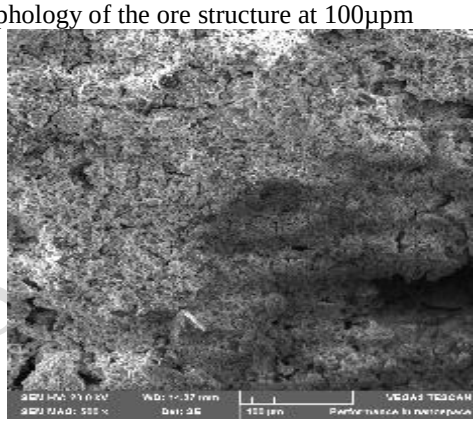

Figure 25(a): show the examination of Resultant Pellets from Agbaja Iron ore @1000°C

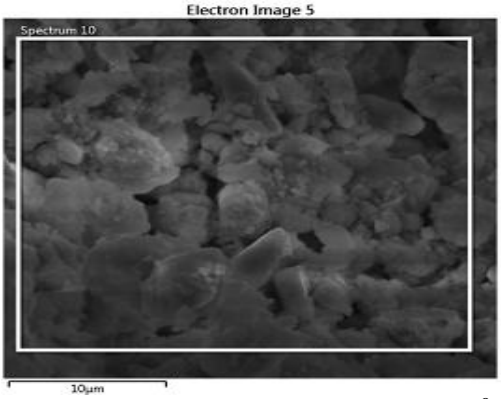

Figure 25(b): show electron Image 5 @1000°C

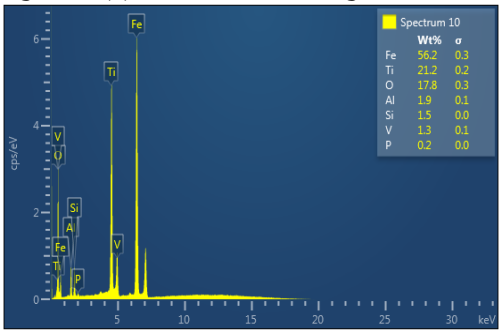

Figure 25 (c): shows the EDS where pellets from iron ore @ 1000°C after the reducibility test

ANALYSIS OF RESULTS AFTER THE REDUCIBILITY TEST (SEM AND EDS)

S/NO	Sample Name	Temperature In degree	Fe (Wt %)
1	Itakpe Pellets	800^{oC}	4.5
2	Itakpe Pellets	920 ^{oC}	84.5
3	Itakpe Pellets	1000 ^{oC}	6.4
1	Agbaja Pellets	800 ^{oC}	40.7
2	Agbaja Pellets	920 ^{oC}	60.2
3	Agbaja Pellet	1000 ^{oC}	56.2

Table: 5 Analysis of Results of pellets After Performing the Reducibility Tests using SEM and EDS Resultant Pellets

The results obtained on table 5 indicate the SEM and EDS analysis of Resultant Pellets from Itakpe and Agbaja iron ore. It is noted that the value of Fe Content was 4.5wt% when the sample was subjected to a temperature of 800^{o}C for 120minutes. While the Fe content roses astronomically from 4.5wt% to 84.5 wt% when the sample was subjected to a temperature range of 920^{o}C for a period of 120minutes and then decreased from 84.5wt% of Fe content to a practical value of 6.4 wt%. By implication, it was observed that the reducibility of the samples at various temperature was put at these temperatures of 800^{o}C to 920^{o}C to 1000^{o}C. This is shown in 5 . Similarly, the same trend was obtained for the resultant pellets from Agbaja Iron Ore. It was observed that at 800^{o}C for 120minutes, Fe Content value was obtained as 40.7 wt.%. A similar trend was also noticed for sample decreased at 920^{o}C for 120minutes with a rise from 40.7 wt% to 60.2 wt% following a similar trend as obtained by the Itakpe Iron ore and the Fe content value obtained at 1000^{o}C for 120 minutes also decreased from 60.2 wt% to 56.2 Wt% of Fe content. By implication, it could stated that the pellets produced from Agbaja iron ore fully reduced at 1000^{o}C for 120minutes as indicated on the table.

Figure 3.12 shows the behaviour of the graph which indicates a starting value of Fe Content wt% at 4.5wt% to 84.5wt% to 6.4wt% and also in figure 3.13 similar trend was observed the value of the Fe content started from 40.7wt% rose to 60.2 wt% and reduced to 56.2 wt%.

Finally, figure 3.14 shows the behaviour of the values of the resultant pellets graphs plotted.

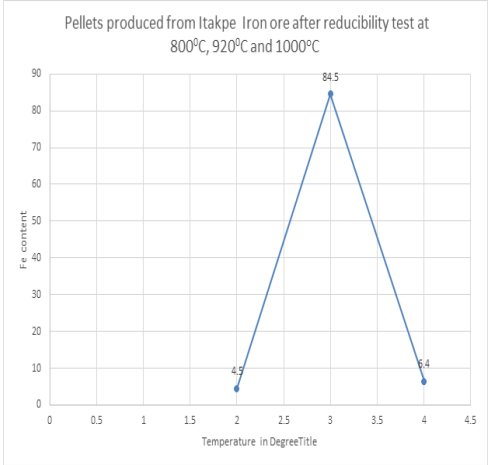

Figure 26: shows the Fe Content of Itakpe Iron Ore after the reducibility test @ 800^{o}C, 920^{o}C and 1000^{o}C using SEM and EDS

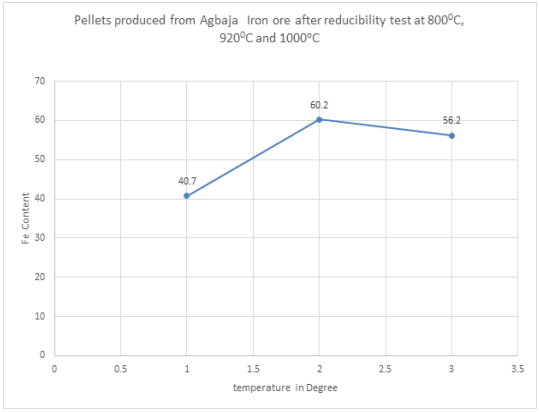

Figure 27: shows the Fe content od Agbaja Iron Ore after the reducibility test @ 800^{o}C, 920^{o}C and 1000^{o}C

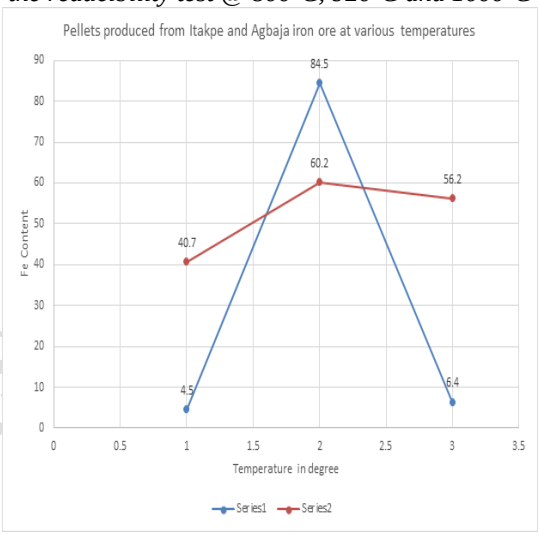

Figure 28: shows the Fe content of Itakpe Iron Ore VS Agbaja Iron Ore after the reducibility test @ 800^{o}C, 920^{o}C and 1000^{o}C

V. CONCLUSIONS

The reducibility study on the was intensively investigated the studies carried out to reveal the followings:

The time of reduction and temperature of the produced pellets indicates that there was a greater influence on the reducibility. It was observed that the reducibility increased with increase in reduction from 800^{o}C -1000^{o}C. The period of time for the degree of reduction was performed within the time range of 20 -120minutes as these processes indicates that the reducibility was high in the produced pellets. The reduction behaviour of the produced pellets are identical in the studies. The use of the Metallurgical coking coal as reluctant had great effects on the produced pellets as there were significant influences on the reducibility on the tested samples.

The produced pellet show reducibility tendency. The pellets shows the tendency as there were increases in the reducibility. The summary of the research work could be stated thus: that the Itakpe pellets reduced at 800^{oC} has a Fe Content of 4.5wt% while at the same temperature the Agbaja pellets has a Fe content value of 40.7wt%. When the produced pellets were subjected to a temperature of 920^{o}C the Fe content was 84.5wt% and that of Agbaja pellets was 40.7wt%

and at 1000^{o}C the Fe Content value obtained was 6.4 wt% for Itakpe Iron ore while Fe content obtained for Agbaja iron ore Fe Content was put at 56.2wt%. By this, it is noted that the pellets produced from Itakpe Iron ore was more reduced than that of Agbaja iron ore.

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