Black sand and iron stone: iron smelting in Modakeke, Ife, south western Nigeria

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Abstract
Fourteen fayalitic bloomery slags, possible ore and furnace wall samples from an ancient smelting site in southwestern Nigeria were analysed by chemical and mineralogical techniques. They derive from a skillful bloomery smelting operation extracting the maximum possible amount of iron from the ore. For most of the slags the analyses show an exceptionally rich level of titanium oxide (up to around ten wt%), indicating the use of a mixed ore comprising limonite iron stone and ilmenite-rich black sand. Some of the slags were tapped, while others may have solidified within the cooling furnace, as indicated by their morphology and mineralogical texture.

Introduction
The Yoruba, whose homeland covers the eastern part of the Republic of Benin and a large portion of south western Nigeria (Fig. 1), have been a centre of rich cultural heritage, which has been appreciated by many scholars. Their forefathers were basically farmers and hunters. Therefore they needed to manufacture implements for their farming and hunting as well as weapons for defensive and offensive purposes. Most of the insignia of their office and symbols of their gods were made from iron.

Generations of the Yorubas have smelted iron and the skill has been taught from one generation to another. Iron-working in Yoruba has been the pre-eminent transformative process, a technology greedily sought and jealously guarded, for its control could promote a king’s ambition and a soldier’s fortune. Iron-smelting technology has often been considered divine inspiration brought to humans by culture heroes. Sacred Yoruba kings were sometimes renowned as smelters and blacksmiths. In other circumstances, the transformative powers of iron-workers are deemed so great that smelters and blacksmiths are thought dangerous and avoided by ordinary people (Adepegba 1991).

The widespread occurrence of remains of the bloomery process - mining pits and tunnels, small parts of technical ceramic including furnace shafts and tuyeres, traces of charcoal, as well as copious amounts of smelting slags - provide a rich materials base for reconstructing the technology used by the ancient Yoruba smelters. Excavations at several locations in the Ife area have revealed evidence of Late Iron Age iron smelting in the Yoruba area of southwestern Nigeria. In many areas, layers of smelting debris, such as ash, charcoal, slag, and pieces of possible tuyeres was found together with fragments of mud-brick walls associated with what may be the collapsed remains of furnaces.

Historical background
Ife-Ife is regarded by all Yoruba as their immediate origin. From there, their ancestors dispersed to establish towns in their present homeland in West Africa. Ife was a centre of the iron manufacture, though chiefly of small wares, such as nails, horse-shoes, keys, locks, and common agricultural tools; and it was estimated that there were about 500 iron smelters, smiths and other workers in iron of various kinds living within a radius of about twenty kilometers (Adeniji 1977).

From the history and art of the Yorubas it is certain that the knowledge of mining and metallurgy was prevalent among the royalty. As they spread across the land they founded kingdoms and empires, and iron mining and smelting continued because of the need to acquire weapons to fight and conquer more lands. The oldest and the most powerful of these kingdoms is the Old Oyo. However the northward growth of the Yoruba kingdom (see Fig 1) was halted by the Fulani jihadists. No sooner had the Fulani jihadists smashed Old Oyo, in about 1830, there was a decline in iron production. The Old Oyo empire suffered the greatest collapse that drove the people back to their original homelands in the Ife area. As they migrated homewards they continued to spread the art of smelting especially in towns like Igbaja, Oyo, Isundunrin and back to Ife. Among the returning soldiers and smelters were those who had carried the technology of iron smelting with them; upon their return to the Ife area, they separated themselves from those who had stayed, and formed a separate settled area within the confines of Ife, called Modakeke. It is from this area that the slag samples studied here were taken.
It is not clear what eventually stopped the smelting operation in the region. Evidences gathered suggest the instability caused by intertribal wars, the appearance of European scrap iron, and perhaps the agrarian revolution that altered the commercial leaning of the local people, or a combination of all this factors. Lack of raw materials can be ruled out as there is still an abundance of various grades of lateritic ironstone in almost all known Yoruba towns. However, as reported by other authors (eg Miller 1995), local iron production in many African countries declined sharply with European colonization as imported iron assumed increasing economic importance.

Site location
The site for this study is located near a disputed boundary between the Modakeke and Ile communities in the ancient town of Ile-Ife. Geologically it is situated within the basement complex of Nigeria which consist of early Proterozoic (2200 my) gneisses and schists. The schists consists of mica-garnet bearing rocks intimately associated with mafic to ultramafic rocks. These rocks have been extensively studied and characterized by several authors (e.g. Ige & Asubiojo 1991; Ige et al. 1998). The greenstones which are the parent rocks for the lateritic ores occur as lenses within the polydeformed migmatite-gneiss complex. Several outcrops occur along the 800 km-long greenstone belt of Nigeria. Their mineralogy consists mainly of iron-magnesium amphiboles and ore minerals such as spinel, pyroxhinite, and pyritite. The area is located within the tropical rain forest with high temperature and high humidity. A distinct characteristic of the area is the abundance of thick lateritic ironstones formed from the in situ weathering of high Fe-Mg ultramafic rocks.

The mining site is called Ereta, meaning the area where bullet-like ore nodules are found. The smelting site is about one to one and a half kilometers away from the mining site. The mine consists of a large open pit grading into tunnels and underground mines. That there are several underground mines is evidenced in the collapsed structures built above these tunnels, which can be seen in several places in Eleta. The most prevalent ore material is an aggregate of limonite and goethite derived in situ from greenstones.

These ores are abundant in almost all Yoruba towns and provided the needed raw materials for the operation of hundreds of mines and iron smelting centres. In other areas of Yoruba land where smelting was still in operation up until 1970, Adeniji (1977) and other oral evidences available described the major type of ore used is gravel-stone known as oko or eta stone (lateritic ironstones) derived from the breakdown of ultramafic amphibole rocks which are prevalent in the area. The oko stone is rich in siderite (iron carbonate), goethite (iron hydroxide) and limonite. According to oral tradition, black sand is washed from the soil during heavy rainfalls and accumulates in pockets and gutters, from where it is easily collected, and was also added to the ore. A preliminary sample of this black sand was studied and found to be rich in ilmenite and magnetite. The combination of these two iron ores, the lateritic stones and the sand, provides the furnace charge, providing the iron mineral for reduction to metallic iron as well as fluxes that will allow the formation of a good slag during melting in the furnace.

Mining methods
According to oral evidence and field observations, the iron smelters were prospecting for the raw materials by following the presence of iron stones on the surface; they then deduced that more iron ore abounds under ground. They then mined the ore using two different methods

1. Open pit grading into underground or tunnel when the ironstone is deposited vertically in the ground.
2. Trenches and channel mining if the iron-stone lies horizontally in the ground.

When the ore has been mined, it is sorted into smaller pieces of gravel size and larger lumps and blocks, which are crushed to a coarse sand or gravel fraction. The ore is then carried to a stream for washing, or water is fetched from the surrounding streams. The ore is poured into baskets in small quantities and immersed in the stream, or water is poured over it and shaken repeatedly until the dirt which is sticking to the ore is washed away from it completely. Then a coarse mat is spread on the ground and the washed ore is poured on it and left there until it is bone-dry.

The following are the equipment used in preparatory work before the iron-stones can be collected: Cutlasses for clearing the bush; pickaxes for digging up the soil or digging out any roots that may hinder mining operations; heavy picks for excavation or for digging up the ground, hoes for gathering the earth and for removing the earth; baskets and small, light calabashes for removing the earth from the pit; ladders for descending into and for climbing out from inside the pit and lamps, to illuminate the underground tunnels and different kinds of food which miners consume during breaks and may eat from morning to evening.

Iron smelting
Archaeological and ethno-archeological investigations in Modakeke have helped in the understanding of the early smelting in the community; these investigations were made possible by leaders of the town who had assisted their parents in the iron smelting. These people were persuaded to re-enact the technique for recording at the Natural History Museum, Obafemi Awolowo University, Ile-Ife, Nigeria.

Ethnographic observations
The re-enacted furnace was about 1.7 m high with constricted top which was provided with openings through which the ore is charged. The type of furnace used is a shaft furnace operated without any bellow. This furnace takes advantage of the buoyancy of hot air. The rising of this air results in a low pressure in the furnace, which in turn causes more air to be drawn in through the tuyere below, creating a natural draft with no needs to operate bellows.

Charcoal is used for smelting and it is produced from special hard wood which are distinguished by their poisonous characteristics. The most important of them is the sapwood tree (Erythrophleum Guineense) whose other name is obo. This produces hard charcoal with high combustible power.

To produce the charcoal the wood is cut into short logs and arranged in a trench about two feet deep. The branches from the trunks are sandwiched between these logs leaving small gaps between them. The space between the logs and the branches are filled with reeds and dry reed grass. The fire is then lit. Whilst burning is going on, fresh palm fronds are used to cover it, together with a layer of dry, dusty earth. Then water is mixed with dry earth until it is thoroughly wet, and this is then heaped on the dry earth to cover it.
After this, another complete layer of dry earth is added. The fire is left burning for complete three full days and nights before a section of it is uncovered to find out how well it has burned to form charcoal. In case of partial burning the remaining logs are set on fire again and covered with earth. All the charcoal which can be got from that hearth is collected for use for the smelting furnace.

The raw materials were transported for about one and a half kilometers from the mining sites to the smelting site. Much of the movement of these raw materials was done by women and children. The raw materials were charged into the ‘tunnel mouth’, i.e. the top of the furnace. There could be, for example, 6 baskets of gravel-size iron ore, 10 baskets of charcoal and 2 baskets of the crushed ore. Ilmenite-rich sand is also added to the ore. Exactly how much charcoal, iron ore and sand are needed to be added was governed by the skill and experience of the furnace tender who worked at the base of the furnace.

Ritual and taboo
Of course, the ancients did not understand the chemistry of burning or the physics involved in the incandescence of hot gas molecules. They were able to judge the temperature by the colour of the flames and the hot metal, but did not understand why one kind of sword needed to be thrown into a tank of water to harden it, and the other needed only to be waved around vigorously in the air. What they did learn to do was to discover elaborate rituals which maintained the procedures which controlled the carbon content of the smelted bloom, the temperature to which the finished work had to be reheated, and the rate at which it was cooled during the quenching process. Smelters and blacksmiths consider the strict observance of taboos as necessary to the successful outcome of a smelt. These taboos have been instituted long ago by the ancestral smelters to ensure success by keeping the whole process ritually pure and free from pollution or change. The first taboo is that a libation must be poured to appease ogun, the god of iron. The ogun shrine is always located at the entrance to the mining and smelting sites where the smelters worship before and after the daily operation. The offerings or sacrifices to the god of Ogun must be performed before mining for ironstones commences. This includes the following: palm-wine, which is poured on the mining site; life snails to be broken on the precise spot; and kola nuts, which are used to propitiate Ogun at this very place. These appeasements are necessary so that the miners may strike a good iron deposit and to guard against accidents.

There are also taboos that must be observed by iron miners and smelters. Among these are freedom of mind from any bitterness and must have no ill-will towards anybody; must not commit adultery or theft.

Analytical procedure
Fourteen samples in all (11 slags, 2 ore, 1 furnace wall) have been subjected to microscopic and compositional analysis. The slag samples are here identified by the designation NG for Nigeria, followed by MO2, MO3 and MO4, and IP1 to IP5 for slags from the upper cultural layer (see above). IS1, IS2, IS2d and IS2h are from the middle cultural layer, and MO6 and MO7 are the two ore samples from the lowermost layer. MO1 is a sample from the furnace wall. It was decided to analyse all samples by optical microscopy and by bulk chemical analysis using standard XRF procedures, and to investigate a subset of representative samples using scanning electron microscopy with energy-dispersive analysis. All analyses were performed at the Wolfson Archaeological Science Laboratory, Institute of Archaeology, University College London. Cleaned and cut sections of the samples were crushed and ground to a fine powder in an agate mortar, oven-dried at 105°C over night, and mixed with wax before being pressed into pellets. Bulk chemical analyses were performed by X-ray Fluorescence Spectrometry, using a Spectro Xlab 2000 and evaluating the measured values against certified reference materials for quantitative analysis.

Results
The slags are predominantly composed of iron oxide and silica, in keeping with most bloomery slags analysed so far. Most remarkable, however, is the very high titania content of all of these slags, with six to eleven weight percent, except one which has just under one percent (Table 1, arranged in increasing order of titania content). The concentrations in alumina, lime, phosphorous oxide and manganese oxide are all within the typical range known from other bloomyer sites. Several of the minor and trace elements, however, are present at remarkable concentrations. The zirconia content of most slags analysed here is in the range of between half a percent and one percent, as compared to typical values known from several European bloomery sites of below 400 ppm, and often below 100 ppm (e.g. Yalin & Hauptmann 1995; Ganzelewski 2000). Similarly, there is a very high level of vanadium present, of around 2000 ppm V2O5, as compared to published values elsewhere of typically less than 200 ppm V. Strontium and the Rare Earth Elements are apparently also enriched.

The slag IS 2
Only one of the samples of slag from the middle layer of the excavation is very different from the others. While IS1 is almost indistinguishable from the other slag samples, IS2 has less than one percent titania, and the highest level of iron oxide of all slags analysed, with around 67 percent. Furthermore, the phosphorous level is much lower than with the other slags (0.3 wt% P2O5 as compared to an average of 0.9 wt%), as are the levels of vanadium, manganese, zirconium, and lanthanum and cerium (the latter two not reported in the table). From this, it appears that a very different ore was used for the smelt which produced this particular slag. A subsequent screening of more slag samples from this particular layer, using XRF to analyse the cut surfaces of a further eight samples, indicated that all of them belonged to the titania-rich group, indicating that the sample IS2 is an outlier among the assemblage.

Ore samples MO6 and 7
These two samples were massive and reddish to brownish in colour, formed in situ from the weathering of the parent rock. One is a piece of the gravel-sized ore, while the other is a large lump which would have been crushed to gravel size before being fed into the furnace. According to microscope analysis, they both consist of an aggregation of goethite and limonite in a groundmass of decomposed anthophyllite and chlorite. The angularity of the quartz fragments indicates that the quartz was not transported geologically over a long distance, i.e. that the ore formed locally. In their chemical composition, with silica, alumina and phosphorous oxide, they resemble self fluxing ores. Most important, however, is their very high percentage of iron oxide, of up to 80 percent, and accordingly low amounts of slag-forming components. Significantly, they have only very little titania, of less than half a percent only, and low levels of zirconia and vanadia as well. Overall, the two samples are mineralogically and chemically similar enough to group them as one type of ore.
Optical metallographic studies show that the main phase present in the slags is light grey fayalite in a dark glassy matrix. The glass represents the reservoir for CaO, MgO, K₂O, P₂O₅ and Al₂O₃. According to the SEM-EDX analyses, the fayalite is relatively pure Fe₂SiO₄ with only limited substitution of manganese and calcium for iron. There is a characteristic skeletal and elongated structure of fayalite, indicating rapid cooling. This is in good agreement with their shape, indicating flow pattern typical for tap slags. All samples show an abundance of an opaque cubic phase, often with skeletal growth pattern, identified by SEM-EDX as ulvite spinel (Fig. 2; Fe₂TiO₄). This mirrors the high titania content of these samples as identified by XRF analysis. Most notably, there is a marked absence of wuestite in the slags, indicating the high efficiency of the furnace. According to Tylecote et al. (1971) the content of wuestite in the bloomery slag provides significant clues towards the estimation of the metallurgical success of the ancient smelters. Slags with little or no wuestite indicate a more efficient process than those with higher proportions.

Furnace lining
Sample MO1 is from the furnace lining. The quartz which was originally rounded is now completely shattered as a result of intermittent heating and cooling and perhaps, the chemical attack by the melt. Chemically, the furnace lining is remarkably rich in iron oxide (27 wt%) when viewed as a ceramic material. Similarly, its level of titania (just under three weight percent), zirconia and rare earths elements is much closer to the dominant slag group than to the limonitic ore samples. This probably reflects the composition of the black sand, which is reportedly washed from the soil during heavy rain falls; the local earth, being used for the building of the furnace, will contain a significant amount of such heavy minerals.

Discussion
Apart from oral evidence, scattered excavations and ethnographic data, not much has been known previously about the technological process of iron smelting in south-west Nigeria. However, extensive work has been done on the archaeometallurgy of slag samples MO2, 3 and 4, and IP1-5

Mineralogy of slag samples MO2, 3 and 4, and IP1-5
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NG-IS1 and 2
NG-IS1 and 2 are slag samples from the inner part of the furnace. One of them forms part of the titania-rich group, the other (IS2) does not (see above). Mineralogically, they consist of fayalite, glass, and spinel. The fayalite crystals are bulky and show evidence of slow cooling. While IS1 has the ulvite spinel typical of these slags, the titania-poor slag sample IS2 shows instead a much less reflective spinel (Fig. 3). According to SEM-EDX analysis, this is a solid solution between magnetite (Fe₃O₄) and hercynite (~Fe₆Al₂O₄) with roughly equal numbers of iron and aluminium atoms in the core of the crystals, and up to three times as many iron atoms in the outer rims. This marked zoning with an alumina-rich core and a more iron-rich rim is typical for hercynitic spinel in bloomery slags. In larger crystals, there is an epitactic growth of ulvite around the spinel, indicating much increased titania concentrations in the residual melt when cooling and crystallisation had much progressed. Smaller hercynitic crystals are found as inclusions in fayalite, showing less evidence of coring and no epitactic growth of ulvite. Still, there is the absence of wuestite in this sample which has been suggested as evidence for a high efficiency of the furnace.

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of the Nsukka, about 250 km to the east (Okafor 1992), and at Taruga about 600 km northeast of the study area (Tylecote 1975).

Our own work aims in the long run to contribute to filling the gap, to then enable comparative studies of the skills and methods used during Late Iron Age iron smelting.

The analytical study of the Modakeke slag and ore samples demonstrates a high level of skill of the ancient iron smelters, in that they left no free iron oxide (i.e. wuestite) in the slag. This shows that they exploited all the iron metal from the ore that was possibly extractable using the bloomer process, producing a rather lean slag with an average only 50 percent iron oxide. In addition, the chemical data confirms the regular use of a blended ore for the smelting operation. The high titania content of around ten weight percent of most slags is most likely due to the addition of ilmenitic black sand to an otherwise goethite or limonite rich ore. The two ore samples of this latter type are very low in titania, and could not possibly have resulted in the production of the slag found. Ilmenite (FeTiO₃), which has around 50 wt% titania, will have been the major source of the titania in the slag to account for the current composition. It is impossible to estimate from the current data the relative proportions of black sand and limonitic ore used in the smelting, because we have no reliable data for the average composition of the ore, or the black sand. The ratio of silica to alumina in the slag, however, is higher than in the two ore samples, indicating that at least a certain amount of the silica in the slag was derived from the black sand, likely as quartz sand contamination. Similarly, the higher concentration of phosphorous oxide in the titania-rich slags as compared to the titania-poor sample IS2 indicates that phosphorous may have been introduced together with the black sand, but probably not as apatite since the calcium oxide levels are on average only slightly higher.
than the phosphorous oxide levels. More likely, the black sand contained a certain amount of monazite and similar minerals, since also the level of rare earths elements is much higher in these slags than in the ore, or the slag IS2. Alternatively, the phosphorous could have come as iron phosphate together with the limonitic ore, in which case the slag IS2 would derive from an altogether different ore. In all of these assumptions, no allowance has been made for any contribution of the furnace wall or the fuel ash, both of which may have contributed considerably to the slag forming (Crew 2000).

Overall, there are apparently several advantages from using a blended ore rather than just one or the other. Pure black sand would probably have been not rich enough in silica to produce a suitable slag in the presence of large amounts of titania. David et al. 1989 report about 15 wt% silicate minerals in magnetite sand smelted by the Mafa in northern Cameroon, but in this process, the single internal tuyere contributed critically to the slag formation. Unfortunately, no chemical analyses are available for the ore and slag from that process. The limonitic ore on the other hand could possibly have been smelted on its own, as is indicated by the slag sample IS2. However, the overall content of iron oxide in that one slag sample is much higher than in the others (67 wt% vs. an average of 58 wt%), suggesting that the mixture did smelt better than the pure ore. It also appears that the black sand was easily obtained, at least after heavy rain fall, and thus preferred over the more tediously mined limonitic iron nodules.

Furthermore, the iron oxide content of the various slag phases does not differ much; fayalite (FeSiO₃), the hercynite spinel (Fe₂(Fe, Al)₂O₄) and ulvite (Fe₂TiO₄) all have between 60 and 70 weight percent iron oxide in the EDX analyses, with ulvite being the lowest in terms of iron oxide content. Thus, allowing the formation of ulvite rather than fayalite through the introduction of ilmenite instead of quartz as a gangue mineral is beneficial for the efficiency of the process; each silicon atom of the silica (SiO₂) will require two iron atoms to form fayalite (Fe₃SiO₄), while each titanium atom in ilmenite (FeTiO₃) needs only one more iron atom to form ulvite (Fe₂TiO₄). In effect, the yield of iron metal increases with increasing formation of ulvite. This is reflected in the composition of the titania-rich slag which is rather lean in terms of bloomery slags. Furthermore, the absence of wuestite indicates that the iron produced in the smelting operation was probably steel and not soft iron.

Conclusion

The iron smelting process at Modakeke in south western Nigeria was based on the smelting of a blended ore, mixed from limonitic ore nodules mined from the subsoil, and black sand gathered from the surface after heavy rain falls. The chemical composition and the mineralogy of the slag samples corroborates this ethnographic information. The yield of the process must have been very high, with an ore grade of around 80 percent iron oxide and a relatively lean slag with less than 60 percent iron oxide. The efficiency of the smelting was accordingly very high, leaving not more iron oxide in the slag than absolutely necessary for slag formation. One may assume that the metal produced under these strongly reducing conditions was probably steel rather than soft iron, allowing for the manufacture of good quality implements and weapons. However, the current conclusions are based on a few initial samples only, and clearly require a much more in-depth study before more general and representative conclusions concerning the state of iron metallurgy in the Ife region can be drawn. Similarly, it is clearly necessary to obtain more archaeological information from the excavated site in order to assess the real age of the smelting site, which at present can only most broadly be placed in the Late Iron Age, probably prior to the colonial period.

References


