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Research Article

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The Study on the Reducibility of Itakpe and Agbaja Iron Ore

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Abstract The reducibility study on Itakpe and Agbaja iron ore were investigated. The X-Ray Fluorescence (XRF) was used to determine the chemical compositions while the Scanning Electron Microscopy (SEM) with Energy Dispersive Spectroscopy (EDS) were used to determine the morphologies. Thermogravimetry (TG), Differential Thermal Analyzer (DTA) were used to determine the deviation of wt. percent. The X-Ray Diffraction method was used to determine the characterization of the ore, while the Optical Inverted Metallurgical Microscopy was used for the determination of the microstructures of the ores before and after the reducibility tests. The reducibility studies were performed with the use of a muffle furnace. The iron ore lumps were kept inside six (6) stainless steel containers with diameter 0.675 cm x 0.5940 cm. The internal diameter of the stainless steel containers were properly covered with a small opening for the escape of gases during the experiments. Some quantities of Metallurgical coking coal sourced from the Ajaokuta Steel Company Limited were used as reducing agents. The highest reducibility value for the Itakpe iron ore was 74.33 per cent with the corresponding temperature at 1000 °C fired for 120 minutes, while the highest reducibility value for the Agbaja iron ore was 33.00 per cent with the corresponding temperature at 1000 °C fired for 120 minutes.

Keywords Study, Reducibility, Itakpe, Agbaja, Iron ore

Introduction

Nigeria is a country blessed with abundance of natural resources some of which are the Iron ores. These are found in some locations in Nigeria. The first Iron ore deposit was discovered in 1904 which has launched Nigeria into the technology space. Ever since this period more iron ore deposits have been discovered some of which are the Itakpe and Agbaja iron ore, both located in Kogi State. The deposits discovered are found to be made of hematite, magnetite, goethite or siderite – goethite grades. The reserves have been estimated at over 3 billion metric tonnes and their utilization deposits in iron and steel production will contribute meaningfully to the economy of the nation. [1]

The Itakpe iron ore deposit is located at the northeast part of Okene in the eastern part of Kogi State. The ores are one of the most investigated ferrous deposit in order to optimize the benefits of using them for steel production. These investigations have been tailored towards their utilization for the production of liquid steel at the Ajaokuta Steel Company Limited and the Delta Steel Company, Aladja.

The Itakpe deposits are made up plateau surrounded by hills. The ore deposit has been estimated to be over 300 million tonnes of reserves, while a proven reserve had been put at 200 million tonnes [1].

The Agbaja iron ores are traded in lumps and sometimes in fines. Divine of natural occurrence limits production/availability of lumps. The generation of lot of fines during the crushing of large, lumps present in the run-off –mines. 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 ores are upgraded to higher iron content through concentration. Iron ores are 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 other agents, 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 the velocity of iron oxide transformation to metal by effect of reduction gas, or time allowed for complete iron oxide reduction. The reduction value rate is the metallic charge weight loss per time unit.

The Influence of reducibility on the blast furnace operation is very essentials and necessary. It is used for fuel consumption determination and selection of proper lumpiness. The reducibility value changes particularly when hot treatment (sintering) is applied to it. The charge porosity influences the reducibility processes too. The higher the porosity of the charge, the larger the reaction surface and the faster gas reduction. The percentage loss of oxygen per minute this represents the value of the reducibility index [4].

The volume of the melting phase plays an important role during sintering. Overheating will cause homogeneous glassy structure in the ore, that will, in turn, become low in the reducibility per cent, A very low concentration of melting will cause insufficient sinter strength, that will result in a high amount of return fines [5,6].

An approach for an assessment of the influence of reducibility on indicators of blast furnace smelting with use as the main balance logic-statistical model of the blast furnace process [7–10].

Research Methodology

Materials

Itakpe Iron Ore

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 [1]. 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. The 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 [2]. It cannot be used directly in the Blast Furnace processes or another reduction process without further treatment e.g. palletization or briquette. The ore is a lean ore and sedimentary origin [2]. It is, therefore, necessary to harness the opportunities created to work upon the ore in order to provide services and economic development to our nation.

The ore is considered oolittic in nature, limonite that occur in mannmilated origin stalactite forms having fibrous structure resembling hematite [2]. The Agbaja Iron ores are 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 [three]. The ore particles could be processed further 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 magnetically Strong [3]. The picture below shows the iron ore as being sourced at the Agbaja Plateau in Kogi State.

Methods

The chemical compositions of the obtained iron ores are given in Tables 1 and 2. The Itakpe iron ore concentrate has phosphorus in traces while the Agbaja iron ore contains some amount of phosphorus.

Chemical analysis of Itakpe and Agbaja Iron ore

Table 1 and 2 show the results of the chemical analyses the Itakpe and Agbaja iron ore using an X-Ray Fluorescence (XRF) methods for the experiments.

Component	Unit	Result
Na ₂ O	mass%	0.3538
MgO	mass%	0.3853
Al_2O_3	mass%	12.2129
SiO ₂	mass%	20.5276
P_2O_5	mass%	1.5560
SO_3	mass%	0.1376
K ₂ O	mass%	0.6138
Cao	mass%	0.1096
TiO ₂	mass%	1.3976
V_2O_5	mass%	0.0846
Cr_2O_3	mass%	0.0640
MnO	mass%	0.1641
Fe_2O_3	mass%	52.5350
NiO	mass%	0.0247
CuO	mass%	0.0327
ZnO	mass%	0.0201
Rb ₂ O	mass%	0.0130
SrO	mass%	0.0142
ZrO ₂	mass%	0.0519
BaO	mass%	0.2015

Table on ore

Component	Unit	Result
Na ₂ O	mass%	0.1183
MgO	mass%	0.2464
Al_2O_3	mass%	6.0470
SiO ₂	mass%	25.2321
P_2O_5	mass%	0.0227
SO ₃	mass%	0.1899
Cl	mass%	0.0955
K ₂ O	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

Ray Diffraction (XRD) of Itakpe and Agbaja Iron Ore

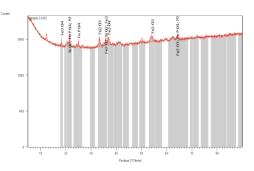


Figure 1: XRD of Itakpe Iron Ore

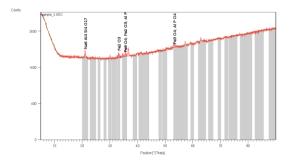


Figure 2: XRD of Agbaja Iron Ore

Mineralogical studies of Itakpe Iron Ore

The Mineralogical studies of deposits have been well documented in the recent years. They have yielded the following features; Hematite - 29.7%

Magnetite – 40.1% Silicon Oxide – 17.7 % Iron Phosphate-6.47%

Phosphorus Oxide 6.03%

The analysis is given in table 3.

 Table 3: Identified Patterns List: Itakpe 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	0.35	Na ₆ Al ₄ Si ₄ O ₁₇
*	86-0550	28	Hematite, syn	0	0.13	Fe ₂ O ₃
*	48-0652	9	Aluminum Phosphate	0	0.1	Al P O ₄

Mineralogical studies of Agbaja Iron Ore

The Mineralogical studies of the ore are as follows Hematite- 11.30% Magnetite – 49.6 % Sodium Aluminum Silicate 30.4 % Aluminum Phosphate-8.7% The analysis is given in table 4.

Table 4:	Identified	Patterns	List: A	Agbaja I	ron Ore	

Visible	Ref.	Score	Compound	Displacement	Scale	Chemical
	Code		Name	[°2Th.]	Factor	Formula
*	85-0599	50	Hematite	0	0.69	$Fe_2 O_3$
*	76-0931	49	Silicon Oxide	0	0.93	Si O ₂
*	74-1910	31	Magnetite	0	0.41	Fe_3O_4
*	31-0647	22	Iron Phosphate	0	0.15	Fe P O ₄
*	23-1301	12	Phosphorus Oxide	0	0.14	P_2O_5

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 vendor did the proximate analysis of the coal 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 5: Chemical	Analysis of	Metallurgical	Coking Coal
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Experimental Procedure of Reducibility

Reducibility gives the summary of raw materials properties that determine the rate of conversion of iron oxides to metals by treatment with reductant. 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

Iron ore lumps were used for these experiments, while a metallurgical coking coal was obtained from the Ajaokuta Steel Company Limited was used as the reductant. The collected iron ores lumps were crushed into 15-20 mm sizes. The collected Metallurgical coking coals were crushed to -5+ 15 size. The chemical compositions of the Metallurgical Coking coal is shown in table 5. The crushed iron ore lumps were dried in the laboratory dry oven to eliminate moisture content that was present in the ores. These ores were subjected to a temperature 120°C. The crushed lumps were kept inside six (6) stainless steel containers (size: 0.6075cm height × 0.5940cm inside diameter) with mouths tightly closed by airtight covers allowing escape of gases. The coking coals were used as redundant by putting them around the crushed ores in the containers. The coals serve as a reducing agent in the experiments at various period and time.

The crushed iron ore lumps were properly placed at the center of the solid packed bed. This was done to ensure that the lumps inside the packed were surrounded.

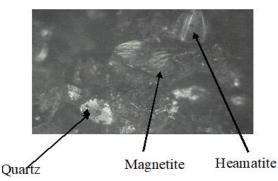
The experimental procedures were strictly followed. The muffle furnace was used to heat up samples of the lumps to the required temperatures of 800°C, 840°C, 860°C, 880°C, 920°C, 960°C and 1000°C, at 8°C per minute's rate. The samples were allowed to soak at various temperatures by varying the soaking period in the range of 20 -120 minutes.

The experimental process was performed to determine the reducibility at the stipulated temperatures. Each of the containers was properly labelled for specific identifications. The samples were brought out from the furnace at a designated interval of 20 minutes. The same processes were observed for the rest samples. The containers containing the samples that were brought out from the furnace were allowed to cool at the room temperature. The weight losses analyses of the iron ore were determined while the values obtained were recorded. The reducibility ore were calculated using the formula.

 $Degree of Reduction = \frac{weight}{Total weight of removal oxygen in Iron oxide} X 100 \%$

Microscope Examination

The Inverted Metallurgical Microscope was used to examine the ore before the reducibility tests were performed. Some important distinct phases were identified in the Itakpe ore are grey like structures, some whitish mottled and blackish/whitish location. This is shown in Figure 3. The micrographs of the Agbaja 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.





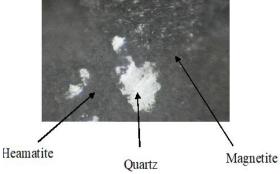


Figure 4: The microscope of the Agbaja Iron ore before the reducibility experiment

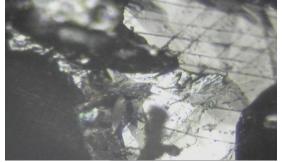


Figure 5: The Micrograph of Itakpe Iron ore e reduced @ 1000 °C (Fully reduced)

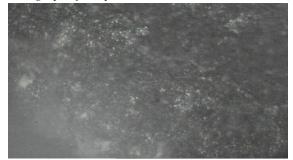


Figure 6: The Micrograph of Agbaja Iron reduced @ 1000 °C (Fully reduced)

Reducibility of the Ores

Figures 5 and 6 show the microstructures of the ore after the reducibility tests. In this case, it was observed that the ores reduced fully at 100 °C for 120 minutes for both ores

Thermogravimetric Analysis (TGA)

The Thermogravimetric Analysis (TGA) of Itakpe and Agbaja Iron Ores

The line with blue colour runs on 30° C and moved upwards until it got to the peak value of Derivation weight (%C) of 0.0048. This value declined and raised until it achieved a stable value and finally attained a value of

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0.0007 at the Derivation 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° C with corresponding value at 99.2.

Figure 7 shows the Isothermal behaviour of the thermal decomposition of the Itakpe iron ore. The line blue colour runs on 30° C got to 200° C and moved upwards it got to the peak value of Derivation. 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 Derivation weight (%C). While the light green line started from 100 weight (%), continue to decline, and dovetailed at 88.2 (weight %) with corresponding value at 1000° C.

Figure 8 shows the Isothermal behaviour of the thermal decomposition of the Agbaja iron ore.

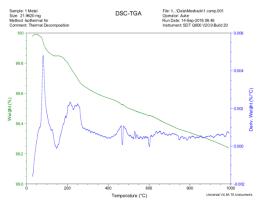


Figure 7: Thermogravimetry Analysis (TGA) performed on Itakpe Iron ore

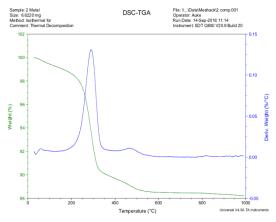


Figure 8: The Thermogravimetry Analysis (TGA) Analysis (TGA) performed on Agbaja Iron ore

Scanning Electron Microscopy (SEM) of Itakpe and Agbaja Iron Ore

The SEM with EDS was used to determine the morphologies of the ores. Figure 9 shows that there are grey phases such as quartz, white hematite, and mottled areas, which are intergrowths of hematite and magnetite, while figure 10 shows the distributions of elements. Figure 11 and 12 show the morphological structures of the ore after the reducibility tests at 100° C for 120 minutes. And the distributions of elements with wt%

Figures 13 and 14 show the scanning electron microscopy show the pisolitic/oolitic nature of this ore, which is typical of many sedimentary iron ores, considerably both with respect to amount and compositions of the ooliths, which may consist of hematite, goethite, siderite or chamosite In the same vein figures, 15 and 16 also shows the morphologies of the ore and the structure after the reducibility test at 100°C for 120 minutes.



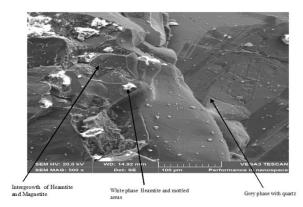


Figure 9: The scanning SEM of the Itakpe Iron ore at 100µm before the reducibility Test

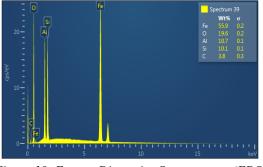


Figure 10: Energy-Dispersive Spectroscopy (EDS)



Figure 11: The scanning SEM of the Itakpe Iron Ore at 100µm after the reducibility Test at 1000 °C for 120m9ns

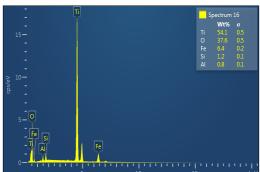


Figure 12: Energy-Dispersive Spectroscopy (EDS) of Itakpe Iron Ore @1000 °C after the reducibility Test

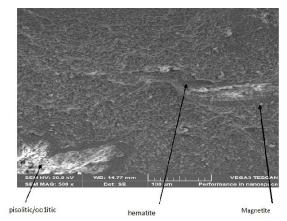


Figure 13: The scanning SEM of the Agbaja Iron ore at 100µm before the reducibility Test

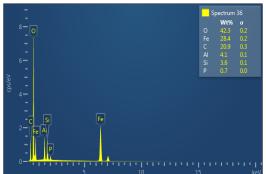


Figure 14: Energy-Dispersive Spectroscopy (EDS) Agbaja Iron ore before the reducibility Test

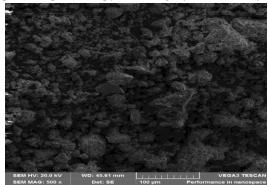


Figure 15: The scanning SEM of the Agbaja Iron ore at 100µm after the reducibility Test at 1000°C for 120m9ns

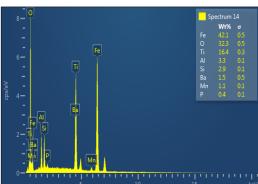


Figure 16: Energy-Dispersive Spectroscopy (EDS) of Agbaja @1000 °C after the reducibility Test



Results and Discussion Results

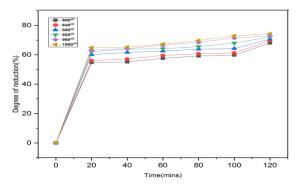


Figure 17: Reducibility of Itakpe iron ore lumps Versus Time (in mins lumps Versus Time (in mins) at temperature

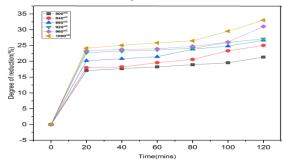


Figure 18: Reducibility of Agbaja Iron ore lumps versus Time (in mins lumps Versus Time (in mins) at temperature

Discussion

Reducibility Studies on the Itakpe and Agbaja Iron Ore Lumps)

In this study, reducibility behaviour of the dried Itakpe and Agbaja iron ores lumps were investigated.

The experiments were performed, where the obtained metallurgical coking coals were used reducing agents. The ore were subjected under identical slow heating temperature rate at 8°C min⁻¹.

The samples were heated up to various temperatures ranging from 800°C-1000°C. The samples were allowed to soak for periods ranging from 20minutes to 120minutes.

The corresponding values were collected and recorded. The obtained values were used to plot graphs as a function of furnace holding time for the reducibility of ore at a temperature between $800^{\circ}C - 1000^{\circ}C$.

The obtained results from the experiments showed that there were lower reducibility in the ores.

It was noticeably observed that the ores have lower porosity values. It was clearly discovered that the lower level of porosity obtained from the ores indicate the reasons why there were lower level of reducibility.

The breakdown in these iron ore was attributed to the higher rate of $Fe_2O_3 - Fe_3O_4$ transformation and generation of higher thermal strain. An increased degree of $Fe_2O_3 - Fe_3O_4$ transformation increases the volumetric strain and thus the cracking tendency. In another case, the Agbaja ore was not fragmented at all levels. The ore only scattered as shown in figure 18 and 19 at the same temperature of 920°C for 120 minutes. The same trend also occurred when the iron ore lumps were subjected to temperature of 1000°C for 120minutes. These observations were also indicated in figures 20 and 21 respectively.



Figure 19: The reduced Itakpe Iron Ore @ 960 °C for 120 mins



Figure 20: The reduced Agbaja Iron ore @ 960 °C for 120 mins



Figure 21: The reduced Itakpe Iron Ore @ 1000°C for 120 mins



Figure 22: The reduced Agbaja Iron Ore @ 1000 °C for 120 mins

Effect of time on Reducibility

Rapid heating of the ores noticeably had effects on the ores. These were indicated on the heating times, which approximately show significant effects on the reduction behaviour of the ore.

For effectiveness and efficiency of the furnaces, these results, therefore, translate to greater amount of energy savings and will extend the lifespan of the furnaces. The excessively high reducibility in the first 40 minutes was majorly due with the release of volatiles from the metallurgical coking coal used because of 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 increases 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 Reducibility

In this research work, the effect of heating mode on the samples indicated that they have effects on the reducibility on the ores with respect to the temperature range of 800° C - 1000° C. The allowed soaking times were varied from 20 minutes -120 minutes at an interval of 20 minutes). These experiments were performed under rapid and slow heating conditions.

It is clear that in comparison to rapid heating to slow heating, the reduction temperature gives appreciably higher reducibility. It is more likely that rapid heating from 920°C to 1000°C causes a higher rate of volatile matter from escaping from the coking coal, thereby providing less time for H_2 and CO.

The results in this work thus indicate that there was lower reducibility with tendency of the ore lowering during heating operation. The volatile matters were released from coal at a slower rate. The more depositions of highly reactive pyrolytic carbon and increased time of contact of carbon and reducing gases (H_2 and CO) with the ores appearing to be the obvious reasons for the higher reason for the reducibility attained.

Heating of iron ores from room temperature to the required reduction temperature of 1000°C in reducing atmosphere, to some extent, is also responsible' for the high level of reducibility.

Summary of the Analysis on Results after the Reducibility Test

 Table 6: Analysis of Results of Sample after Reducibility Tests using SEM and EDS on Itakpe and Agbaja Iron

 Ore Lumps

S/No	Sample Name	Temperature in degree	Fe (Wt. %)
1	Itakpe Iron Ore	800 °C	84.5
2	Itakpe Iron ore	920 °C	71.4
3	Itakpe Iron Ore	1000 °C	6.4
1	Agbaja Iron Ore	800 °C	90.2
2	Agbaja Iron Ore	920 °C	84.5
3	Agbaja Iron ore	1000 °C	42.1

The results on Table 6 indicates that the Itakpe Iron ore reduced at 800°C for 120minutes has a Fe content value of 84.5 wt. per cent, the Fe content reduced from 84.5 wt. percent to 71.4 wt. percent when the samples were heated to a temperature range of 920°C for 120minutes and at 1000°C for 120minutes the Fe content reduced to 6.4 wt. per cent. By these results, it means that the Itakpe iron ore was fully reduced at this temperature and time. This can be translated further to mean that the Itakpe iron ore reduced dues 800°C to 920°C to 1000°C. The results obtained were also represented on figure 23.

Similarly, the same trend was also observed for the Agbaja iron ore, which followed the similar pattern, but not with the same rate of reduction. It should be noted that at 800°C for 120 minutes the Fe content obtained stood at 90.2wt per cent, When the ore was heated to 920°C for 120 minutes a corresponding value Fe content was obtained as 84.5 wt. per cent. The sample was further subjected to reducibility test at 1000°C for 120minutes it was observed that the value of Fe content obtainedwas 42.1wt. percent as shown in figure 23. This process further indicates that the Agbaja iron ores showed a similar trend of reducibility from 800°C to 920°C to 1000°C, but it is a suffix to state that the Itakpe Iron ore is more reducible than the Agbaja iron ore this can be seen on table 6 and also on the figure 24. In the final analysis, the Itakpe iron ore reduced fully at 1000°C for a period for 120 minutes.

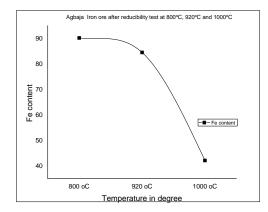


Figure 23: The Fe Content of Itakpe Iron Ore after the Reducibility test @ 800°C, 920°C and 1000°C using SEM and EDS

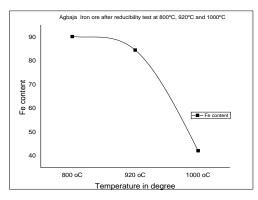


Figure 24: The Fe content of Agbaja Iron Ore after the reducibility test @ 800°C, 920°C and 1000°C using SEM and EDS

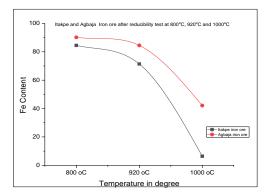


Figure 25: The Fe content of Itakpe Iron Ore VS Agbaja Iron Ore after the reducibility test @ 800°C, 920°C and 1000°C using SEM and EDS

Conclusions

The study on the reducibility of Itakpe and Agbaja iron ores were intensively investigated. It is worthy to note that the followings were revealed at the end of the research work:

(a) The time of reduction and temperature of the ores, indicate that there was a greater influence on the reducibility. It was observed that the reducibility increased with an increase in time and temperature from 800° C -1000°C. The period of time for the reducibility was performed within the time range of 20 -120 minutes as these processes influences the level reducibility

(b) The reducibility behavior of the ores are identical in all the studies. The use of the Metallurgical coking coal as reductant had great effects on the ores, as there were significant influences on the level of reducibility on

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all the tested samples.

(c) From the findings of the studies, it was discovered that the results and data obtained could be used for further studies while the other iron ore deposits in the country could also be subjected to some experimental investigations or processes on reducibility tests.

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