

Overcoming the phosphorous issue at the Agbaja iron ore project in Nigeria

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ABSTRACT

The Agbaja Iron Ore project in Kogi State Nigeria is a unique sedimentary hosted channel iron goethite deposit. The sedimentary ore allows for a simple beneficiation circuit resulting in a low-cost concentrate feed for pre-reduction and smelting to produce steel billets. However, the ore contains high levels of phosphorous (1-2 %) which need to be reduced for the crude steel to be suitable for steel billet production.

Previous work focused on generating an iron ore concentrate using magnetic separation and bioleaching techniques to improve the iron grade and reduce the phosphorous levels. This product would then be suitable for sale to steel manufacturers on the open market. However, the novel bioleaching method is unproven at commercial scale, with limited local markets available and able to accept the final product, hence there are high transport costs associated with getting the product to the international market. The ability to produce a crude steel product which can feed the local Nigerian steel mills, significantly improves the economics for the Agbaja project and reduces the reliance of the local steel manufacturers, who currently import 100 % scrap metal to satisfy their requirements.

Recent pilot scale test work investigated methods of producing such a crude steel low in phosphorus. Four main phases of testing were completed, namely, ore beneficiation, pre-reduction, smelting and refining test work. These tests have successfully demonstrated that a steel product, low in phosphorus suitable for local steel mills, can be produced from the Agbaja ore. The results of these phases of testing and the interpretation and development into a flowsheet suitable for the Agbaja ore are discussed.

INTRODUCTION

The Agbaja iron ore project is in the Kogi State, Republic of Nigeria. The Agbaja plateau hosts an extensive, shallow, flat-lying channel iron deposit and lies 15 km northwest of the city of Lokoja and 165 km south west from Nigeria's capital city Abuja.

Nigeria is the most populous country in Africa with a population of more than 170 million people. It is Africa's largest oil producer and oil accounts for 95 % of the country's exports. Because of this reliance on oil, the Nigerian Government is working hard to encourage new forms of investment and the mining sector is one of the highest priority industries. Currently all the steel requirements of Nigeria are reliant on imports and production through recycling scrap metal.

Agbaja run of mine ore can contain between 40 and 51.5 % Fe and is very rich in phosphorus (1.5-3.0 % P₂O₅) which is uniformly associated with the iron minerals. Phosphorus is a deleterious inclusion that is responsible for steel brittleness causing it to fracture and snap at very low stress values, also associated with phosphorus are the problems of strong primary segregation during solidification of castings and the formation of high phosphorus brittle streaks between metal grains thereby impeding plastic deformation (Anyakwo & Obot, 2011). Thus, for billet steelmaking iron the acceptable phosphorus level is < 0.045 Wt%.

Numerous investigations have taken place evaluating the potential to exploit the Agbaja ore. These have considered conventional mineral processing techniques used for treating magnetite iron ores, such as tabling and magnetic separation to producing a concentrate with sufficiently low enough levels of phosphorous that it can be marketed to steel producers without penalty. Unfortunately, the levels of phosphorous using such processes with Agbaja ore remain stubbornly high, leading to significant penalties being applied to the concentrates by the steel producers, hence rendering such routes as being economically unviable. Attempts to produce marketable concentrates using unconventional processes for iron ore typically found in base metal plants have been investigated and proven technically viable at prefeasibility study level. These were reported by Kogi in 2014 based on a bioleaching process of concentrates via VATs (Kogi Iron Limited, 2014).

With the fall of iron ore prices of late 2013 the unconventional bioleaching technique was considered to have too high a risk for successful project development in a non-mining country by Kogi. Alternative processing scenarios based on metal production for consumption by the local industry were then ranked higher for evaluation purposes as it was considered a local market was available for the product, if the phosphorous could be removed during the refining process.

A plant design based around size reduction comprising a three-stage crushing and screening circuit, followed by scrubbing forms the basis of the metal production flowsheets. Following washing, the ore is converted to sponge iron before being refined into steel. The conversion to sponge iron and hence steel, removes a significant portion of the impurities to within international standards producing a product suitable for consumption by the local Nigerian market.

While the design is specific to the oolitic orebody, Kogi's reduction process is long established as a method of making steel products throughout the world.

Fastmarkets in 2019 concluded that Nigeria has capacity to handle additional new billet production of up to 1.5 Mtpy and in the longer term forecast that if Nigeria were to reach per capita consumption of 21 kg in 2030 (still less than the 2017 average of 28 kg), billet demand could be ~4.5 Mtpy (Kogi Iron Limited, 2019).

Test work at bench scale to evaluate the full metal production route was completed at Mintek under the supervision of Tenova and SGS Bateman in 2016. This test work demonstrated that steel with suitably low phosphorous can be produced from Agbaja ore. To demonstrate this on a larger scale a 50-tonne sample of ore from Agbaja was mined and tested at pilot scale at Mintek in 2017/2018 which again showed steel with phosphorous levels <0.045 % can be produced from the Agbaja ore.

GEOLOGY AND MINERALISATION

Magnetite iron ore deposits generally grade around 25-40 % Fe, however the Agbaja mineral resource is a unique sedimentary hosted magnetite deposit with a resource grade averaging 41.3 % Fe.

Magnetite deposits are typically found in banded ironstone formations (BIFs), however Agbaja is unique in that it is a channel iron deposit (CID), with only two known similar deposits of this kind in the world. Typical BIF magnetite deposits require large amounts of energy intensive grinding to liberate the iron from its associated natural matrix, however the Agbaja CID material is relatively soft and friable and only requires moderate grinding resulting in coarse ground particle sizes necessary to liberate the iron.

The Agbaja formation and the iron mineralisation is laterally and aerially extensive across the Agbaja plateau and consists of an upper unit comprising beds of ferruginous sandstone and reworked oolite/pisolite material (referred to as the laterite unit) that overlies a sequence of massive ferruginous oolite and pisolite in a ferruginous matrix.

The sedimentary lithologies which comprise the Agbaja plateau are part of a cretaceous to tertiary rift and post-rift sequence. These sediments are overlain on a Pan-African metamorphic basement comprising granitoids, quartzofeldspathic orthogneiss and paragneiss, schist and quartzite including meta-BIF and magnetite quartzite. The Agbaja plateau occurs within the southern Bida Basin along the western margin of the Cretaceous Benue Trough.

The first detailed study of the mineralogy of the ironstone deposits was carried out by (Jones, 1958) as part of the early 1950s drilling program in the NE of the Agbaja plateau. The primary mineral comprised goethite including two principal varieties, "a-goethite" and "b-goethite". The black, sub-metallic a-goethite formed the majority of the oolites with the latter dull-brown to near black b-goethite forming the matrix to the a-goethite oolites. Magnetite was common throughout the unweathered and weathered sections of the sequence occurring as fine round granules (<0.005 mm in diameter and average ~0.003 mm) evenly distributed through the oolites and groundmass, but more commonly concentrated to form concentric zones in the oolites or on their rims. Siderite was abundant in unweathered ironstone with pseudomorphic relics common in the weathered rocks.

Other less common mineral components include: Pyrite, irregularly present in the unweathered oolites and as local relict thin bands or irregular patches rich in pyrite within the weathered zone; Chlorite, present in both the oolites and the groundmass; Quartz, generally absent in most of the ironstones but Jones describes its presence "as discrete lenses or in concentrations of coarse non-oolitic grit or gritty clay in the upper part of the ironstone" which appears to refer to the lateritic overburden. Minor secondary hematite was only present as a thin crust on exposed surfaces of the rock. The presence of phosphorous is assumed to be in a form of cellophane within less weathered lithologies. In the weathered oolites, phosphate was commonly present as cavity filling.

Phosphorus bearing minerals include apatite and a crandallite group mineral. Apatite was present as fine grains within siderite in the matrix. Crandallite group mineral are present as fine discrete grains within ooids and typically contained both calcium and strontium.

PRELIMINARY INVESTIGATIONS

Preliminary investigations into the development of a pyrometallurgical process to produce billet steel from the Agbaja ore were carried out at Mintek in 2016. The test work employed laboratory scale equipment to simulate key processes in the flowsheet of the envisaged commercial operation. The first scoping test work studied pre-reduction and smelting characteristics of the ore concentrate. The second scoping test work expanded the scope to include refining.

Thermogravimetric analysis was used to assess pre-reduction reactions of the concentrate in the presence of reductant and flux. Optimum conditions were established with the ore concentrate blended with 110 % carbon and 10 % CaO at a reaction temperature of 1100°C. Pellets were prepared and placed in sintered alumina crucibles and reacted at 1100°C in an induction furnace for 2 hours under an inert argon (Ar) atmosphere. The smelting tests established 1600°C as a suitable reaction temperature. Each test packed 100 g of pre-reduced materials in sintered alumina crucibles and reacted the materials at 1600°C in an induction furnace for an hour under an inert Ar atmosphere. The pre-reduction step achieved 89 % reduction and 85 % metallisation of Fe. The smelting step produced a pig iron with a grade of 93 % Fe. However, the pig iron did not meet specifications for billet steel due to high concentrations of impurities like C (2.9 %), P (0.7 %) and Si (2.29 %). The smelting tests also assessed the effect of trimming additions of reductant and flux which was found to have minimal effect on the recovery of Fe and grade of pig iron. Further test work was thus recommended to investigate the removal of the impurities from the pig iron.

The second scoping study focused on refining of the pig iron to remove the impurities through converting. The converting tests remelted 165 g of pig iron in sintered alumina crucibles using an induction furnace and introduced oxygen gas into the melt at 180-250 cm³/min to oxidise the impurities at reaction temperatures between 1500-1600°C while making small but regular additions of lime (CaO). These tests managed to readily oxidise all other impurities from the pig iron but difficulties were encountered with oxidising P sufficiently enough to meet the billet specifications (<0.045 % P). The P was eventually refined below 0.045 % using MgO crucibles and CaO-SiO₂ synthetic slag with a basicity of 2. Some of the parameters that were identified as critical to P removal included fluxing requirements, reaction temperature, and slag basicity. The lowest P concentration obtained in the crude steel was 0.039 % P. It was crucial to conduct further test work on a larger scale in order to quantify the parameters dictating effective removal of P.

ORE BENEFICIATION TEST WORK

Historical test work (pre-2017) on the Agbaja deposit involved a series of investigation into the upgrade of the ore. Initial test work focused on production of a final concentrate that can be sold to downstream pyrometallurgical customers. Upgrading by means of heavy liquid separation (HLS), low intensity magnetic separation (LIMS) as well as wet high intensity magnetic separation were investigated. However, physical separation techniques were unable to reduce the phosphorous in the ore to acceptable limits, which necessitated the consideration of additional, alternative processing unit operations. The circuit selected for study at prefeasibility level included grinding to minus 600 microns followed by coarse LIMS. The coarse magnetics were subjected to further grinding followed by multiple stages of LIMS to produce a final magnetic concentrate as feed to the downstream bioleaching circuit. The objective of the bioleaching circuit was to reduce phosphorous levels in concentrate to below 0.25 %.

A technology using high pressure washing scrubbing was investigated. High pressure water scrubbing in the range of 50 to 140 bar was tested. The tests showed the possibility of producing clean concentrates in the coarse (+2 mm) fraction, however there wasn't any significant upgrade in Fe content. Phosphorous levels were also not reduced. However, as this test work showed promise to scrub and then de-slime the ore, the technique was considered for further investigation.

In early 2017, Tenova and SGS Bateman were commissioned to oversee the test work for the development of a metal production flowsheet. At this stage, it was already decided that the final product would be billet steel. As the pyrometallurgical smelting and refining unit operations were to be the final steps in the circuit, the ore beneficiation test work was aimed at producing feed suitable for smelting on site as opposed to external customers.

A 50-tonne bulk sample of iron ore was shipped from Agbaja, Nigeria to Mintek in Johannesburg, South Africa for beneficiation and pyrometallurgical test work.

Mineralogical Characterisation

The ore was crushed to -30 mm and X-Ray diffraction (XRD) performed to quantify the relative abundance of the minerals present in each of the size fractions assayed as per Table 1.

Table 1 - Mintek XRD test results showing relative abundance of minerals

Mineral	Formula	Head	-425 µm	-1.18 mm + 425 µm	-13 + 1.18 mm	30 + 13 mm
Maghemite	$\gamma\text{-Fe}_2\text{O}_3$	13.7	11.0	17.1	11.5	12.8
Hematite	$\alpha\text{-Fe}_2\text{O}_3$	5.9	0.0	4.5	7.7	5.4
Goethite/Limonite	$\text{FeO(OH)/FeO(OH)nH}_2\text{O}$	72.4	60.3	71.9	75.5	77.9
Quartz	SiO_2	1.5	5.6	4	1.9	1
Clay (kaolinite)	$\text{Al}_2\text{Si}_2\text{O}_5(\text{OH})_4$	5.9	11.4	2.7	3.5	2.9
Fe spinel	$(\text{Fe,Mg})(\text{Cr,Fe,V})_2\text{O}_4$	-	7.8	-	-	-
Plagioclase	$(\text{Na,Ca})\text{Al}_2\text{Si}_2\text{O}_8$	-	4.0	-	-	-

Silicate minerals are mostly present in the -425 µm fraction at a lower iron concentration compared to the coarser fractions. Screening out the -425 µm fraction will remove a larger fraction of gangue and clay compared to iron.

Physical Ore Characterisation

Mintek performed characterization test work to evaluate the comminution properties as well as the amenability of the ore to magnetic separation.

The ore was characterised as very soft to medium hardness, with the following indices:

- Uniaxial compressive strength (UCS) tests classified the samples as very soft with average UCS values of 6.9 to 21.4 MPa.

- Bond crushability work index (CWI) testing classified the samples as very soft with average CWI values of 8.8 to 9.8 kWh/t.
- The Bond abrasion index (Ai) test showed that the material can be classified as being non-abrasive with abrasion index values ranging from 0.03 to 0.05 g.
- Bond rod work indices (BRWI) ranging from 10.0 to 10.2 kWh/t were measured and this classified the samples as being medium hard in response to rod milling.
- Bond ball work index (BBWI) test results classified the material as medium hard for both limiting screens tested. BBWI values of 11.61 and 11.98 kWh/t were reported at a 212 µm limiting screen. Testing at a 106 µm limiting screen size reported BBWI values of 13.2 and 13.3 kWh/t.

The results of the scoping level magnetic separation tests on the scrubber product showed that no discardable fraction was produced, as most of the non-magnetic fraction still contained 45.5 % Fe compared to a feed grade of 52.3 % Fe. Mineralogical analysis of the magnetic separation products showed that all products comprised of unliberated ooids of hematite/magnetite/maghemite cemented by the hematite/magnetite and goethite. Magnetic separation was therefore not a viable technology to use for waste rejection.

Scrubbing Tests

Scrubbing tests were conducted to evaluate the possibility of upgrading the iron content by rejecting a slime fraction high in silica. Conventional scrubbing tests were conducted at Mintek and comparative high pressure scrubbing test work conducted in Australia.

Mintek Scrubbing Tests

Batch scrubbing tests were carried out on ore crushed to -30 and -16 mm, followed by pilot scrubbing tests on -16 mm material to remove surface coatings and clays.

Samples pre and post scrubbing were assayed by size to understand how iron and gangue minerals are distributed within the different size fractions. The Fe grade was found to be consistent throughout the different size fractions except in the finer size fractions, which had Fe concentration of <50 %. This size class also had a higher concentration of the gangue minerals.

Scrubbing at 30 and 16 mm feed produced similar mass fractions of fines (-1 mm and -425 µm), so the discard fraction on either scrubber feed sizes would be similar. The scrubber feed size of 30 mm seems to be more efficient in removing the surface coatings, clays and other gangue minerals on the surface of the iron minerals. This was assumed to be the effect of larger particle sizes in the rotary scrubber. Fe and the Si were shown to follow the mass split, as a result, the higher the discard fraction, the higher the Fe losses will be.

Post scrubbing analysis showed the consistency of the Fe grade within different size fractions. It also showed that the silica was mostly concentrated in the finer size fractions. Assuming a scrubber feed size of 30 mm, screening out the -425 µm fraction will only lead to ~13 % Fe losses, at ~38 % silica rejection. Scrubbing and screening out the -425 µm size fraction helps remove a larger fraction of the gangue and clay minerals.

High Pressure Scrubbing Test work

Three samples were selected for scrubber testing designated as West Oolite (W/O), East Oolite (E/O) and the Top Oolite samples. In order for a fair comparison of the two scrubbing technologies, the same samples were split into two, with one half subjected to rotary scrubbing and the other to high pressure scrubbing. There was insufficient sample of the Top Oolite sample to do both the rotary scrubbing and high-pressure scrubbing, thus only the West and the East Oolite samples were tested by high pressure scrubbing.

Figure 1 (for the West Oolite sample) shows that the high-pressure product is significantly finer than the rotary scrubber products and will lead to even higher Fe losses as the Fe follows the mass split.

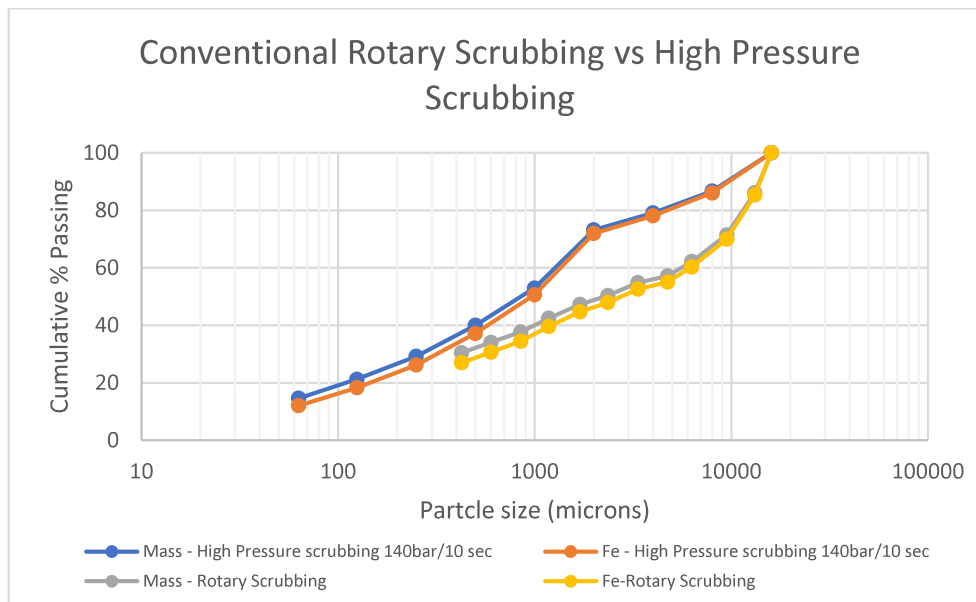


Figure 1 -16 mm Scrubber product comparison

Although the high-pressure scrubbing is very efficient in removing Silica it also discards a high proportion of Fe to the slimes fraction. These high Fe losses can be attributed to the ore being friable and easily disintegrate when subjected to the high-pressure washing. Instead of just washing the clays and silicates from the mineral surface, high pressure scrubbing actually breaks the ore particles down, generating more high-grade fines and is thus not suitable for this ore. It was thus concluded that conventional scrubbing using a rotary scrubber would be adequate and appropriate for the Agbaja ore.

The ore beneficiation test work resulted in a simple front-end flowsheet with 3 stages of crushing down to 16 mm. A rotary scrubber in closed circuit with de-slimes screens and tertiary crushing produces a clean -16 + 1 mm product for pre-reduction.

PRE REDUCTION TEST WORK

In the proposed flowsheet the beneficiated ore (ore) goes through a pre-reduction stage before it is introduced into the smelting stage in the open slag bath furnace (OSBF) to produce a high phosphorus pig iron. Pre-reduction is necessary to reduce the electrical energy consumption during smelting.

The important variables that needed to be ascertained in pre-reduction tests were:

- The suitability of the two types of reductants for pre-reduction in a rotary kiln
- The degree of metallisation that can be achieved on the as-received ore without pelletising. Due to the significant capital and operating costs associated with pelletising, it was decided to firstly explore the levels of metallisation without pelletising.
- The effect of particle size of both ore and reductants on metallisation
- The optimum operating temperatures, where maximum metallisation is maintained while balling of the material is avoided to prevent build up on the kiln lining

A 200 kg sample (crushed to 16 mm, scrubbed and de-slimes at 1 mm) from the Mintek beneficiation tests was used for the pre-reduction test work.

The pre-reduction test work consisted of two stages. In the first stage laboratory scale tests were performed for ore characterization, to establish expected operating temperatures and to find out which reductant of the two types that were investigated performed better. The second stage consisted of rotary kiln tests carried out to confirm the results from the first stage and to obtain process data that can be used for economic evaluation of the proposed process flow sheet, as well as the basis for the design of the commercial rotary kiln.

Laboratory Tests

The raw materials used in the tests were Agbaja iron ore concentrate obtained from Mintek beneficiation test work, and black and yellow coals sourced from Nigeria. No bentonite or fluxes were used in the test work.

Iron Ore Concentrate Characterization

The following tests were performed on the concentrate:

- Chemical and moisture analysis
- Ore grain-size distribution with sieving
- Thermal Differential Analysis (TDA) tests
- X-ray diffraction and fluorescence analyses

Chemical Analysis

The chemical composition of the ore sample is shown in Table 2.

Table 2 - Chemical analysis of the ore from Mintek beneficiation tests

Item No.	Chemical compound	Weight ratio, %
1	Fe _{tot}	52.52
2	Fe _{Met}	not detected
3	FeO	not detected
4	Fe ₂ O ₃	75.1
5	SiO ₂	4.89
6	Al ₂ O ₃	6.9
7	MgO	0.056
8	CaO	0.093
9	MnO	0.073
10	TiO ₂	0.183
11	P	0.98
12	S	0.004
13	C	0.17
14	LOI	10.81
15	Moisture	2.08

Particle size analysis

The particle size of concentrate obtained from the beneficiation test work at Mintek was smaller than 30 mm. All the tests were carried out on -15 to +5 mm material. Particles larger than 15 mm would be difficult to reduce and those smaller than 5 mm would melt and form undesired wall accretions on the kiln lining during reduction. Furthermore, using smaller than 5 mm material would promote excessive dust generation during charging into the kiln. Both these assumptions were later verified in subsequent tests.

TDA Tests

TDA tests were conducted to evaluate the physical and chemical processes occurring during heating of the ore from ambient to kiln operating temperatures. The tests were conducted under ambient atmospheric conditions up to temperatures of 1400°C.

It was found that at 109°C free moisture is eliminated from the ore, when the temperature increases to 325°C the capillary moisture is removed. Weight of the sample changed by 10 % when heated from 400 to 1400°C. This is due to the sample being predominantly Fe₂O₃ at 400°C and at 1300°C the partial dissociation of Fe₂O₃ to FeO.Fe₂O₃ commencing.

X-ray diffraction and fluorescence analyses

X-ray diffraction and fluorescence analyses were carried out to study the ore mineralogy and structure. X-ray structural analysis indicated that the ore consists mainly of poorly ordered goethite, maghemite and silicates, and, quartz and layered silicates. An x-ray amorphous substance is also present in the sample, which, most probably, contains iron. According to x-ray fluorescence analysis, apart from iron oxides, Ti (0.1-1.5 % weight), Pb (70-100 ppm), Rb (60 ppm), Zr (160 ppm) are also present in the sample. These metals, especially Ti, are distributed unevenly and the mineral composition of the ore is shown in Table 3.

Table 3 - Ore mineral composition

Mineral	Formula	Content, %	%Fe in the ore
Goethite	FeO(OH)	81	50.9
Maghemite	γ-Fe ₂ O ₃	11	7.7
Quartz	SiO ₂	1	0
Kaolinite/serpentine	Al ₂ (Si ₂ O ₅)(OH) ₄ , Mg ₃ (Si ₂ O ₅)(OH) ₄	5	0
Mica	KAl ₂ (AlSi ₃ O ₁₀)(OH) ₂	1	0
Talc	Mg ₃ (Si ₄ O ₁₀)(OH) ₂	1	0
Total		100	58.6

Reductants

Two types of coals were available to use as the reductant for pre-reduction and for smelting. Both coal samples were sourced from Nigeria and they were referred as yellow and black coal due to the yellow colour of the high ash containing coal. The analysis of the yellow and black coal samples are shown in Table 4.

Table 4 - Yellow and black coal sample analysis

Value	Unit	Yellow coal	Black coal
DFC (Dry fixed C)	%	49.2	50.3
Volatiles	%	22.5	41.5
Ash	%	28.3	8.20
Total C	%	56.8	69.3
Total S	%	0.83	0.52
Hydrogen	%	4.40	5.60
Moisture	%	2.20	9.60
Ash analysis			
SiO ₂	%	67.5	72.8
Al ₂ O ₃	%	21.8	12.8
TiO ₂	%	2.48	0.82
Fe ₂ O ₃	%	3.99	9.84

Value	Unit	Yellow coal	Black coal
CaO	%	1.20	0.15
MgO	%	0.16	0.41
K ₂ O	%	0.12	0.48
Na ₂ O	%	1.65	0.99
SO ₃	%	1.08	1.73

Calorific values were 8154 and 7330 Kcal/kg for the yellow and black coals respectively. The ash fusion temperatures were found to be 1440°C and 1500°C respectively.

Laboratory Scale Reduction Tests

The main focus of the laboratory scale reduction tests was to determine the effect of C-to-ore ratio in the blend on metallisation, as well as to find out which coal performed better in terms of metallisation levels that can be achieved.

Heat treatment of the ore, and reduction tests were performed in a laboratory scale furnace. The furnace is a vertical cylinder, its internal space lined with refractory and insulating materials. Heating elements are silicon-carbide rods located on the circumference. Tests were conducted under N₂/Ar gas mixture. The maximum temperature in the reaction zone is 1400°C. A controller ensures the furnace heating rate and maintains the required temperature during holding.

The test procedure consisted of preheating the furnace under neutral (N₂/Ar) atmosphere to 180-200°C and placing a wire basket containing the test mixture into the hot zone of the furnace. During the test, the change in the sample weight and temperature were registered every sixty seconds on a workstation. After reaching the desired temperature, the sample was exposed to heat for a pre-determined period of holding time that lasted 60 minutes, while weight and temperature recording continued. After the completion of the test, the sample was cooled to 150°C under neutral atmosphere before the furnace was switched off.

Laboratory Scale Test Results

As shown in Table 5, 29 % metallisation was achieved when C content in the raw material mix was at 30 % by using black coal, compared up to 11 % metallisation at C content in the mix of 9 to 21 %. Black coal resulted in higher metallisation levels compared to yellow coal at all C levels in the mix. The furnace temperature was kept constant at 1155°C throughout all the test runs.

Table 5 - Summary of results from laboratory scale tests performed at 1155°C.

Wt. %C in recipe	Metallisation, %	
	Black coal	Yellow coal
9	2.3	2.5
13	3.1	2.7
21	10.7	6.5
30	29.0	20.7

Tests in Rotary Kiln

The rotary kiln tests were conducted to evaluate the effect of temperature, raw material particle size, C content of the ore and reductant mix, and the type of coal on the degree of metallisation that can be achieved in a rotary kiln. The energy source in the rotary kiln was natural gas. The experimental set-up is shown in Figure 3.

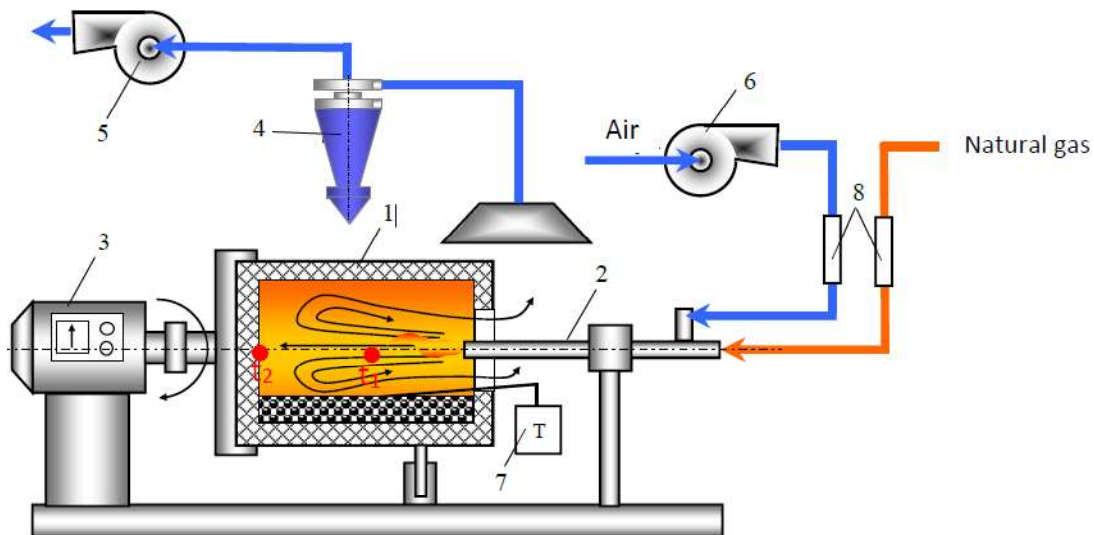


Figure 3 - Rotary kiln experimental set-up: 1: rotating drum, 2: burner, 3: electric drive, 4: cyclone, 5: exhaust fan, 6: forced-draft fan, 7: thermocouple and 8: rotameters

Experimental Procedure

The kiln was rotated at 11-12 rpm and the sample size was approximately 2 kg for all the tests. The experimental procedure consisted of:

- Preheating the kiln to 250-300°C for approximately 30 minutes followed by charging the raw material mixture into the kiln through the opening at the burner end of the kiln.
- After charging the feed mix, the burner is changed to operate with $\alpha < 1$. Below 850°C the heating rate is maintained at 10-15°C/min to ensure the removal of coal volatiles and homogenous heat up of the coal lumps.
- After reaching 850°C the heat up rate to the desired temperature (1100-1200°C) was not limited.
- After reaching the pre-set temperature the holding time starts. At the end of the holding time material is discharged.
- The discharged material is divided in two batches: the first one is cooled in the water, and, the second one under Ar/N₂ atmosphere.
- The cooled batches are then sent for chemical analysis.

Results from Rotary Kiln Tests

The products from the tests were first tested with a magnet to determine if they were magnetic before being sent for analysis. Only samples that were magnetic were sent for analysis. It was observed that the rate of reduction visibly increased with increasing temperature from 1000 to 1200°C. At 1000°C the samples were mostly nonmagnetic and contained large amounts of unreacted coal particles. Whereas, the samples treated at 1100°C and higher were mainly magnetic with unreacted coal particles mostly diminished or significantly reduced. The safe operating temperature was determined to be between 1100 and 1150°C considering the negative effect of high temperatures on the kiln refractory, and material starting to stick on the kiln walls. Higher levels of metallisation were achieved with C content in the mix of 30 % and higher, and that black coal produced considerably better results than yellow coal.

Particle size of the ore has major influence on the degree of metallisation. Tests indicated a higher level of metallisation on the -10mm particles compared to the +10mm particles. Using black coal at 30 % carbon content, and ore less than 10 mm resulted in 74.7 % metallisation at 1117°C and a retention time of one hour. Increasing the C content to 35 % resulted in a minimal increase in the metallisation from 74.7 to 76.7 % for the same particle size.

Tests to determine effect of particle size of ore and reductant on metallisation

The ore and coal particle sizes are compared to metallisation levels achieved in Table 6. The C content in the blend was kept constant at 35 %, temperature was in the range 1106 to 1120°C, and holding time of one hour.

Table 6 - Particle size versus % metallisation

Test No	Particle size, mm		% Metallisation
	Ore	Black coal	
19	12-15	5-10	71.5
20		3-5	77.0
21		1-3	83.1
22	5-10	5-10	82.3
23		3-5	85.3
24		1-3	91.8

Data from Table 6 is plotted in Figure 4 as % Metallisation vs. coal particle size. The degree of metallisation increases with decreasing coal and ore particle sizes. As the coal size decreases from 5-10 mm to 1-3 mm, and ore size decreases from 12-15 mm to 5-10 mm, the average metallisation increases by nearly 12 % from 77.2 to 86.5%.

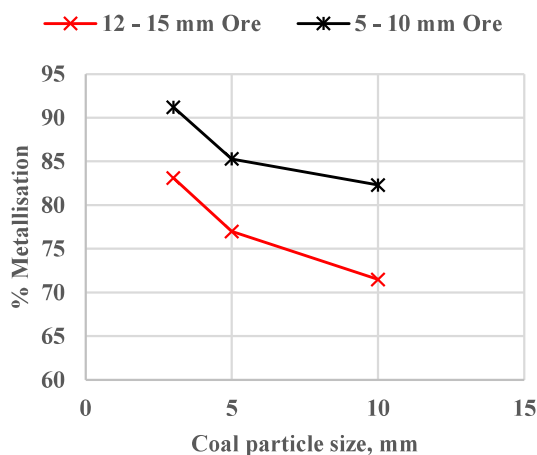


Figure 4 - Coal particle size vs % metallisation

Conclusions from the Pre-reduction test work

The following conclusions were drawn from the pre-reduction test work:

- Of the two types of coals identified as being available for the project locally, black coal consistently produced better metallisation compared to the yellow coal.
- The optimal operating temperature was determined to be between 1100 and 1150°C considering the negative effect of high temperatures on the kiln refractory, and material starting to stick on the kiln lining, while maintaining the highest levels of metallisation.
- Metallisation increases with decreasing coal and ore particle sizes. Highest metallisation of 91.8% was achieved with 1 to 3 mm coal and 5 to 10 mm ore sizes.
- Ore particles less than 5 mm were excluded from the test work due to excessive dust formation and balling tendency of the ore at the kiln operating temperatures.
- The average metallisation, for the ore sample between 5 and 10 mm and coal of between 1 to 10 mm is 86.5%.

SMELTING TEST WORK

Traditional crude steel production is carried out through the blast furnace/basic oxygen route and contributed about 70 % of the 1.7 billion tonnes of world crude steel production in 2017 (World steel association, 2018). Blast furnaces have the benefit of economies of scale but their requirement of constant supply of large quantities of coke result in significant amounts of CO₂ emissions that create environmental issues. Production of steel through electric arc furnace have seen increasing interest by the steel industry. The electric arc furnace route offers advantages of lower capital costs and improved control over the process for increased thermal and process efficiency. The use of an electric arc furnace was identified as a better engineering solution for the exploitation of the Agbaja deposit as this will enable prompt supply to the rapidly growing demand of the local Nigerian steel market estimated at some 20 million tonnes per annum (Bolaji, 2017).

A feasibility study to demonstrate production of crude steel from the ore was therefore initiated (by Kogi Iron Limited). The proposed test work flowsheet involved pilot scale smelting of ore concentrate prepared through the established ore beneficiation flowsheet. The pilot smelting test work made use of Mintek's 200 kVA DC arc furnace facility with the aim (i) to demonstrate the technical feasibility of smelting beneficiated ore in an electric arc furnace to produce a crude pig iron, (ii) to evaluate the quality of the products with respect to iron extraction and deportment of impurities, and (iii) to estimate the energy requirement of the process when smelting beneficiated ore. Take note that the commercial scale furnace will treat pre-reduced ore in order to lower the energy requirements of the smelting process. The specific energy requirement measured during the pilot test work will thus not be applicable for the commercial scale furnace. Phosphorus was of particular interest because the Agbaja ore had an average P concentration of 1.62 % P₂O₅ which is an order of magnitude higher than typical iron ores found in South Africa, for example, Sishen iron ore has a P concentration of 0.16 % P₂O₅ (Taylor, Page, & Geldenhuys, 1988).

Facility Equipment and Operating Procedure

The facility featured a feed system, single electrode DC arc furnace and an off-gas handling system equipped with a bag filter plant, extraction fan and a stack. The furnace had a water cooled 980 mm shell diameter lined with alumina castable refractory (93 % Al₂O₃, 5.5 % CaO + MgO) with wall thickness of 100 mm. The raw materials were charged into dedicated feed hoppers resting on load cells. The control system allowed the feed system and furnace power input to be fully controlled remotely. Furnace operating parameters and process variables were monitored and logged continuously. Operation of the furnace followed a series of operating conditions with varied amounts of reductant additions. Reductant addition was increased from 19 – 25 % C (Sascarb) through the different operating conditions but limestone (CaCO₃) was constant at 18 %.

An average batch mass of 131 kg was fed at 37 kg/h with the furnace operated at an average power of 202 kW for each batch. A total of 45 batches was processed through the furnace. The smelting operation was batch-continuous meaning a predetermined batch mass was continuously fed to the furnace and tapped at the end of the batch. The power-to-feed balance was based on a theoretical estimate of the Specific Energy Requirement (SER) of the process.

Feed and product materials were weighed, sampled and analysed continuously to provide data about the behaviour of the elements in the process. Sampling of metal and slag was performed in the molten state during tapping operations. Feed concentrate and dust were sampled after every five taps by the grab sampling technique. Base metals in each material type were analysed by variants of appropriate ICP-OES methods. Carbon and sulfur were determined by combustion method (LECO) and phosphorus by colorimetric method.

Results and Discussion

The furnace was operated for 7 days with an overall throughput of 600 kg concentrate per day, and processed 4197 kg concentrate, 933 kg Sascarb and 767 kg limestone to produce 1959 kg crude pig iron (47 % of concentrate), 1127 kg slag (27 % of concentrate), and 243 kg dust (4 % of total feed). The weighted average composition of pig iron, slag and dust generated in the optimum furnace operating condition is given in Table 7. This condition made use of 24 % C addition and obtained a pig iron grade of 94.1 % Fe with 2.73 % FeO remaining in slag. The grade of pig iron produced in the pilot tests was similar to that obtained in the preliminary crucible tests (93 % Fe).

Operating conditions with lower C additions were characterised by higher FeO concentrations in slag. Higher C additions achieved Fe extractions as high as 98 % as well as higher C and lower S in metal, associated with lower FeO in slag. Figure 5(a) illustrates relationship between C in metal and FeO in slag and Figure 5(b) shows similar curve for C and S in metal. High Fe extractions corresponded with slags that showed unfavourable properties. These slags were slightly viscous as evidenced by increased metal entrainment in slag. Low concentrations of carbon in metal yielded metals with higher liquidus temperatures that presented tapping difficulties. It is evident that optimisation of C and flux addition is crucial to ensure acceptable Fe extractions as well as promote slags with favourable properties.

Phosphorus and carbon predominantly reported to the metal during smelting and constituted the major impurities in the crude pig iron. The metal content of C was a strong function of C addition but P on the other hand showed a weak response to the different levels of C additions (see Figure 5(c)). Other impurities like Mn, Si, and Cr were low and consistent throughout the campaign (see Figure 5(d)).

The smelting campaign achieved an overall Fe recovery of approximately 90 % with a corresponding FeO concentration of 7.4 % in the slag. The individual smelting conditions however demonstrated that higher Fe recoveries are achievable. The smelting process had an overall energy requirement of 2.88 kWh/kg of metal produced, which was well in agreement with the theoretically estimated average SER of 2.5 kWh/kg of metal. The calculated SER for each tap ranged between 2.55-3.9 kWh/kg of metal with furnace heat losses averaging 165 kW.

Table 7 - Weighted average composition of pig iron, slag and dust from optimum furnace operating condition, mass %

Pig iron	Al	Si	Ca	Mn	Fe	C	P	S	Total
	0.03	0.03	0.11	0.16	94.1	4.62	0.90	0.02	100
Slag	Al ₂ O ₃	SiO ₂	CaO	MnO	FeO	C	P ₂ O ₅	S	Total
	49.2	18.6	27.0	0.12	2.73	0.03	0.21	0.03	98
Dust	Al ₂ O ₃	SiO ₂	CaO	Mn ₂ O ₃	Fe ₂ O ₃	C	P ₂ O ₅	S	Total
	7.97	17.1	8.13	1.62	54.2	0.37	4.58	0.18	94

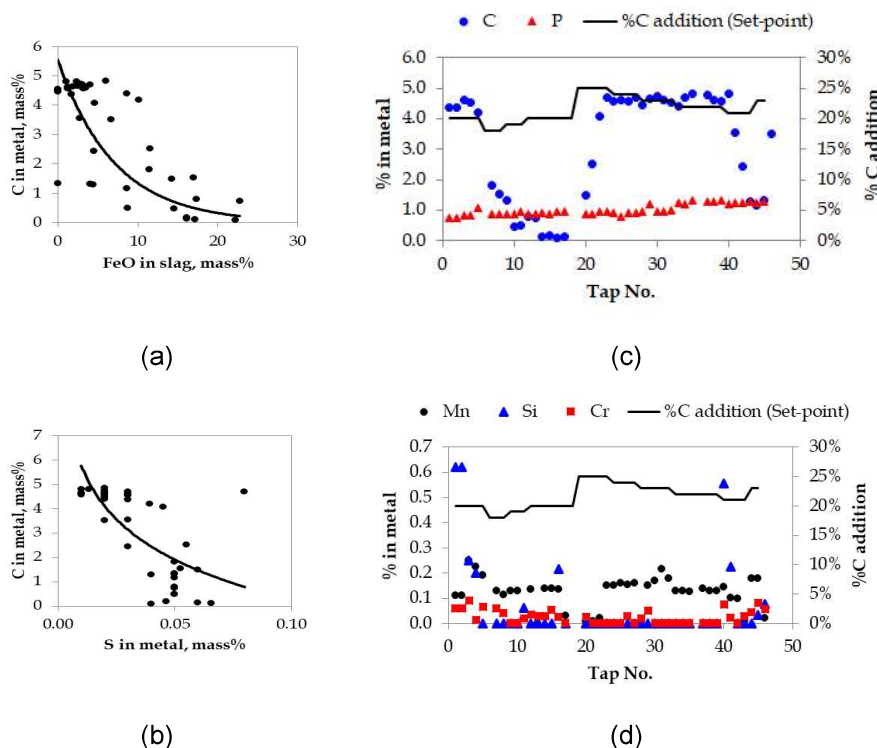


Figure 5 - Graphs showing relationship of (a) C in metal with FeO in slag, (b) C and S in metal, (c)(d) impurities in metal with C addition

PIG IRON REFINING TEST WORK

The primary objective of the refining test work was to demonstrate refining of the crude pig iron on a large crucible scale (10 kg start metal) to produce a crude steel suitable for billet steelmaking. The first stage of the test work was exploratory in nature and sought to establish optimum converting conditions through submerged lance oxygen injection, similar to the preliminary converting tests but on a larger sample size. Converting tests with oxygen injection encountered several technical challenges, relating to the scale of the test set-up, including limited gas pressure, low gas linear velocity, excessive splashing of melt at high gas flow rates, and frequent pre-mature failures of gas lances. Powder refining was proposed as an alternative refining method in which ferric oxide (Fe_2O_3) was added to the molten pig iron as the oxidant. The test work proceeded to explore powder refining with the aim of determining a suitable oxidising and fluxing strategy, operating temperature and total amount of each chemical reagent necessary to bring about the required removal of the impurities. Once the optimum refining conditions were established, 3 x 10 kg of pig iron metals were refined to the billet specification to evaluate the repeatability of the process.

The powder refining tests were preceded by a literature review on refining of pig iron with specific focus on dephosphorisation. It was established from the preliminary converting test work that there are several process parameters that are critical in ensuring effective removal of P from the pig iron. The review of literature on powder refining confirmed the same. The work published by Nakamura et al (1981) investigated the use of CaO-based fluxes containing chlorides and/or fluorides and reported that addition of halide compounds enhanced the rate and degree of P removal. Ohguchi et al (1984) reported that the level of the initial Si content of the pig iron interferes with the rate and degree of dephosphorisation achievable. A patent published by Sasaki et al. (United States Patent No. 756,968, 1977) stated the importance of lower reaction temperatures for improved dephosphorisation. Arguing that dephosphorisation can be effectively carried out at temperatures less than 1450°C with high dephosphorisation attained at 1300°C. The patent further emphasized that reaction temperatures higher than 1450°C do not allow for effective dephosphorisation.

Ikeda and Matsuo (1982) studied the effect of slag basicity on dephosphorisation of molten pig iron with 0.12 % P. It was found that metal having P content of less than 0.005 % can be easily obtained by a slag with a basicity ($\text{CaO}:\text{SiO}_2$) of 2.0-3.0 at 1300-1325°C along with addition of CaF_2 . Ikeda and Matsuo's work also investigated the effect of FeO content of slag on dephosphorisation reaction and found that the higher the FeO content of slag (>15 % FeO) the higher the degree of dephosphorisation. As such, based on the discussion above, the process parameters that are critical for attaining high degrees of dephosphorisation from molten pig iron can be summarized as:

- Use of CaO-based fluxes containing chlorides and/or fluorides (CaCl_2 , CaF_2)
- Level of initial Si content of the pig iron (<0.05 % Si)
- Lower reaction temperatures (<1450°C)
- Slag basicity ($\text{CaO}:\text{SiO}_2 > 2.0$)
- FeO content of slag (>15 % FeO)

Use of chloride-based reagents is discouraged because chlorides are associated with the release of Cl_2 gas which poses serious health risks. As such, the refining test work made use of CaF_2 . The use of CaF_2 as flux has been demonstrated (Sarkar, 2018) to be more effective at reducing viscosity of basic slags than that of acidic slags. The Si content of the crude pig iron produced in the pilot smelting test work was very low (<0.05 %). Therefore, interference of dephosphorisation reaction by Si was not expected. The first set of powder refining tests made use of high reaction temperatures (>1500°C) which proved futile for dephosphorisation. Lower reaction temperatures (<1450°C) were applied in subsequent tests as well as slags with basicity values > 2.0. All slag products obtained from the tests had FeO contents well above 15 %.

What was of concern was the high P (>1.0 %) content of the pig iron obtained relative to P content of typical pig iron produced in the iron and steelmaking industry (<0.15 % P). This suggested that fluxing requirements for refining this pig iron could be much higher than typical quantities observed in literature, and the slag-to-metal ratios will be higher as well, thus necessitating frequent de-slugging to ensure a slag with a higher P_2O_5 capacity is always in contact with the metal. De-slugging

will also avoid flooding the crucible with slag. The frequent removal of slag was evidenced in literature where it was reported that metals containing high content of P require more than one slag to dephosphorise the metal to the desired level (Sarkar, 2018).

Experimental Procedure

Ingots of crude pig iron from the pilot smelting campaign were selected and prepared for the test work. Ingots with high C content (>4 %) were particularly selected because the high C content renders the alloy brittle and thus amenable to size reduction by crushing. The average chemical composition of the selected pig iron ingots was 93.7 % Fe, 4.55 % C, 1.09 % P, 0.13 % Mn with Si, Cr and S <0.05 %. The experimental procedure described here is that of the repeatability tests after the optimum refining conditions were established.

For each test, approximately 10 kg of pig iron was packed in a 175 mm ID x 300 mm cast magnesia-chrome (68 % MgO, 15 % Cr₂O₃, 9 % Fe₂O₃) crucible. The crucible was fixed inside an induction coil furnace with a rammable MgO refractory material. The alloy was slowly heated to a reaction temperature of 1250-1300°C under inert Ar atmosphere where a completely molten bath of metal was obtained. Reagents consisting of 100 g Fe₂O₃, 100 g CaF₂, 180 g CaCO₃ and 20 g SiO₂ were mixed and slowly added into the crucible using a hand-held funnel. Addition of the reagent mix was characterised by violent reaction of the bath signifying release of process gases (predominantly CO gas) and the exothermic nature of oxidation reactions. Metal pin samples were withdrawn from the melt with a quartz tube after addition of every flux mix (after bath settled). The reaction temperature was measured with an optical pyrometer and maintained between 1300-1350°C during the early stages of the refining test.

Total reagent masses of up to 1850 g Fe₂O₃, 1350 g CaF₂, 2650 g CaCO₃ and 300 g SiO₂ were added during the tests. The oxidant and flux additions generated large quantities of slag that required regular de-slugging to avoid filling the crucible with slag. The reaction temperature was slightly increased to 1400-1450°C in the later stages of the test in order to keep the slag and metal in a molten and fluid state.

After completion of the test, the final metal was tapped into small fireclay crucibles by tilting the furnace and allowed to cool. The tapped metal ingots were polished and analysed by spark spectrometry. Slag samples were collected, prepared and analysed for base metals by ICP-OES. Carbon and sulfur were determined by combustion method (LECO) and phosphorus by colorimetric method.

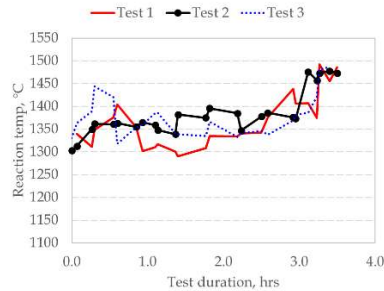
Results and Discussion

The reagent consumption and chemical composition of the crude steel metal and slag from the repeatability tests are shown in Table 8. The reaction temperatures maintained in each test are depicted graphically in Figure 6(a). The change in the concentration of C and P in the metal during the refining test is illustrated in Figure 6 (b), while the change in S and Fe is depicted in Figure 9(c). The behaviour of Cr, Cu and V is depicted in Figure 9(d).

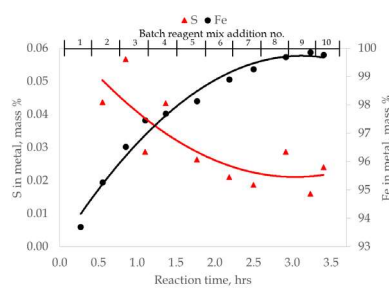
The results provided clear evidence that the established optimum refining conditions were repeatable. The three repeatability tests all made use of calcium fluoride as a fluxing reagent, applied the same reagent addition ratios of Fe₂O₃:CaF₂:CaO:SiO₂ = 1:1:1:0.2 and similar reagent additions per ton of start metal, and maintained similar average reaction temperatures between 1300-1400°C. All the slags produced from the tests consistently had similar chemical compositions with basicity values (CaO:SiO₂) between 2 - 3. None of the crucibles showed signs of excessive chemical attack by the slag. A small amount of MgO was absorbed by the slag as evidenced in the composition of the slags. And lastly, all three final metals of crude steel produced from the tests met the billet steel specification with the best final crude steel reported with a content of 0.005 % P.

Table 8 - Reagent consumption and composition of metal and slag from repeatability tests, mass %

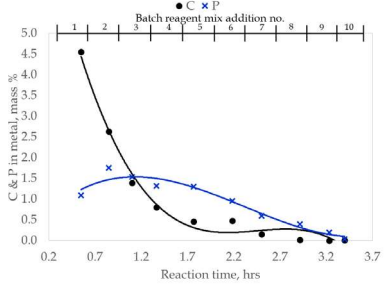
	Start alloy, g	Consumption, kg/ton start metal				Temp °C	Test time, hr			
		Fe ₂ O ₃	CaF ₂	CaCO ₃	SiO ₂					
Test 1	10.5	157	157	280	31	1364	03:30			
Test 2	10.6	156	156	279	31	1386	03:41			
Test 3	10.9	151	151	270	30	1375	03:47			
Metal	Fe	P	S	C	Si	Mn	Total			
Test 1	99.77	0.005	0.024	<0.0015	<0.0015	<0.001	99.8			
Test 2	99.70	0.005	0.025	0.015	<0.0015	0.001	99.7			
Test 3	99.60	0.021	0.015	0.027	<0.0015	0.008	100			
Billet steel	>99.00	<0.045	<0.045	<0.310	<0.300	<1.100				
Slag	Al ₂ O ₃	CaO	FeO	MgO	SiO ₂	P ₂ O ₅	S	CaF ₂	Total	CaO/SiO ₂
Test 1	0.78	23.1	28.4	4.02	9.73	4.30	0.18	29.3	99.9	2.37
Test 2	0.86	25.9	25.1	4.43	10.0	3.78	0.20	28.2	98.5	2.58
Test 3	0.72	27.6	32.8	3.39	9.23	3.59	0.21	22.7	100	3.00



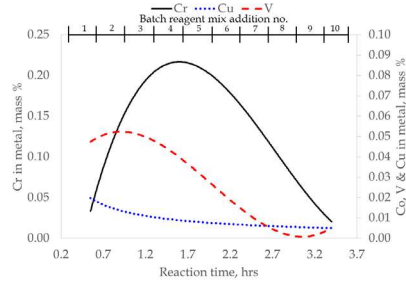
(a)



(c)



(b)



(d)

Figure 6 - Profile curves for (a) test reaction temperatures, (b) change in C & P in metal, (c) change in S & Fe in metal, and (d) change in Cr, Cu & V in metal

The curves showing change in concentration of impurities indicate that C and S were the first elements to immediately drop in concentration within the first hour of refining. Sulfur continued to drop gradually to 0.024 % towards the later stages of refining while C rapidly dropped below 0.5 % after 1.7 hours of refining. Chromium experienced a sharp increase in concentration in the early stages of refining followed by a sharp drop after 1.7 hours (after 4 reagent mix additions) to a concentration of 0.053 % at end of the test. The Fe concentration had a steady and gradual increase from 93.7 % to 99.77 % throughout the refining test. The concentration of P briefly increased in the first hour of refining followed by a gradual decrease throughout the duration of the test to a low of 0.005 %.

CONCLUSIONS

The Agbaja mineral resource is a unique sedimentary hosted magnetite deposit with a resource grade averaging 41.3% Fe. The ore is relatively soft and friable and the primary mineral comprised goethite with oolitic formations. Magnetite was common throughout the unweathered and weathered sections. The presence of phosphorous is assumed to be in a form of cellopene within less weathered lithologies. In the weathered oolites, phosphate was commonly present as cavity filling. Phosphorus bearing minerals include apatite and a crandallite group minerals. Other less common mineral components include pyrite, chlorite and quartz.

Previous development work on the Agbaja ore assessed conventional mineral processing techniques like magnetic separation to produce a concentrate with low levels of phosphorous, but these techniques were unsuccessful. Attempts to produce marketable concentrates using unconventional processes was proven technically viable using a bioleaching process. However, the novel bioleaching method is unproven at commercial scale and was considered to have too high a risk for successful project development. Alternative processing scenarios based on metal production for consumption by the local industry was considered a better option.

Preliminary investigations to produce billet steel from the Agbaja ore were carried out on a laboratory scale at Mintek in 2016. The test work successfully demonstrated pre-reduction of the ore and smelting to produce a pig iron with 93 % Fe. The pig iron was further refined to produce a crude steel with 0.039 % P. It was therefore recommended to demonstrate these processes on a larger scale.

Beneficiation test work investigated comminution and scrubbing of the ore to upgrade the iron content by rejecting a slime fraction high in silica. Conventional scrubbing tests were conducted at Mintek and comparative high pressure scrubbing test work was conducted in Australia. The conventional scrubbing tests showed that scrubbing at -30 mm and screening out the -425 µm size fraction helps remove a larger fraction of the gangue and clay minerals. The high pressure scrubbing tests proved to be very efficient in removing silica but it also discards a high proportion of Fe to the slimes fraction. These high Fe losses can be attributed to the ore being friable and easily disintegrate when subjected to the high-pressure washing. It was thus concluded that conventional scrubbing using a rotary scrubber would be adequate and appropriate for the Agbaja ore.

In the proposed flowsheet the beneficiated ore goes through a pre-reduction stage before smelting to produce a high phosphorus pig iron. Pre-reduction is necessary to reduce the electrical energy consumption during smelting. The pre-reduction test work evaluated two locally available reductants, namely, black and yellow coal. The tests found that the black coal consistently produced better metallisation compared to the yellow coal. The optimal pre-reduction temperature was determined to be between 1100 and 1150°C. The test work showed that metallisation increases with decreasing coal and ore particle size, and the highest metallisation of 91.8% was achieved with 1 to 3 mm coal and 5 to 10 mm ore sizes.

The pilot smelting test work successfully demonstrated that it is technically feasible to process beneficiated Agbaja iron ore in an electric arc furnace to produce a crude pig iron metal. The smelting campaign achieved an overall iron recovery of approximately 90 % with a corresponding FeO concentration of 7.4 % in the slag. The individual smelting conditions however demonstrated that iron recoveries higher than 98 % are achievable. Phosphorus and carbon predominantly reported to the alloy during smelting and constituted the major impurities in the crude pig iron. The smelting process had an overall energy requirement of 2.88 kWh/kg of metal with the calculated SER for each tap ranging between 2.55-3.9 kWh/kg of metal.

Refining of the crude pig iron to a crude steel suitable for billet steel was successfully demonstrated through powder refining on a large crucible scale (10 kg start metal). The pig iron was refined at temperatures between 1300-1400°C with reagent additions of 157 kg Fe₂O₃: 157 kg CaF₂: 280 kg CaCO₃: 31 kg SiO₂ per ton of start metal to produce a final crude steel with a phosphorus content of 0.005% P.

This test work has technically demonstrated the ability to produce a steel product within industry acceptable specifications, particularly phosphorous. Conventional ore beneficiation processes combined with rotary kiln pre-reduction, generate a suitable product for smelting via electric arc furnaces producing pig iron. Refining of this pig iron targets impurity removal and has been

successfully demonstrated on Agbaja ore. Optimisation and improvements to unit operation efficiencies along with additional engineering feasibility assessments are necessary as the next steps in the development of this project.

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