

COMPARATIVE STUDY ON THE REDUCIBILITY AND SWELLING OF ITAKPE AND AGBAJA IRON ORE AND THE RESULTANT PELLETS

Ocheri C.*¹, C. N. Mbah², Adams S. M.³ and Alabi O. O.⁴

¹Dept. of Metallurgical and Materials Engineering, University of Nigeria, Nsukka.

²Dept. of Metallurgical and Materials Engineering, Enugu State University of Science and Technology.

³Dept. of Metallurgical and Materials Engineering, University of Nigeria, Nsukka.

⁴Dept. of Metallurgical and Materials Engineering, Federal University of Technology, Akure.

Article Received on 08/03/2020

Article Revised on 29/03/2020

Article Accepted on 19/04/2020

*Corresponding Author

Ocheri C.

Dept. of Metallurgical and Materials Engineering,
University of Nigeria,
Nsukka.

ABSTRACT

The comparative study on the reducibility and swelling of Itakpe and Agbaja Iron Ore and the resultant pellets were performed. The chemical and morphology of the samples were examined. Thermogravimetry (TG), Differential Thermal Analyzer (DTA), XRF, Optical and Electron Microscopy and Scanning Electron Microscopy (SEM) with Energy Dispersive Spectroscopy (EDS). The reducibility and swelling studies were performed in using a muffle furnace. The resultant pellets and the iron ore lumps were taken inside the stainless steel container with diameter 60.75 mm x 59.40 mm inside diameter with a mouth tightly closed by an air tight cover having an out let for exit gas, The samples were surrounded with metallurgical coking coal obtained from the Ajaokuta Steel Company Limited. The highest reducibility value of the resultant pellet for the Itakpe iron ore value was 99.7% with corresponding temperature at 1000°C and time rate of 120mins, while the highest reducibility value of the resultant pellets for the Agbaja iron ore was obtained at 95.7% with corresponding temperature at 1000°C and time rate of 120mins. Similarly, the highest reducibility value of the Itakpe iron ore lump was obtained at 74.33% with corresponding temperature at 1000°C and time rate of 120mins. The highest reducibility value of the Agbja iron ore lumps obtained at 33.00 % with corresponding temperature at 1000°C and time rate

of 120mins..Smiliarly the highest swelling value obtained was 17.2 % for resultant pellets of Itakpe with corresponding value at 1000°C and time rate of 120mins. The highest swelling % of resultant pellets for Agbaja iron ore was as 24.4 with corresponding temperature at 1000°C at time rate of 120mins, while the highest swelling % value of Itakpe iron ore lump was obtained at 15.4 with corresponding temperature at 1000°C and time rate of 120mins. Finally, the highest swelling % value of the Agbaja iron ore lump was obtained aa 4.41 with corresponding temperature at 1000°C and time rate of 120mins.

KEYWORDS: Comparative, Study, Reducibility, Swelling, Iron Ore, Resultant Pellets.

1.1 INTRODUCTION

Several iron ore deposits discovered in Nigeria since 1904. 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 defense and provide employment and revenue generation (Adeleye R.A., 1964).

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 a time unit. The weight loss caused by the charge oxygen transformation into gas.

The reducibility has a lot of influence on the blast furnace operation is highly important; it can be used for a fuel consumption determination and a selection of proper lumpiness. The reducibility value changes in particular by hot treatment (sintering). The charge porosity influences the Reducibility, too. The higher porosity of the charge, the larger reaction surface and the faster gas reduction. .The ISO - R ISO950 standard is the main parameter when considering the reduction velocity v_{40} . The velocity of oxygen degradation in percentage $\cdot\text{min}^{-1}$ at 40% reduction degree are determined.

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

The determination of degree of swelling of pellets and iron ore lumps - Swelling of the pellets and iron ore lumps samples were defined as the volumetric expansion of pellets and iron ore

lumps when they are reduced at high temperatures. Initial and final diameter of the pellets and iron ore lumps were taken before and after swelling experiments with the use of a digital Vernier, callipers and the degree of swelling of the lumps and the pellets were calculated using the formula:

$$\text{Degree of Swelling} = \frac{(\text{Final volume} - \text{initial volume})}{\text{Initial volume}} \times 100\%$$

Swelling usually less than 25% is acceptable but above this value, it is considered catastrophic and is thus not desired.

2.0 RESEARCH METHODOLOGY

2.1 Materials

2.1.1 Itakpe Iron Ore

The topography of the region is a plateau rising gently to the north –east 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. (Adeleye R.A, 1964). 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 tonnes (Adeleye R.A, 1964). The deposit has an average iron ore content of 36%. This has to be beneficiated at 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 Iron making. 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.



Figure 2.1: Show the sourced Itakpe Iron ore (NIOMCO).

2.1.2 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 (BRG Report, 1983). It cannot be used directly in the Blast Furnace process or other reduction process without further treatment e.g. pelletization or briquetting. The ore is a lean ore and sedimentary origin (BRG Report, 1983). 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 oolitic in nature, limonite that occur in mammillated or stalactite forms having fibrous structure resembling hematite (BRG Report, 1983). The Agbaja Iron ore are made of brown compacted fine-grained materials, which consist of extremely larger particles, which show the tendency to be friable. Agbaja iron ore is strongly magnetic (Adedeji F.A. et al 1984). The Agbaja iron ore sample is known to be compacted ground fine particles, which significantly exhibits the characteristics of being friable and magnetically strong. The picture below shows the iron ore as being sourced at the Agbaja plateau in Kogi State.



Figure: 2.2: Shows the sourced Agbaja iron ore (NMDC).

2.1.3 Chemical Analysis of Itakpe and Agbaja Iron ore

Table 2.1 and 2.2 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 South Africa.

Table 2.1: Show the Chemical compositions of Itakpe Iron Ore (XRF Method).

Component	Unit	Result
Na ₂ O	mass%	0.354
MgO	mass%	0.385
Al ₂ O ₃	mass%	12.21
SiO ₂	mass%	20.53
P ₂ O ₅	mass%	1.556
SO ₃	mass%	0.138
K ₂ O	mass%	0.614
CaO	mass%	0.11
TiO ₂	mass%	1.398
V ₂ O ₅	mass%	0.085
Cr ₂ O ₃	mass%	0.064
MnO	mass%	0.164
Fe ₂ O ₃	mass%	52.54
NiO	mass%	0.025
CuO	mass%	0.033
ZnO	mass%	0.02
Rb ₂ O	mass%	0.013
SrO	mass%	0.014
ZrO ₂	mass%	0.052
BaO	mass%	0.202

Table 2.2: Shows the chemical composition of Agbaja Iron Ore (XRF Method).

Component	Unit	Result
Na ₂ O	mass%	0.118
MgO	mass%	0.246
Al ₂ O ₃	mass%	6.047
SiO ₂	mass%	25.23
P ₂ O ₅	mass%	0.023
SO ₃	mass%	0.19
Cl	mass%	0.096
K ₂ O	mass%	0.429
CaO	mass%	0.428
TiO ₂	mass%	0.851
Cr ₂ O ₃	mass%	0.09
MnO	mass%	0.498
Fe ₂ O ₃	mass%	43.76
NiO	mass%	0.053
CuO	mass%	0.011
ZnO	mass%	0.056
ZrO ₂	mass%	0.021
P ₂ O ₅	mass% trace	trace

Table 2.4: Identified Patterns List: Agbaja Iron Ore.

Visible	Ref. Code	Score	Compound Name	Displacement [°2Th.]	Scale Factor	Chemical Formula
*	03-0863	40	Magnetite	0	0.57	Fe ₃ O ₄
*	76-2385	35	Sodium Aluminum Silicate	0	0.354	Na ₆ Al ₄ Si ₄ O ₁₇
*	86-0550	28	Hematite, syn	0	0.129	Fe ₂ O ₃
*	48-0652	9	Aluminum Phosphate	0	0.099	Al P O ₄

Soucre from the XRD Analysis

2.1.6 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 proximately 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:

Table 2.5: Chemical Analysis of Metallurgical Coking Coal.

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



Figure 2: 5 Shows the Metallurgical Coking Coal after being used as reducing agent.



Figure 2. 4 shows the Metallurgical coking coal Coal before they were used as reducing agent.

2.2. Equipment and Methods

The following equipment were used for the preparing 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 and Laboratories 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 South Africa.

- (a) Ball Milling Machine:** The samples were crushed and ground at the National Metallurgical Development Centre, (NMDC) Jos. 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.
- (b) Pelletizing Machine:** Pellets were resultant with the use of the Pelletizing Machine with 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.
- (c) 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 resultant pellets at the laboratory of the Department of Metallurgical and Materials Engineering, University of Nigeria, Nsukka.
- (d) Sample handler:** The resultant pellets were carefully handled with a view to handling them properly without breakage

(e) **Muffle furnace:** A Muffle furnace was used to heat up the resultant pellets and the iron ores lumps. The furnace was also used for reducibility and swelling tests on both the iron ore lumps and the resultant pellets/

2.3 Methods

The Iron ores obtained consist of the chemical compositions given in Tables 2.1 to 2.2. The Itakpe iron ore concentrate has phosphorus in traces while the Agbaja Iron ore contain amount of phosphorus.

2.4 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 transition of oxygen into gas.

2.4.1 Procedure for Reduction Studies

- ❖ The resultant pellets and Iron ore Lumps 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 chemical compositions of the Metallurgical Coking coal is shown in table 2.5
- ❖ 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 120°C.
- ❖ The crushed lumps and resultant pellets were taken inside six stainless steel containers (size: 60.75mm height × 59.40mm inside diameter) with a mouth tightly closed by air tight cover having an out let 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 crushed iron ore lumps were properly placed at the centre of the solid packed bed. This was done with a view to ensuring that the lumps and pellets inside the packed were surrounded with coking coals
- ❖ The experimental procedures were strictly followed .The muffle furnace was used to heat up both the samples (lumps and resultant pellets) to the required temperatures of 800°C, 840°C, 860°C, 880°C, 920°C, 960°C and 1000°C ,at 8⁰C per minutes rate. The

samples were all allowed to soak at various temperatures by varying the soaking period in the range of 20 -120 minutes.

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

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

Determination of degree of swelling of pellets and iron ore lumps - Swelling of the pellets and iron ore lumps samples were defined as the volumetric expansion of pellets and iron ore lumps when they are reduced at high temperatures. Initial and final diameter of the pellets and iron ore lumps were taken before and after swelling experiments with the use of a digital Vernier, callipers and the degree of swelling of the lumps and the pellets were calculated using the formula:

$$\text{Degree of Swelling} = \frac{(\text{Final volume} - \text{initial volume})}{\text{Initial volume}} \times 100\%$$

Swelling usually less than 25% is acceptable but above this value, it is considered catastrophic and is thus not desired.

3.0 Resultant Pellets from Blends of Itakpe / Agbaja Iron Ore

The experimental materials used for the studies, the iron ore lumps were obtained from the mine site in Itakpe a town close to Okene. The Agbaja iron ore was obtained from the Agbaja plateau in Oworo both in Kogi State Nigeria. The chemical compositions of the ores were determined using the XRF and the morphology was determined using XRD, The iron ores lumps sourced locally were properly investigated, which are readily available in Nigeria and the nature of the two ores were determined.

3.1 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 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.



Figure 2.7: Shows the crushed Itakpe Iron ore prior to pulverization.



Figure 2.8: Shows the crushed Agbaja Iron ore prior to pulverization.

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 trade mark – 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 mls of water by volume was measured and added to the iron ore mix in the rotating pelletizing disc which work gradually; while the charges were been scrapped on a continuous basis to avoid sticking to the disc. As the experiments

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 3.1: Shows the pulverized Itakpe iron ore.



Figure 3.2: Shows the pulverized Agbaja Iron ore.



Figure 3.3: Show the bentonite used as binding agent of the ores.



Figure 3.4: Shows some resultant pellets from Itakpe Iron ore,



Figure 3.5: Shows some resultant pellets from Agbaja Iron ore.



Figure 3.6: Shows how some resultant pellets for both Itakpe and Agbaja iron ores were dried in the normal atmospheric which is known as green pellets

3.2 Thermo gravimetric Analysis (TGA)

3.2.1 The Thermo gravimetric Analysis (TGA) of Itakpe Iron Ore

The line with blue colour runs on 30°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 0.0007 at the Deriv weight (%C). On the other hand, the line with light green colour indicate weight (%) versus temperature. The weight (%) started from 100 and continue to decline until it got to 1000°C with corresponding value at 99.2. Figure 3.5 shows the Isothermal behaviour of the thermal decomposition of the Itakpe iron ore.

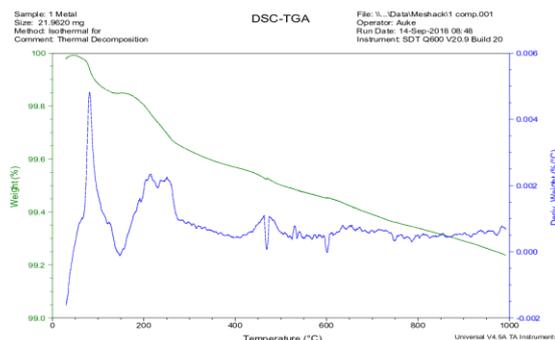


Figure.3.5: Show the Thermogravimetry Analysis (TGA) performed on Itakpe Iron ore.

3.2.2 Thermogravimetric Analysis (TGA) of Agbaja Iron Ore

The line blue colour runs on 30°C got to 200°C and moved upwards it got to the peak value of Deriv.weight (%C) at 0.13 with corresponding temperature value at 300°C. This value declined and continued at a steady , movement and dovetailed at 0.00 Deriv 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 1000°C . Figure 3.6 shows the Isothermal behaviour of the thermal decomposition of the Agbaja iron ore.

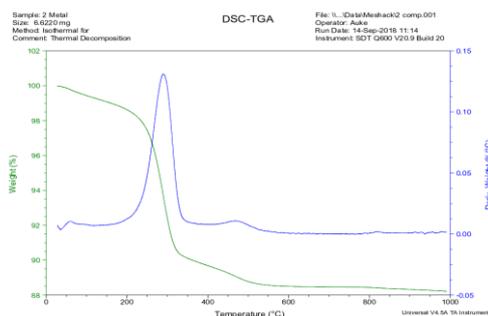


Figure 3.6: Show the Thermogravimetry Analysis (TGA) performed on Agbaja Iron ore.

3.2.3 Scanning Electron Microscope (SEM)

3.2.3.1 Scanning Electron Microscope (SEM) of Itakpe 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.

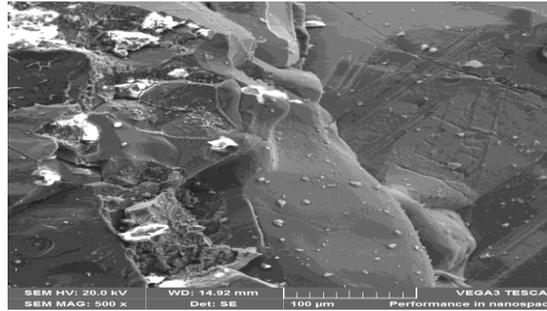


Figure 3.7: Shows the Scanning Electron Microscopy SEM of the Itakpe Iron ore at 100µm.

3.2.4 Electron Image of the spectrum location 37, 38 and 39 at 10µm Itakpe Iron Ore

The figure 3.8 shows the three-spectrum location that were taken in order to determine the concentration of ore. The locations of the spectrum are 57 38 and 39 at 10µm.

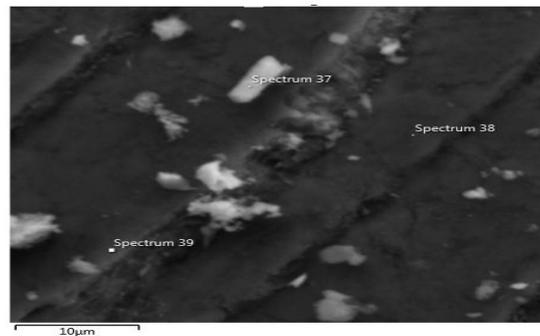


Figure 3.8: Shows Electron Image of the spectrum location 37, 38 and 39 at 10µm.

2.2.3 Energy-Dispersive Spectroscopy (EDS) of Itakpe Iron Ore

Figure 3.9-show spectrum 39 where the concentration of the elements as distributed with weight (%) concentration ranging from Fe, O Al, Si and C and with their corresponding density values.

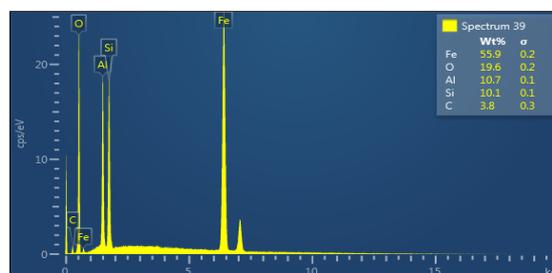


Figure 3.10: Shows Energy-Dispersive Analysis of the Itakpe Iron ore.

3.2.4. Scanning Electron Microscope (SEM) of Agbaja Iron Ore

Examination by Scanning Electron Microscopy (SEM) /Energy-Dispersive Spectroscopy (EDS) shows that there are fine and whitish structures.

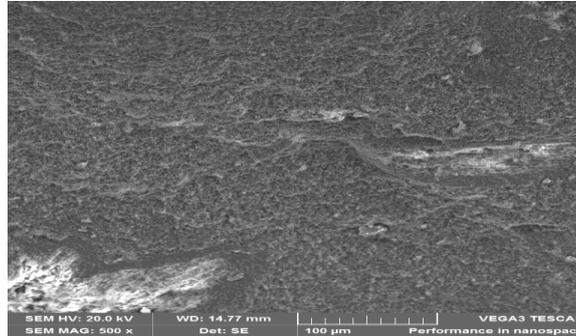


Figure 3.11: Shows the Scanning Electron Microscopy (SEM) of the Agbaja Iron ore at 100µm

3.2.5 Electron Image of the spectrum location 36 at 10µm Agbaja Iron Ore

The figure 3.12 shows the three-spectrum location that were taken in order to determine the concentration of ore...

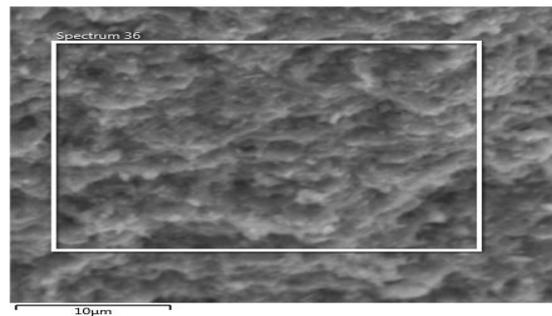


Figure 3.12: Shows Electron Image 12 of the spectrum location 36 at 10µm.

3.2.6 Energy-Dispersive Spectroscopy (EDS) of Agbaja Iron Ore

Figure 3.13-show spectrum 39 where the concentration of the elements as distributed with weight (%) concentration ranging from O, Fe, C, Al, Si and P and with their corresponding density values.

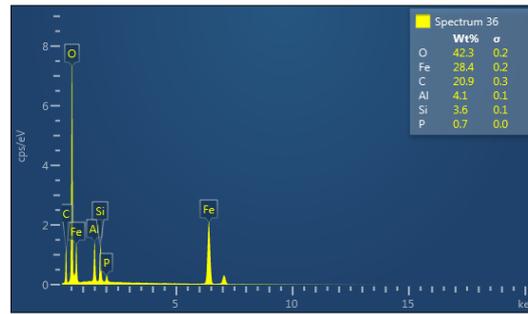


Figure 3.13: Shows Energy-Dispersive Spectroscopy of the Agbaja Iron ore.

4.1 RESULTS AND DISCUSSION

4.1.1 RESULTS

Table 4.1 and figures 4.1 and 4.2 show the degree of reduction(%) of resultant pellets (Itakpe Iron ore and degree of reduction (%) of Agbaja iron ore). While figures 4.3 and 4.4 show the degree of swelling(%) of produced pellets of Itakpe and Agbaja iron ore as functions of furnace holding time for the degree of Reduction(%) and swelling(%) of resultant pellets of Itakpe iron ore at temperature between 800°C – 1000°C.

Table 4.1: Resultant Pellets from Itakpe and Agbaja Iron Ore.

Temperature	Time (in min)	Degree of Reduction (%) Itakpe iron ore	Degree of Reduction ((%) Agbaja iron ore	Degree of Swelling (%) of Itakpe iron ore	Degree Swelling(%) Agbaja iron ore
800°C	20	36.8	34.6	0.2	0.4
	40	38.6	36.7	1	2.1
	60	49.5	39	2.8	3.8
	80	51.3	43.3	4.5	5.5
	100	52.6	44.7	6.8	7.8
	120	55	49.7	9.6	10.6
Temperature	Time (in min)	Degree of Reduction (%)	Reduction index in percentage	Degree of Swelling (%)	Swelling index in percentage
840°C	20	40.6	39.1	0.5	1.2
	40	44	39.2	1.2	2.9
	60	51	42	3.1	4.6
	80	52.9	49.6	7.8	9.7
	100	54.5	50.8	9.8	11.5
	120	56	52	10.5	13.7
Temperature	Time (in min)	Degree of Reduction (%)	Reduction index in percentage	Degree of Swelling (%)	Swelling index in percentage
880°C	20	41.4	40.6	0.9	1.9
	40	42.8	41	1.6	3.6
	60	52.5	45.5	3.6	6.6

	80	54.2	51.3	9.1	10.9
	100	56	52.3	10.7	13.7
	120	57.3	54	11.6	15.6
Temperature	Time (in min)	Degree of Reduction (%)	Reduction index in percentage	Degree of Swelling (%)	Swelling index in percentage
920°C	20	44.4	42.1	1.2	3.2
	40	45.8	43.4	2.2	6.2
	60	55	49.5	4.1	9.1
	80	56.7	53.8	9.9	14.9
	100	58.3	54.1	12.1	16.1
	120	64	62	13.4	19.4
Temperature	Time (in min)	Degree of Reduction (%)	Reduction index in percentage	Degree of Swelling (%)	Swelling index in percentage
960°C	20	49.6	45.9	1.6	3.6
	40	61.4	53	2.8	6.8
	60	68.5	64.5	4.7	10.7
	80	73.9	67.2	10.5	16.5
	100	75.2	70.3	13.7	19.7
	120	80.7	75.3	14.4	21.4
Temperature	Time (in min)	Degree of Reduction (%)	Reduction index in percentage	Degree of Swelling (%)	Swelling index in percentage
1000°C	20	57.9	55.6	1.8	4.8
	40	66.3	65.1	3	8
	60	71	71.5	12.9	12.9
	80	83.6	78.6	14.4	19.4
	100	94.4	88.3	16.6	22.6
	120	99.7	95.7	17.2	24.2

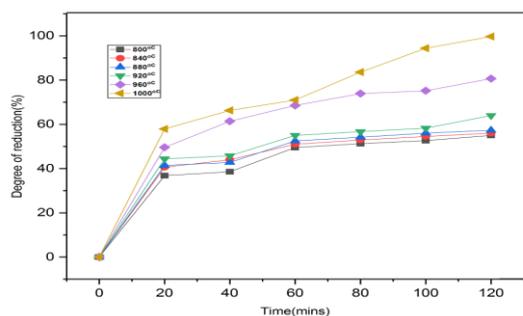


Figure 4. 1: Degree of Reduction (%) of resultant pellets from Itakpe iron ore Versus Time (in mins)

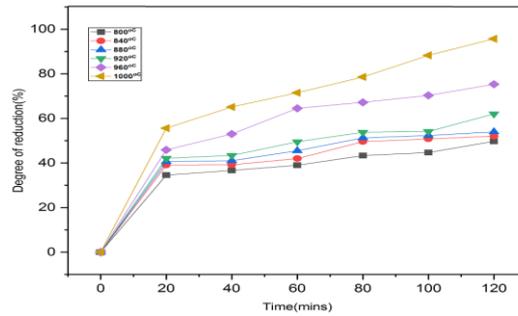


Figure 4. 2 Degree of Reduction (%) of resultant pellets from Agbaja e iron ore versus.

Time (in mins) at temperature between 800°C to 1000°C at temperature between 800°C to 1000°C

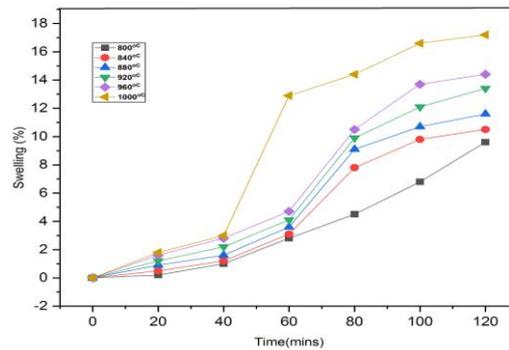


Figure 4. 4: Degree of Swelling (%) of resultant pellets from Itakpe iron ore versus.

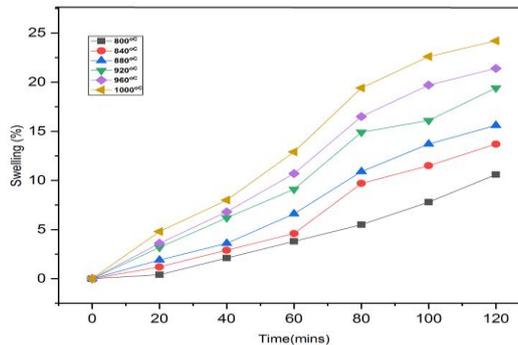


Figure 4. 5: Degree of Swelling (%) of resultant pellets from Agbaja iron ore versus at temperature between 800°C to 1000°C at temperature between 800°C to 1000°C.

Table 4.2 and figures 4.5 and 4.6 show the degree of reduction (%) of Itakpe Iron ore lumps and degree of reduction (%) of Agbaja iron ore lumps, While figures 4.7 and 4.8 show the degree of swelling(%) of Itakpe and Agbaja iron ore lumps as functions of furnace holding time for the degree of reduction(%) and degree swelling(%) Itakpe iron ore at temperature between 800°C – 1000°C.

Table 4: 2 Itakpe and Agbaja Iron Ore Lumps.

Temperature	Time (in min)	Degree of Reduction (%) Itakpe iron ore	Degree of Reduction (%) Abaja iron ore	Degree of Swelling (%) Itakpe iron ore	Degree of Swelling (%) Agbaja iron ore
800°C	20	54.8	17.1	3.67	2.01
	40	55.2	17.7	4.12	2.11
	60	57.7	18.2	5.45	2.24
	80	59.2	18.9	7.72	2.36
	100	59.8	19.6	10.5	2.5
	120	68.1	21.3	11.3	2.55
Temperature	Time (in min)	Degree of Reduction (%)	Degree of Reduction (%)	Degree of Swelling (%)	Degree of Swelling (%)
840°C	20	56.1	18	4.38	2.13
	40	57.1	18.2	5.39	2.25
	60	59.5	19.6	6.34	2.31
	80	60.7	20.6	8.33	2.46
	100	61.3	23.3	11.4	2.71
	120	69.7	25	12.3	3.33
Temperature	Time (in min)	Degree of Reduction (%)	Degree of Reduction (%)	Degree of Swelling (%)	Degree of Swelling (%)
880°C	20	60.1	20.2	5.12	2.44
	40	61.7	20.8	6.65	2.38
	60	62.6	21.4	7.97	2.5
	80	63.9	23.8	9.93	2.64
	100	64.3	24.8	12.3	2.98
	120	70.7	26.7	13.5	3.44
Temperature	Time (in min)	Degree of Reduction (%)	Degree of Reduction (%)	Degree of Swelling (%)	Degree of Swelling (%)
920°C	20	62.2	22.8	5.47	2.43
	40	63.9	23.2	6.65	2.56
	60	64.1	23.6	8.86	2.68
	80	65.6	24.1	10.3	2.82
	100	68.1	25.9	13	2.96
	120	72	27.1	13.6	4.09
Temperature	Time (in min)	Degree of Reduction (%)	Degree of Reduction (%)	Degree of Swelling (%)	Degree of Swelling (%)
960°C	20	63.3	23.4	6.35	2.51
	40	64.2	23.8	8.45	2.67
	60	66.5	24	9.68	2.98
	80	68.5	24.7	10.5	3.03
	100	71.4	26.1	13	3.13
	120	73	31	14.2	4.16
Temperature	Time	Degree of	Degree of	Degree of	Degree of

	(in min)	Reduction (%)	Reduction (%)	Swelling (%)	Swelling (%)
1000°C	20	64.9	24.2	7.58	2.82
	40	65.3	25.1	9.13	3
	60	67.4	25.8	10.6	3.13
	80	69.7	26.5	11	3.27
	100	72.9	29.5	13.9	3.99
	120	74.3	33	15.1	4.41

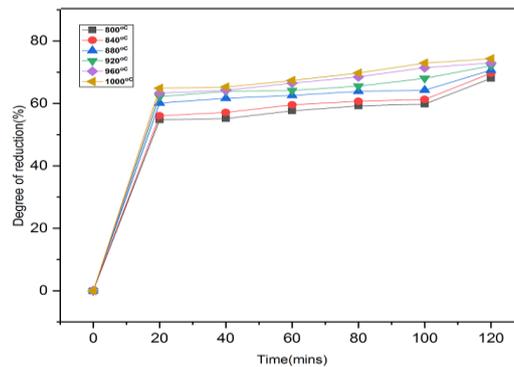


Figure 4.5: Degree of Reduction (%) of Itakpe iron ore lumps Versus Time (in mins) at temperature between 800°C to 1000°C.

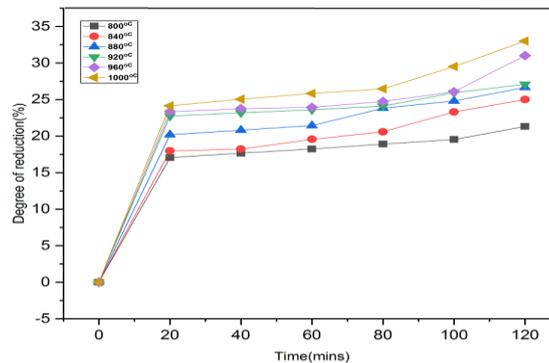


Figure 4.6 Degree of Reduction (%) of Agbaja iron ore Lumps Versus Time (in mins) at temperature between 800°C to 1000°C.

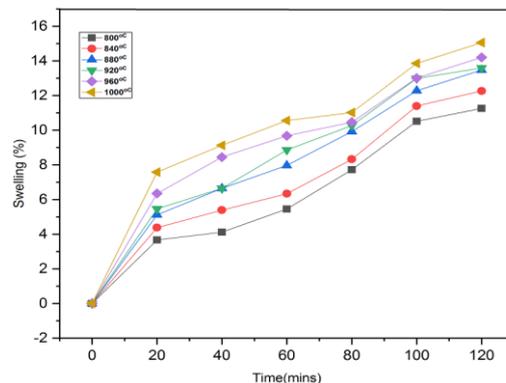


Figure 4.7: Swelling (%) Itakpe Iron ore Lumps Versus Time (in mins) at temperature between 800°C to 1000°C.

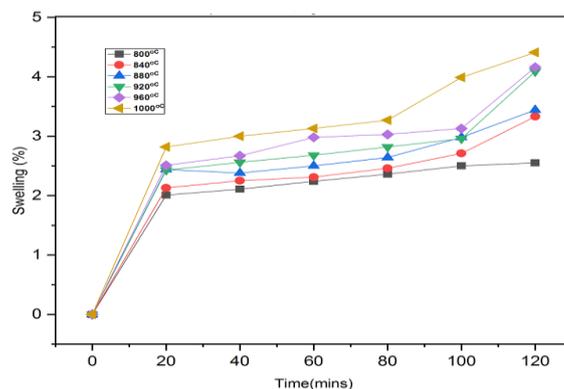


Figure 4.8 Swelling (%) Agbaja Iron ore Lumps Versus Time (in mins) at temperature between 800°C to 1000°C.

5.0 DISCUSSION

5.1 Reducibility and Swellability Studies on the Itakpe and Agbaja Iron Ore (Resultant Pellets and Lumps)

5.2 Reducibility and swellability comparison of resultant Pellets and Iron ore Lumps

In this study, reducibility behaviour of the Itakpe and Agbaja iron ores lumps (size: 20 mm approximately) was compared with those corresponding resultant pellets fired in muffle furnace.

The reducibility experiments were performed using the obtained metallurgical coking coal obtained from the Ajaokuta Steel Company Limited, Ajaokuta in Kogi State as reductant. The iron ore lumps were subjected under an identical slow heating temperature where the heating rate was at $8^{\circ}\text{C min}^{-1}$ was maintained. The samples were heated up to various temperatures ranging from 800°C - 1000°C . The samples were allowed to soak for periods ranging from 20min to 120mins. The corresponding value were collected and recorded and these obtained values were used to plot graphs as function of furnace holding time for the reduction and swelling resultant pellets and iron ore lumps at temperature between 800°C – 1000°C . The results obtained from the experiments showed that there were lower degree of reduction (%) and degree of swelling (%) in iron ore lumps than the corresponding resultant fired pellets.

It was noticeably observed that the iron ore lumps have lower porosity values that those of the corresponding values of the resultant fired pellets. It was clearly discovered that the lower level of porosity obtained from the iron ore lumps indicates the reasons why there was lower level of reducibility and swelling respectively.

The resultant fired pellets showed higher level of reducibility and swelling. The reasonable presence of the lower porosity values in both Itakpe and Agbaja iron ore lumps reveals the major reasons for the level of lower reducibility. From relevant publications, hematite and pisotilic pellets tend to form disordered and hence more reactive/easily reducible wustite (FeO). This could be the major reasons for the reducibility observed at relatively higher levels of the resultant pellets. The Itakpe iron ore lumps cracked into fine fragments figure 5.1, reduced Itakpe iron ore @ 1000°C for 120mins.

The breakdown in these lumps may be attributed to the higher rate of $\text{Fe}_2\text{O}_3 - \text{Fe}_3\text{O}_4$ transformation and generation of higher thermal strain. An increased degree of $\text{Fe}_2\text{O}_3 - \text{Fe}_3\text{O}_4$ transformation increases the volumetric strain (abnormal swelling) and thus the cracking tendency. In other case, the Agbaja iron were not fragmented at all. The Itakpe iron ore lumps are shown from figure 5.1 reduced iron ore at 1000°C for 120mi The same trend also occurred when the resultant pellets were subjected to temperature of 1000°C for 120mins. These observations were also indicated in figures 5.3 and 5.4 respectively.



Figure 5.1: Shows the reduced Itakpe Iron ore @ 1000°C for 120 mins.



Figure 5.2: Shows the reduced Agbaja Iron Ore @ 1000°C for 120 mins.



Figure 5.3: Pellet resultant from Itakpe Iron Ore @ 1000°C for 120 mins.



Figure 5.4: Resultant pellets from Agbaja Iron Ore @ 1000°C for 120 mins.

5.3 Effect of time on degree of reduction (%) and degree of swelling(%)

The research work carried out shows that the rapid heating of both the lumps and pellets shows that the heating time has an approximately significant effect on the reduction behaviour of both the iron ore lumps and pellets. As shown in Table 5.1 and 5, 2, all the fired iron ore pellets showed highest degree of reduction(%) and lowest degree of swelling(%) 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 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 H₂, CO, etc.

The major participation of these reducing gases in the reduction of iron oxide (i.e. an appreciable presence of H₂ 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 product iron layer offers greater resistance in the diffusion of carbon and reducing gas to the surface of unreduced iron oxide.

5.4 Effect of heating mode on the Degree of Reduction (%) and Degree of Swelling (%)

In this research work the effect of heating mode had effects on the degree of reduction(%) and the degree of swelling(%) in iron ore lumps and the resultant pellets. The fired pellets and lumps were reduced using metallurgical coking coal at temperature range between 800°C - 1000°C (the soaking times was varied from 20mins -120mins at an interval of 20mins), these experiments were performed under rapid and slow heating conditions in the muffle furnace.

It is clear that in comparison to rapid heating, the slow heating to reduction temperature gives appreciably higher degree of reduction and lower degree of swelling (%). It is more likely that rapid heating from 920°C to 1000°C causes higher rate of volatile matter escaping from the metallurgical coking coal, thereby providing less time for H₂ and CO (reducing gases) to be in contact with iron ore lumps and the pellets.

The results in this work thus indicates that there was lower degree of reduction in rapid heating in the iron ore lumps 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 (%) and lower degree of swelling (%).

Heating of hematite pellets from room temperature to the required reduction temperature ($1000^\circ C$) in reducing atmosphere, to some extent, was also responsible' for higher degree of reduction(%) and lower degree of swelling(%) under slow heating condition.

6.0 FINDINGS AND CONTRIBUTION TO KNOWLEDGE

The Itakpe and Agbaja iron ore have been known to have large deposits in Kogi State of Nigeria, it is therefore penitent to take the advantages created to convert these ores into pellets production for use in both the Blast Furnace and Direct Reduced Iron processes.

In the past, the government of Nigeria have been involved in the importation of high-grade ores. It must be noted that the processes of importation and financial constraints and cost implication which in the past has run into over 600 dollar /tonne even when the plants were not operated at the optimal capacity.

The results obtained in this research work carried ou reveal the successes and contributions to knowledge as they are viewed in these ways:

- (a) That the project was successfully completed and the data generated should serve as data bank which could further translate to valid information for further study on similar or other iron ores that are available in the country .
- (b) That the relevant data could assist the would be researchers in this field which has created many opportunities of the needed knowledge on the best methods and necessary reducibility and swellability studies .
- (c) That the experimental techniques explored and revealed the mineralogies and , morphologies of the ores, the reducibility and the swellability of the ores. Some important tests performed on the ores which include the TGA, XRF, XRD SEM and EDS. These tests assisted the researcher and would researchers to understand the techniques and procedures to be used for further studies . e. t. c
- (d) That the reducibility and the swellability tests indicated that the pellets resultant will meet the requirement for the operation and production of steel in both Blast Furnace and Direct Reduced Iron processes.
- (e) The research revealed that the pellets resultant from the Itakpe Iron ore reduced and swell more than the pellets resultant from the Agbaja Iron ore.

- (f) The implications of these results was that the pellets resultant from the blends from Itakpe and Agbaja iron ore could be used as sources of materials for the production of wrought iron or pig iron.

7.0 CONCLUSIONS

The study on the reducibility and swellability on pellets and the iron ore lumps were intensively investigated the studies carried out reveal the followings

- (a) The time of reduction and temperature of the pellets and the iron ore lumps indicates that there were greater influence on the degree of reduction (%) and the degree of swelling (%). It was observed that the degree of reduction (%) and degree of swelling (%) increased with increase in reduction and swelling from 800°C -1000°C. The time for the degree of reduction(%) and the degree of swelling (%) were performed within ranges of 20 -120mins as these processes indicts that the degree of reduction (%) were higher in the pellets than that of the lumps. Similarly, the degree of swelling (%) was lower in the pellets more than those of the iron ore lumps.
- (b) The reduction and swelling behaviour of the pellets and lumps are identical in all the studies. The use of the Metallurgical coking coal as reluctant had great effects on the pellets and the lumps as there were significant influences on the degree of reduction (%) and the degree of swelling (%) on the tested samples.
- (c) The pellets showed more reducibility and swelling tendency than their corresponding lumps samples. The reduced and swelled pellets on the ore samples had similar trend as they increased in the degree of reduction (%) and reduce in the degree of swelling (%).

From the findings of the study, the results and data obtained could be used for further studies while the other iron ore deposits in the country be subjected to other experimental investigations and processes. Such ores could also be investigated and subjected to the reducibility, swelling processes and blending them with other iron ores a view to generating the necessary data and to make comparism with those already investigated.

8.0 RECOMMENDATIONS FOR FUTURE RESEARCH

- (a) Enough funds will be needed to perform this kind of research work because of its capital involvement, which is intensive. In regards to this, some of the research institutions, universities and Industries where such research works should have a rate that could be greatly affordable by students and researchers. They should also be granted waivers in some cases .

- (b) Sourcing of the raw iron ores from the National Metallurgical Development Centre (NMDC), Jos, National Iron ore Mining Company (NIOMCO), Itakpe and Agbaja plateau in Kogi State posed some challenges.
- (c) The accessibility to source for these materials were very difficult as a lot of money was spent to obtain the materials and also the security network of the locations and the terrain were not easily accessible. Efforts should therefore be made by all the stakeholders to always assist researchers, students and other individuals by creating enabling opportunity to have access to these raw materials.
- (d) As regards the equipment used in performing these experiments, some of them are not readily available like Scanning Electron Microscope (SEM) with EDS, the Thermogravimetric Analysis (TA) and the Differential Thermal Analysis (DTA), the X-Ray Fluorescence (XRF) and other facilities, these issues posed a lot of challenges. Some of such equipment that are available are either having issues as in most cases they are faulty and where available the results resultant may be doubted.
- (e) The cost of performing such experiments are relatively high for an average student / researchers to afford, therefore Governments and other research Institutes, Universities and Industries should reduce the cost of performing such experiments with a view to encouraging researchers.
- (f) In view of the above therefore, the Universities, Research Institutes in Nigeria should collaborate with agencies like the TEFUNDS, PTDF, and SHELL CHEVRON, AGIP *et cetera* to supply such equipment and to collaborate with students in order to achieve optimal results.
- (g) The research work performed is not exhaustive as more works could be experimented with other iron ores available within Kogi State like the Agbajanoko, Oshokosho, Konto Karfi, Bassa Nge and Muro and in other States in Nigeria.
- (h) Finally, there should be synergy between Government, Universities, Research Institutes and industries with a view to solving basic problems through collaborations.

REFERENCES

1. Adeleye R.A., "The occurrence of Oolitic iron ore in the Middle Niger Basin in Geology of Nigeria". Kogbe, C.A. Edition Elizabeth press, Lagos, 1964; 1-10.
2. BRG report. survey, Research and test of Agbaja, Lokoja, Koton –Karfe and Bassange iron ore deposits, final report, 1983.

3. Adedeji F.A. & Sale F.R. "Characterization and Reducibility of Itakpe and Agbaja (Nigerian) Iron ores". Delta Steel Complex Warri, Nigeria and Department of Metallurgy and Materials Science, University of Manchester Grosvenor Street, Manchester M1 7HS", 1984; 843-856.