The glass crucibles from Ile-Ife, Nigeria

A.B. Babalola[1]*, Th. Rehren[2], A. Ige[3] and S. McIntosh[1]

1 Rice University, Houston Texas; * corresponding author

2 UCL Institute of Archaeology, London, UK; th.rehren@ucl.ac.uk ; College of Humanities and Social Sciences, HBKU, Doha, Qatar.

3 Middlesex County College, Edison, New Jersey

* Visiting Researcher at UCL Qatar, Doha, at the time of preparation of this manuscript. Currently a Fellow at the Department of Anthropology, Harvard University, Cambridge, MA

Abstract

Crucibles to melt glass are very rare in archaeological contexts in sub-Saharan Africa. Recent archaeological excavations at Igbo Olokun, Ile-Ife (Southwest Nigeria) revealed abundant fragments of glass crucibles from 11^{th}-15^{th} century AD deposits, matching the complete and near complete examples earlier reported from Ile-Ife. This paper provides an in-depth examination of these crucible fragments in order to understand the material quality of the crucibles, their typology, and their functions in glass-working. Optical microscopic and SEM/EDS analyses were carried out on ten crucible samples. The composition of ceramic fabrics and the adhering glass are presented and discussed in view of their function. The crucibles were produced from specifically selected highly refractory clay and used for melting glass from its raw materials; colorants were added to the melt in the crucible. The useable capacity of the crucibles varied from 1 to 7 liters, equivalent to about 2.5 to 17.5 kg of finished glass for each crucible. Compositional analysis of a sample of the thousands of glass beads from the excavations indicates that the crucibles were used to melt the glass used in the beads. Archaeological evidence of glass bead making at this scale has not previously been reported from West Africa. The crucibles are unique evidence of indigenous glass-working and probably glassmaking in Sub-Saharan Africa from early through mid-second millennium AD.

(French abstract to follow)

Key words: Glass, crucible, glassmaking, glass-working, Ile-Ife, technical ceramics, melting, Sub-Saharan Africa

1. Introduction

Glass beads play an important role in African societies, mostly for colorful body decoration and ornaments, but they also convey significant socio-economic, political, and ritual meanings (Ogundiran 2002). There is evidence for secondary finishing of glass beads from several places in sub-Saharan Africa beginning as early as the eighth century AD (e.g. Cisse 2010; Cissé et al. 2013; Wood *et al.* 2012), using glass imported either from the Islamic lands north of the Sahara, or from India or Europe via sea-borne trade (Wood *et al.* 2016). In contrast, glass beads found in Ile-Ife, a major Yoruba settlement in southwestern Nigeria (Fig 1), have a unique chemical composition indicative of a local, primary glass making industry in the region (Lankton *et al.* 2006; Freestone 2006; Babalola 2015; Babalola *et al*. 2017). The evidence for local glassmaking is based on two main lines of argument: the unique composition of the glass and the presence of specialized crucibles, each large enough to melt several kilograms of colored glass.

Leo Frobenius (1913[1968]) first documented finds of glass-encrusted crucibles in Ile-Ife over a century ago. Since then glass crucible fragments have been reported from archaeological contexts in Ile-Ife dated to between the 11th and 15th centuries AD (Fagg 1953; Willett 1967; Garlake 1974, 1977; Adeduntan 1985), and as chance discoveries (e.g. during construction, quarrying, or farming (Willett 2004)), or surface observations (Ogunfolakan pers.comm.). The crucibles and related production debris are notably concentrated in the site of Igbo Olokun in the northern section of Ile-Ife (Fig. 2), where the density of these finds suggests the presence of a major glass workshop (Frobenius 1913[1968]; Eluyemi 1987; Willett 2004). The glass crucibles have no parallel elsewhere in sub-Saharan Africa. Compositional analysis of the glass layers within the crucible has shown that it matches the unique composition of glass beads found in and around Ile-Ife and beyond. New analyses expand the data base on the unique glass chemistry of Ile-Ife beads and strongly support the case for an indigenous glass-making tradition distinct from those documented in Europe, the Near East, and India (Lankton *et al.* 2006; Freestone 2006; Babalola 2015; Rehren & Freestone 2015; Babalola *et al*. in press). Study of the Ile-Ife crucibles can provide important insights on this tradition. Prior to the current study, analysis was limited to two unprovenanced Ile-Ife crucibles from the collection of the Natural History Museum, Ile-Ife (Lankton *et al.* 2006). This paper reports the results of analyses on ten crucible fragments, eight of which were recovered from excavations by the first author.

The excavations recovered over 800 crucible fragments, almost 13,000 primarily blue and green glass beads, and several kilograms of glassworking waste (see Babalola *et al*. 2017 for a summary of the project; Babalola *et al*. in press provides detailed description of the glass beads and their chemical characterization). In this paper we describe the crucible fragments from the excavations, and present results of the analysis of ten crucible fragments using Scanning Electron Microscopy (SEM) with Energy Dispersive Spectrometry (EDS).

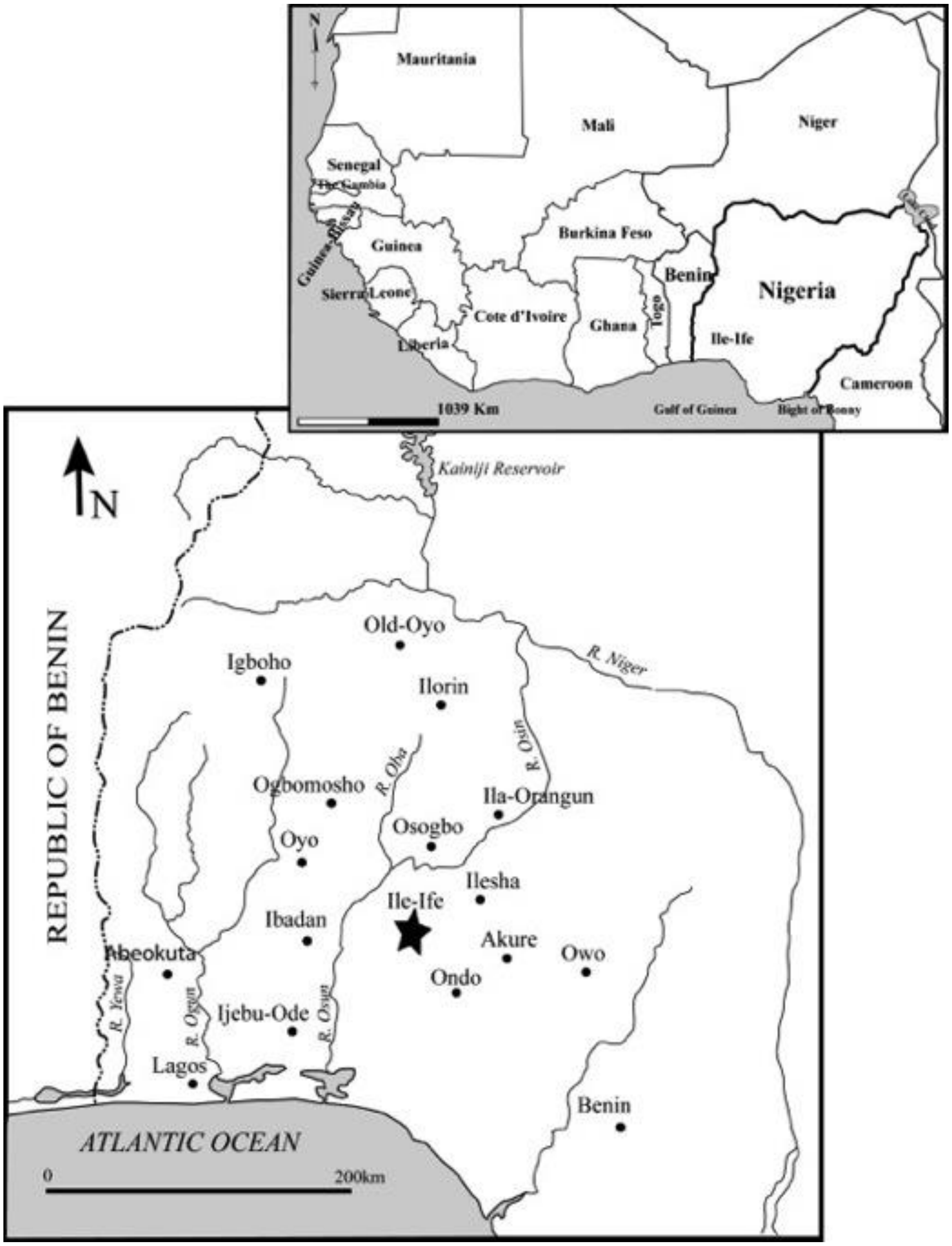


Figure 1. Location of Ile-Ife in southwest Nigeria.

The glass crucibles of Ile-Ife: early descriptions

More than a century ago, Frobenius provided the first description of Ile-Ife crucibles:

> *"Martius [Frobenius' local assistant] picked up a couple of bits ….*
> *wiped the dust off one of them … glaze! We examined the other one –*

glaze again! … the body of them all was a substance like porcelain clay, similar to cement, but they were all coated with glaze of many colors. Afterwards, we found entire jars, with lids belonging to them, glazed both inside and out …" (Frobenius, 1913 [1968]: 93)

Frank Willett (2004) provided photos and dimensions of several complete glass crucibles from Ile-Ife, and an additional two crucibles are on display in the British Museum (Table 1). The rim and base crucible sherds recovered by Babalola are consistent with the forms illustrated by Willett, but none of the excavated fragments was large enough to reconstruct a whole crucible or to estimate its volume. The documented, intact crucibles are barrel-shaped jars with simple inverse rims and flat bases ranging in height from 16 to 35 cm. Measurements taken on the two crucibles in the British Museum were used to estimate missing data from those in Willett's catalogue, in order to gauge the volume and weight of glass processed in each crucible. For this rough estimate, we calculated an average of the diameters along the body of each crucible and used it in the formula for the volume of a cylinder ($\pi r^2 h$). Based on practical considerations, we assume that the effective usable height for molten glass within the crucibles is only about half the measured internal height (for some related discussion, see Merkel and Rehren 2007). The estimated volume was then used to estimate the weight of glass produced, based on the assumption that 1liter of glass weighs approximately 2.5 kg.

Site	ID #	Inventory #	Max Height. cm	Max. Diameter. cm	Shape	Inner glass	Estimated Volume	Additional note
?	BM 1	63.1.15	29	20	Elongated-oval	Light blue	2 liters	Heavy fuel ash glaze
?	BM 2	?	16	13	Barrel	Dark Red	1 liter	Moderate fuel ash glaze around the bottom, Presence of darker clay layering the profile
Ita Yemoo	T828	?	16.5	13.5	Barrel	?	1 liter	Thin fuel ash layer
Itajero	T829	1963.1.14	33	27	Conical-oval	Dark Red	5 liters	Heavy fuel ash, especially at the bottom
Itajero	T830	1963.1.12	31.1	23	Elongated-oval	Pale-green	4 liters	Broken rim
Igbo Olokun	T831	63/45	31.6	23	Oval	Light blue	4 liters	Red glass on the lid, apparent lid ridge
Igbinbin	T832	35/62	18.7	11	Barrel	Red	Half a liter	Thick fuel ash glaze and deep red glass on the exterior
Iremo	T833	66/10	22.2	?	Barrel	Blue	?	Thick fuel ash glaze on the exterior
?	T834	?	18.5	11	Cylindrical	Blue	Half a liter	Heavy fuel ash glaze with a boss of glass on the bottom
Igbo Olokun	T835	IIIC 27550	35.5	30.5	Conical-oval	Green-blue	7 liters	Thick fuel ash glaze on the lower part of the exterior

Table 1: Description of the complete or near complete Ile-Ife crucibles from Willett's (2004) catalogue and British Museum collection (? = no data).

Willett (2004) described the crucible fabric as white or light gray ceramic approximately two centimeters thick. Some vessels have an outer layer of much darker ceramic on the lower part of the crucibles. Most vessels show heavy vitrification on their outer surface, forming an irregular glaze covering the lower half or even higher, with frequent impressions of fuel in the softened outer surface near the bottom. As far as can be ascertained, the inside of all vessels is covered with a thick layer of colored glass. The evidence of smeared glass around the rim indicates that at least some of the crucibles were covered by a lid which was then removed while the vessel was still hot, leading to the pulling-off of the sticky molten glass between the lid and the vessel's mouth. Clearly, these vessels were made for and used in high-temperature conditions well exceeding typical pottery firing temperatures to melt glass.

The 2011-12 Excavations at Ile-Ife

Archaeology and the history of Ile-Ife: a brief overview

Over the last century, numerous archaeological investigations were carried out at Ile-Ife (e.g. Frobenius 1913 [1968]; Fagg 1953; Willett 1960, 1967; Myers 1966; Garlake 1974, 1977; Eyo 1970, 1974a&b; Eluyemi 1975, 1977, 1987), but only a few resulted in detailed site reports. The primary purpose of many of the early excavations was the recovery of brass and terracotta figurines, stone carvings, pottery, and potsherd pavements. In parts of Ile-Ife, house construction, tree plantations, digging for (or looting) ancient artefacts and reburying them for ritual purposes have disturbed the deposits. Despite this, scholars have been able to reconstruct occupational phases for Ile-Ife's periodization (e.g. Fagg and Willett, 1960, Ozanne 1969; Eyo 1974b; Horton, 1979; Drewal 1989; Ogundiran 2003; Blier 2014) suggesting it's apogee to be around 12th century AD.

Prior to the 12th century, Ile-Ife settlement was suggested to be a cluster of agricultural villages (Fagg and Willett 1960, Willett 1967). Between the 10th and 11th centuries the settlements began to coalesce resulting into the emergence of Ile-Ife as a city-state and laid the foundation for the political structure that defined the Yoruba-Edo region (Ogundiran 2003). The 12^{th-}15th century AD was the fluorescence of Ile-Ife's cultural manifestations characterized by proliferation of artworks (Blier 2014), construction of potsherd pavements in compounds and temples (Garlake 1974, 1977; Eyo 1974b; Ogunfolakan 1990, 1994; Ogundiran 2003) and production of glass objects in bracelets and more importantly beads (Willett 1977, 2004; Eluyemi 1987). During this period, Ife reached the peak of its prosperity with a highly centralized political system. The post-15th century AD was marked with the waning of Ife's political and economic influence as well as mass depopulation and decline in production of art works (Ogundiran 2003; Blier 2014). However, the city still maintained its religious and/or ritual significance.

The 2011-2012 excavations

New excavations were conducted in Igbo Olokun (Fig. 2), a once-forested expanse containing a high density of crucible fragments, glass beads, and production debris. Igbo Olokun has been the focus of multiple excavation campaigns for which few details have been published (Frobenius 1913, Fagg 1953; Willett 1967, Eluyemi 1986, 1987). Once an extensive area of forest, banana and palm trees, Igbo Olokun has been progressively built over with residential buildings (Eluyemi 1987). By the time of our excavations, only a small parcel of 21 by 48 meters was enclosed and protected by the Nigerian National Commission for Museums and Monuments (NCMM) for research, education and tourism purposes (Fig. 3). The National Museum Ile-Ife is directly responsible for overseeing the activities at the site.

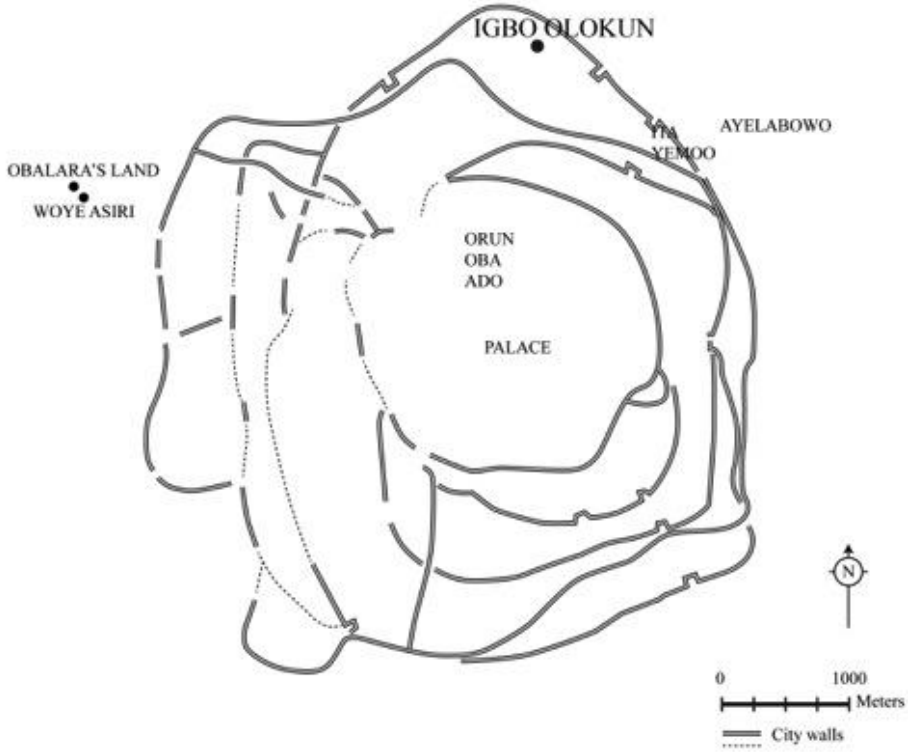


Figure 2. Map of Ile-Ife showing the location of Igbo Olokun and other sites mentioned in the text (modified from Willett 2004).

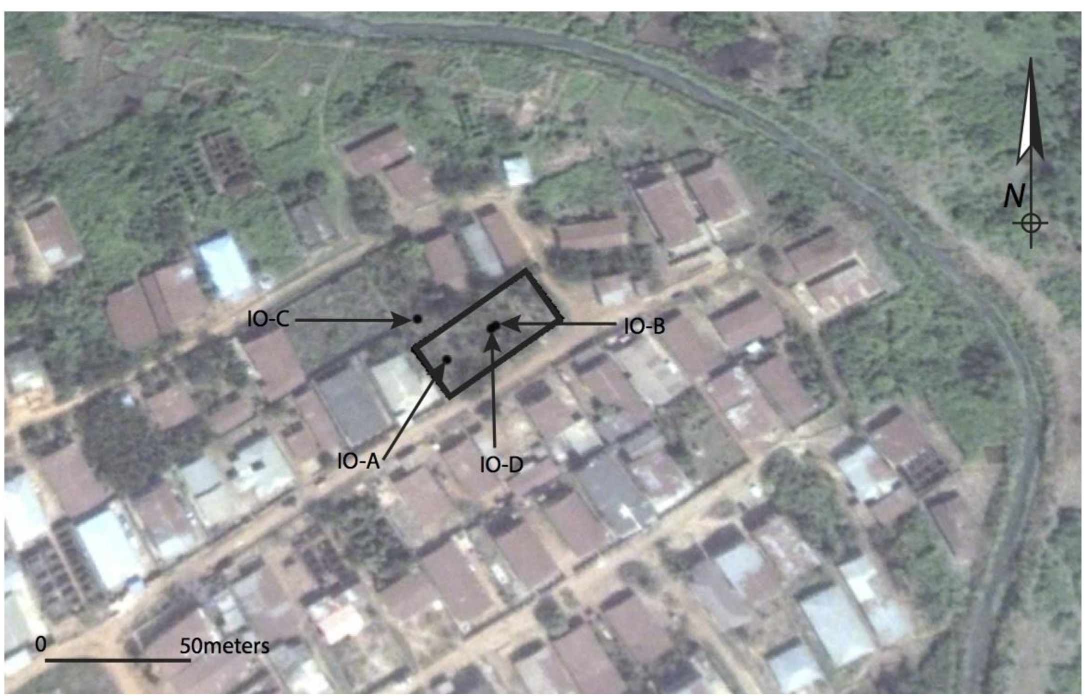


Figure 3. Aerial view of the northern part of Ile Ife, covering the area known as Igbo Olokun, indicating the remaining protected site and the excavated units. (Google Earth image.)

Three 1 x 3m excavation units, IO-A, -B, and –D, were opened within the NCMM fenced plot. A fourth, IO-C, was situated on private property immediately adjacent to the NCMM property (Fig 3). Almost 13,000 beads were recovered, using 1.2 mm mesh for screening. In addition, the excavations yielded 812 crucible fragments, almost three kilograms of glass waste and cullet, and approximately 14,000 potsherds.

In all the excavations, culture-bearing deposits rested on top of, or in pits dug into, the natural lateritic clays and gravels that derive from the weathering of metamorphic schist and gneiss basement rocks in the region. This sterile, extremely compact, reddish-orange clay was reached at a depth of between 70 cm in IO-A and 130 cm in IO-C. In all but Unit IO-A, the sterile clay surface was uneven and penetrated by pits and channels, reflecting various episodes of digging. Above the sterile clay were reddish-brown, generally compact loam or heavy loam deposits with varying amounts of gravel and evidence for disturbance or secondary deposits. The red clays and gravel in these deposits derive from the incorporation of the natural basement clay either as building material or as waste from pits dug through it. Moist, clay-rich, very dark brown deposits characterized the fill of pits dug into the sterile clay and overlay the clay in areas adjacent to the pits (Fig. 4 & 5). Uppermost in the units was a 10-25 cm thick layer of brown soil with organic matter, recent trash, and root penetration by banana and other plants. Trash pits or accumulations were encountered in most of the excavation units, but some were very difficult to detect until they penetrated the sterile clay.

Three pits with dark fill were encountered in adjoining excavations IO-B and -D (Fig. 6); the northernmost one (Pit II) was excavated to a depth of 2.2 meters without reaching the bottom. It was a bell-shaped pit and appeared to have a passage leading into the northwest wall of the unit. Like all levels and pits in all the excavation units, it contained crucible fragments, beads, pottery, and glass debris, but not in any notable concentrations or associated with any elements that would provide a clue as to the original function of the pit. A recent radiocarbon date from 1.4 meters depth in the pit indicates that it had been emptied and refilled sometime in the 18th or 19th century.

Artifacts are relatively dispersed throughout the deposits without any notable spatial focus. Crucible fragments, for example, were recovered from all excavated levels (Table 1). The high level of comminution of both potsherds and crucible fragments in all excavated units and levels suggests that materials had experienced a substantial amount of churning. No workshop features such as intact furnace linings were found in the excavated units. However, the abundance of glass production debris and the presence of vitrified clay fragments, especially in IO-B, -C and -D, indicate that these areas were in or very near a zone of glass workshops.

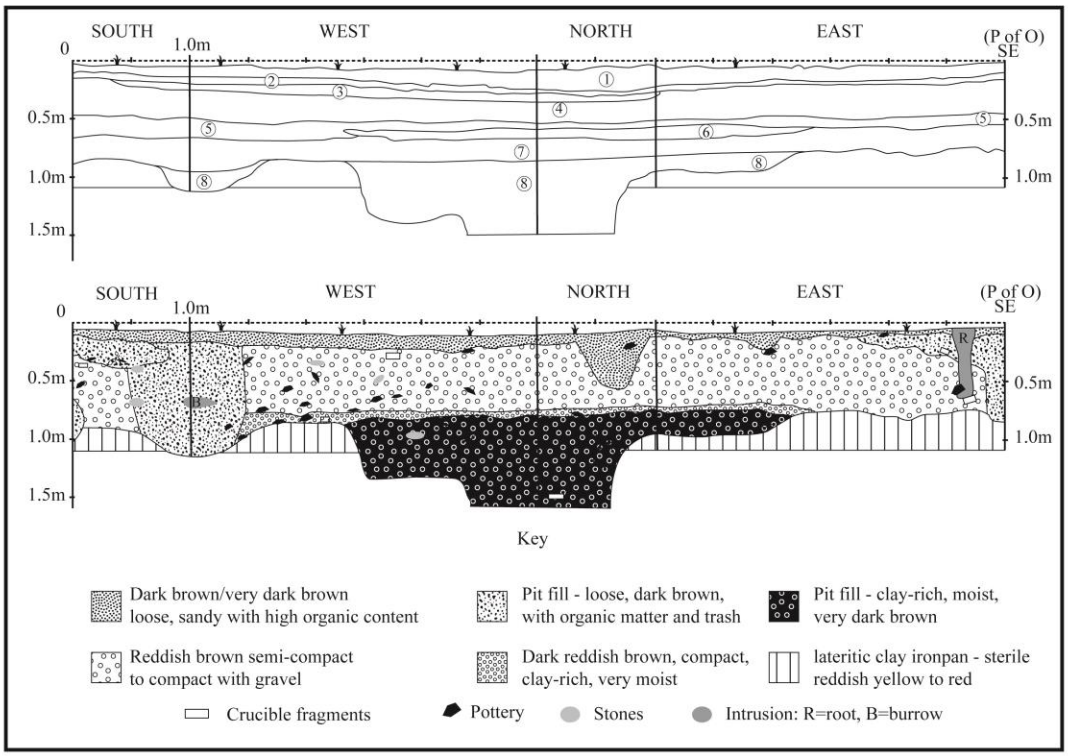


Figure 4: Natural Stratigraphy and excavated levels of Unit IO-B

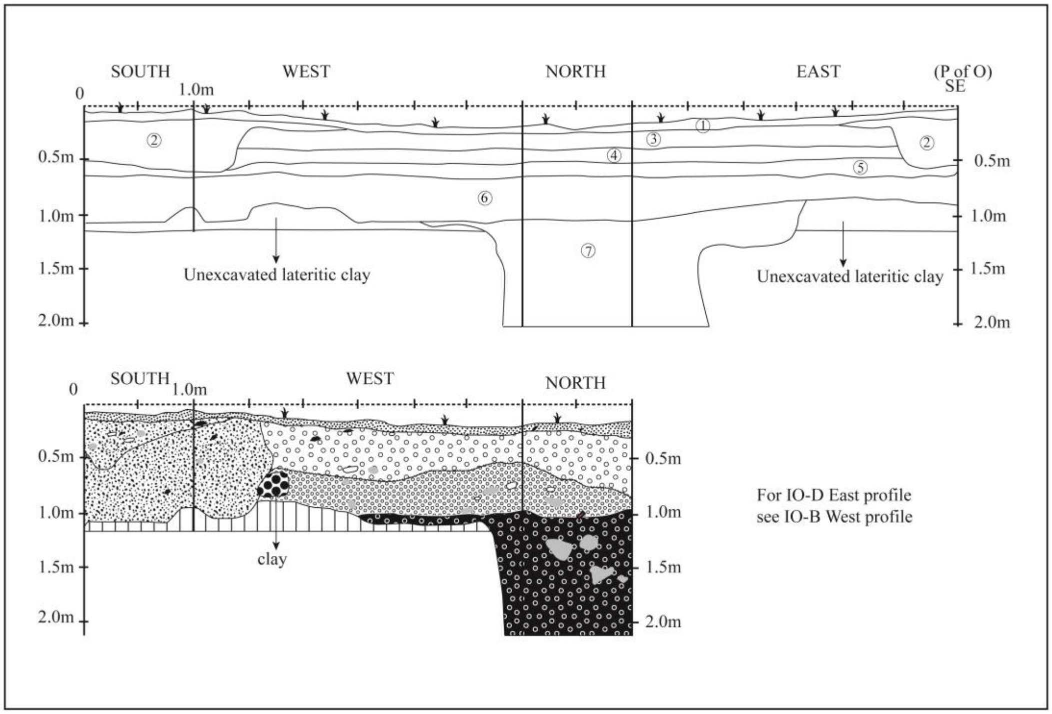


Figure 5: Natural Stratigraphy and excavated levels of Unit IO-D, the adjoining unit to IO-B

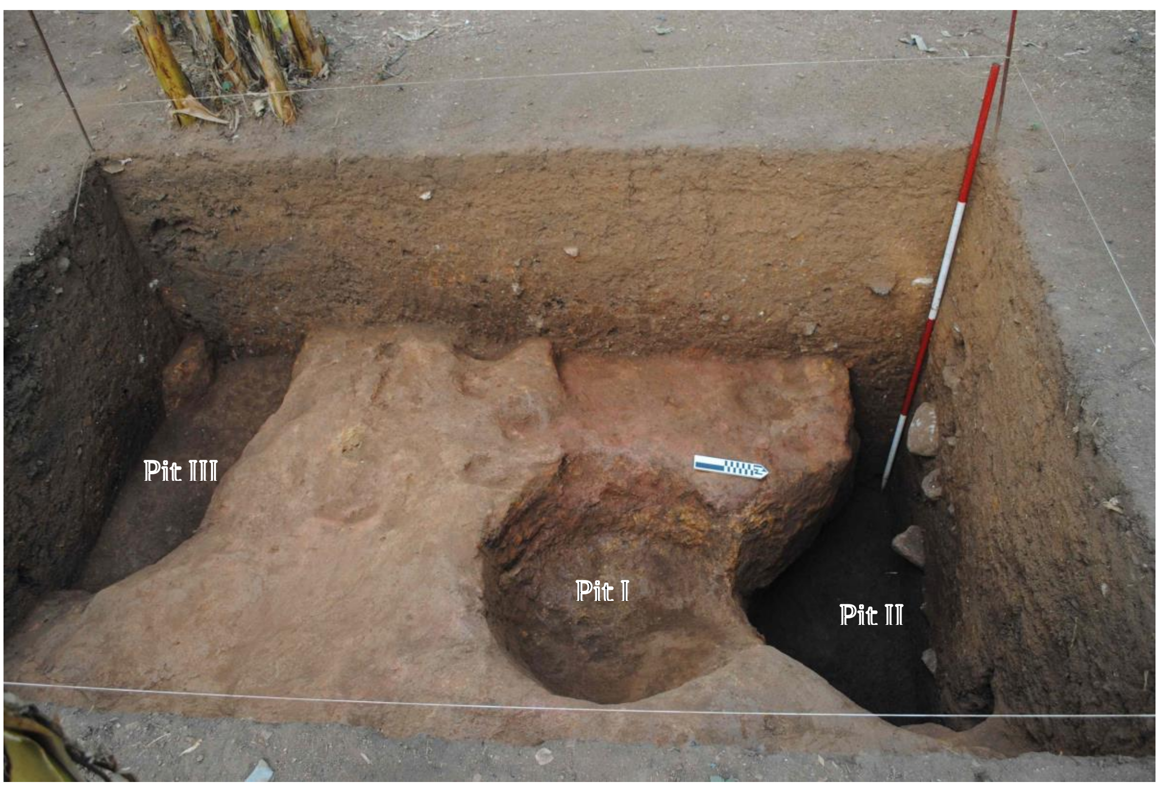


Figure 6. Excavated unit IO-B (east half) and its western expansion, IO-D.

Levels	IO-A	IO-B	IO-C	IO-D	Total
1	14	3	8	8	33
2	4	6	19	39	68
3	25	3	18	25	71
4	4	57	55	35	151
5	——— –	27	74	30	131
6	——— –	29	73	106	208
7	——— –	35	17	48	100
8	——— –	50	———	———	50
Total	47	210	264	291	812

Table 2: Frequency distribution of crucible fragments from excavation units by excavated levels. Dashes indicate that no level with that number was excavated.

Reconstructing the chronology for Igbo Olokun is a challenge because of the evidence of disturbance and secondary deposits. However, based on two radiocarbon dates and the similarity of the excavated pottery to Classic period ceramics described by Garlake (1974, 1977) at other sites in Ile-Ife, we conclude that the production materials, beads, and charcoal were produced in workshops operating between the 11th and 15th c AD (see Babalola 2015 and Babalola *et al*. 2017 for details of the ceramic comparisons). A charcoal sample from the fill at the top (0.70 m depth) of Pit II in unit IO-B produced an AMS date of 840±30 BP (1058-1264 cal AD – Beta 319447). We believe this correctly dates the production debris, which may have originally been deposited in the deep pit but was dug out and redeposited in the 18th–19th century, to judge from the AMS date of 70±30 BP (Beta -319449) from 1.43 m depth in the same pit fill. Basal deposits in unit IO-C were dated by AMS on charcoal to 570±30 BP (1304-1423 cal AD – Beta 319448).

While confirmation of this chronology will require further investigation, it is consistent with available evidence for glass and glass production debris from other sites in Ile-Ife. Crucibles, glass beads, and glass debris were recovered from 11th-15th c contexts in Obalara and Woye Asiri (Garlake 1974, 1977). Similar radiocarbon dates were also obtained from the glass-working site at Ayelabowo (Adeduntan 1985). This may not represent the earliest phase of glass-working at Ile-Ife, but we conclude that it is the period represented by the production materials we excavated at Igbo Olokun. In the remainder of this paper, we describe the crucibles, present the results of the SEM-EDS analysis, and discuss the significant technical information that can be deduced from the crucible materials.

Igbo Olokun crucibles: description and analysis

Description

The four 2011/2012 excavation units at Igbo Olokun yielded a total of 812 crucible fragments weighing over 40 kilograms (Fig. 7). All crucible fragments measuring more than 4 cm in length were recorded in terms of provenience (excavation unit and level), thickness (measured with calipers), vessel part (rim, base, and body), presence or absence and color of glass encrustation on both the inside and the outside, and paste color on a fresh break. Fragments measuring less than four centimeters constituted approximately 45% of the recovered material.

The excavated fragments include rim, body, base, and lid fragments (Fig. 8 a-g). Body fragments constitute over 80% of the assemblage across the units. Their curvature and general appearance are consistent with the complete crucibles described above. About two-thirds of the fragments have a smooth, well-preserved layer of glass on their interior surfaces. Sometimes, striations were noticed in this glass layer, which created bumps or ridges on the interior surface. These striations formed either multidirectional or unidirectional patterns. Willett (2004) has suggested that these ridges formed when glass melt was scraped out of the crucible (Fig. 9 middle). The thickness of the inner glass varies from less than 1mm (thin layer) to 10mm (thick layer) (Fig. 9 top).

Figure 7. Crucible fragments from Igbo Olokun excavations showing different interior glass colors. Representative selection of finds from units IO-A to IO-D.

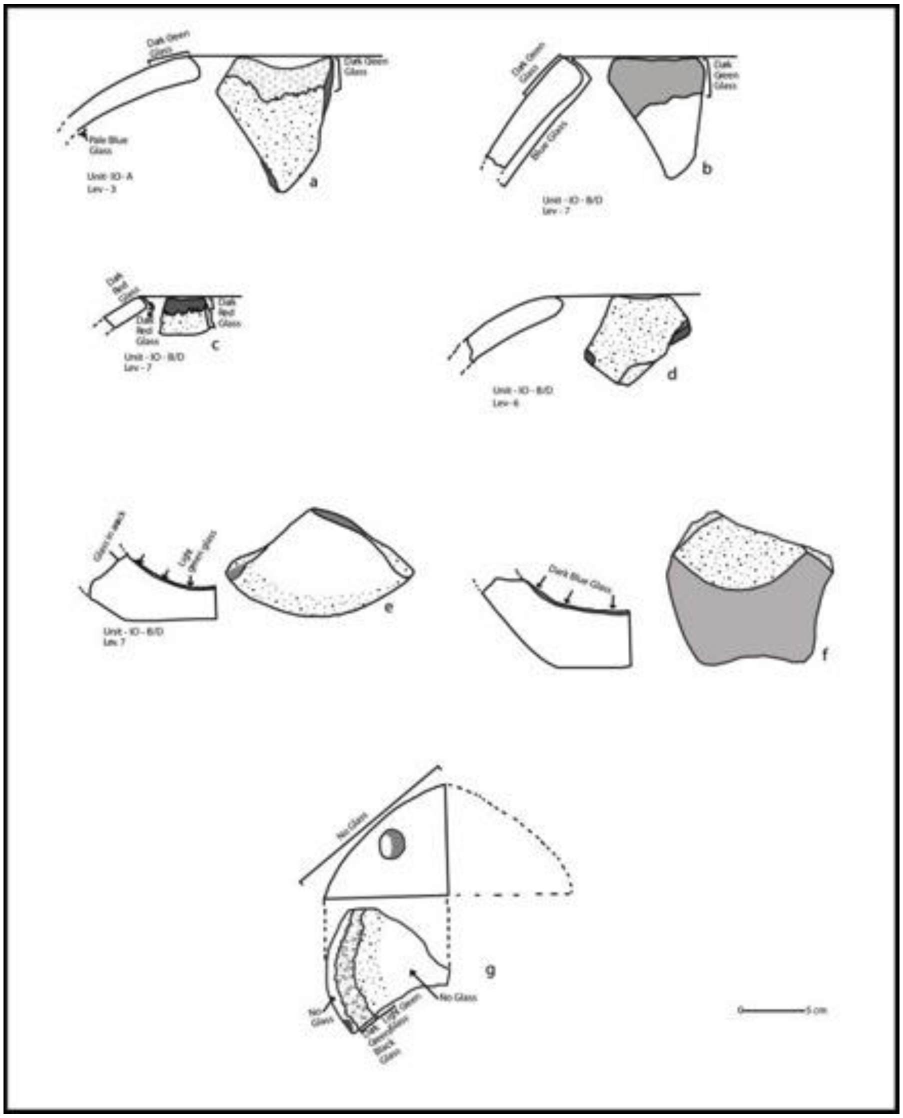


Figure 8. Sample profiles of Ile-Ife glass crucible fragments (Rim a-d; base e & f; lid g) (*Lettering to be improved*)

Figure 9: Selected crucible fragments from Igbo Olokun. Top - Profile view showing the thickness of the glass encrustation. Middle - Showing the ridges/striations on the interior glass. Bottom - Example of crucibles with multi-color glass on the inside.

Approximately one-third of the assemblage has no glass adhering to the interior. These fragments could come from crucibles that broke before they could be used, or on which the glass layer had flaked away, as there is occasional evidence of glass chips. Most likely though, crucible fragments in this category could have come from parts of the crucible closer to the rim and above the "glass level" (Pusch and Rehren 2007: 134).

Rim fragments averaged about 10% of all recorded pieces. Most are simple with a rounded lip. Occasionally, a flattened surface is noticed on the lip of the rim. This surface may represent wear resulting from contact with a lid, or a platform deliberately formed for the lid. Observation of the rims in our collection and the complete crucible vessels on display both at the National Museum Ile-Ife and the British Museum shows that vessels in the assemblage have a restricted orifice, ranging from slightly closed (about 45°) to closed (almost 90°).

Only five base fragments were found, representing less than 1% of all crucible fragments. This is considerably less than the number of base fragments one would expect based on a consistent breakage and preservation pattern. A possible explanation could be the selective retrieval of the better preserved base fragments during earlier looting of the site.

Lids are also not well represented in the assemblage. The only recognizable lid fragment came from level 7 of unit B/D in pit II, and is hemispherical in shape. There are two holes with diameters of 2–3 cm on the side of the fragment. These holes appear to have been a set of possibly three (Fig. 8g) into which rods would have been inserted to lift the lid. Willett (2004: T839) has also noted holes in crucible lids, and suggested that they are possibly for "the insertion of a handle." There is glass/glaze fusion on the inside of the lid. The glass encrustation in the underside of the lid is restricted to the outer circumference of the surface (Fig. 8g). The color and position of the glass on the lid match with one of the rims when the lid is placed on it, although this particular lid might not necessarily be for the crucible vessel that particular rim fragment came from. Compared to those Willett (2004) illustrated, the lid excavated from unit IO-B/D appears to be bigger and more hemispherical in shape.

The fragmentary nature of the assemblage made it difficult to estimate the number of vessels represented in the assemblage. However, since none of the 36 rim fragments seems to belong to the same vessel, we assume that this represents the minimum number of crucibles in the assemblage. Overall, the crucible fragments from the excavations at Igbo Olokun are morphologically consistent with the complete vessels reported by Willett (2004), and those in British and Ile-Ife museums.

The glass colors observed on the inner surfaces are the same as for the glass beads, and consistent with the variety of colors stated in the literature (e.g. Frobenius 1913 [1968]; Eluyemi 1987; Willett 2004). The exception to this is the absence of solid yellow glass, and the addition of a transparent blue-green category for the crucible glass. Although opaque yellow is rare in the glass bead assemblage associated with the crucibles, it is puzzling that crucibles with opaque yellow glass are completely missing in the collection.

Among the identified colors, blue and green are the most common (Fig. 10). Other colors present in low frequencies are red, blue-green, and black. Although dichroic glass has been identified among Ile-Ife glasses (Davison et al., 1971; Lankton et al., 2006; Babalola 2015), none was recognized from the glass inside the crucibles. This lack of dichroic glass may be because the glasses could only be viewed in reflected light due to their encrustation in the crucible. Occasionally, two colors or shades of the same color were observed on a single crucible fragment (Fig. 9 bottom). This could have resulted from variations in the composition of coloring oxides due to incomplete mixing of the colorants (see below), variable redox conditions, or multiple use of the crucible for different color batches (Willett 2004).

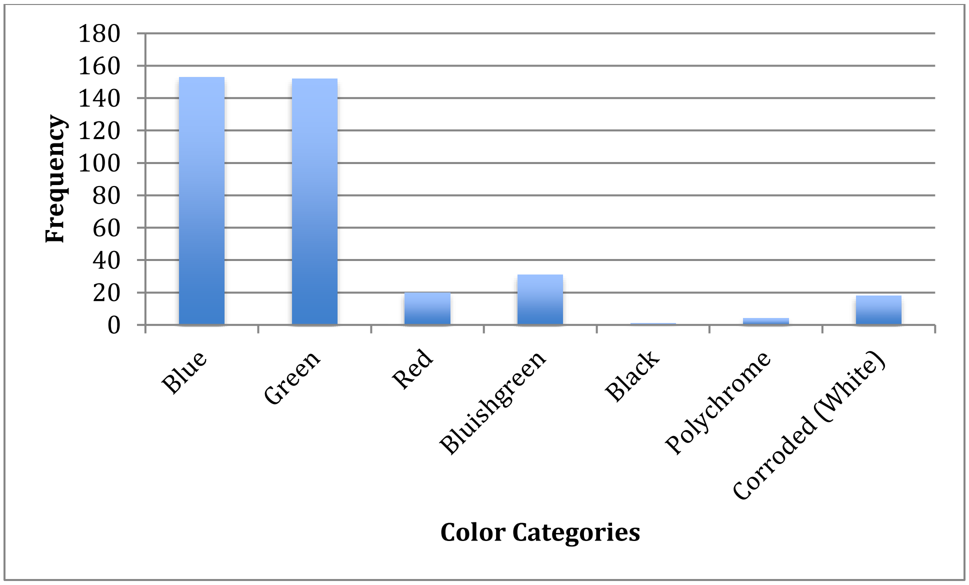


Figure 10: Frequency of glass color categories on the interior surface of the crucibles

The crucible fabric color varies from dark and light gray to white, and occasionally off-white with a light yellowish tone. About 40% of the assemblage has glaze on the exterior surface. The glaze was most likely unintended and would have formed due to the reaction of the ceramic surface with the fuel ash during heating. In addition to the glaze, some of the intentionally made glass is occasionally present on the exterior surface, which could have resulted from slippage or dripping of melted glass during working.

The thickness of the crucible walls varies considerably among different vessel parts, with 2 to 2.9 cm the most common for body fragments (Fig. 11). Although not well represented, bases seem to generally have greater thickness. Crucibles with red and black glass rarely have a wall thickness of 3 cm or greater. Applying a chi-square test to thick and thin walled sherds in the two color categories in Figure 11 resulted in a p value of 0.5, suggesting that the rarity of red and black glass on thick-walled fragments is unlikely to be due to chance. Assuming that differences in wall thickness reflect different crucible sizes, this indicates that smaller crucibles were used for the less-common colors.

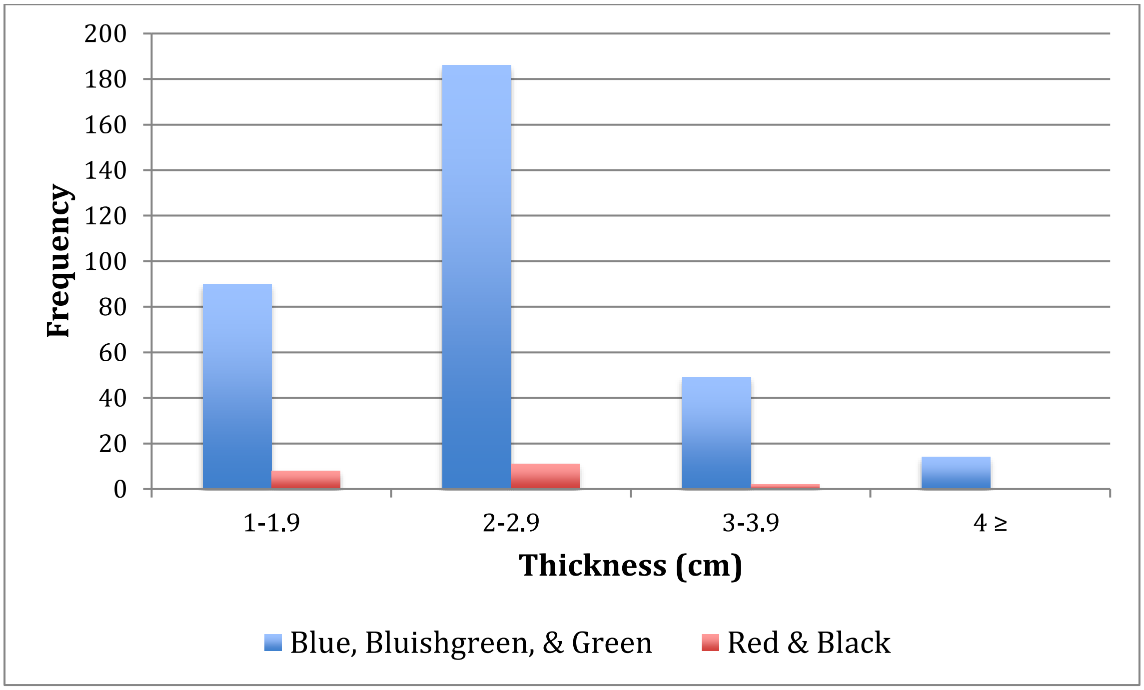


Figure 11: Thickness distribution of Ile-Ife crucibles with regard to color of attached glass.

Microstructural and chemical analysis: Methods

Small fractions of eight crucibles were selected for further microstructural and chemical analysis, covering a range of thicknesses, glass colors and excavation units. Two additional samples, collected earlier on the surface at the site by one of the authors (Akin Ige), were also analyzed to test their consistency with the excavated material. Table 3 describes the ten crucible samples selected for microscopic and chemical analysis. The laboratory analysis was carried out in the archaeological material science lab facility of UCL Qatar.

Small fragments from each of the ten samples were cut and mounted in epoxy resin, and properly polished (Fig. 12). The samples were then examined in a JEOL JSM6610LV scanning electron microscope (SEM) and their chemical composition determined by Energy Dispersive Spectrometry (EDS) using an Oxford Instrument Aztec detector. The areas selected for analysis were the outer glaze, the crucible fabric near that outer glaze, the crucible fabric near the inner surface in contact with the glass, the interior glass layer, and the transition between the latter two in order to see if there were any recognizable differences in the microstructure and composition among the crucible fragments. The interior transition area was examined for evidence of the interaction of the glass melt with the fabric. Analytical totals were in the order of 95 to 105 wt% for dense glassy areas, and 85 to 95 wt% for ceramic areas. Deviations from 100 wt% reflect beam intensity variation and porosity of the ceramic. To facilitate easy comparison the data are reported as oxides by stoichiometry re-calculated to 100 wt%.

Unit	**Level**	**Sample #**	**Thickness. mm**	**Vessel Part**	**Int. Glass Color**	**Ext. Glaze**	**Fabric Color**	**Comments**
Surface		IFE CC 1	2.4	Body	Blue	Yes	White	Interior glass: midnight blue; smooth surface. Exterior glaze: thin layer
Surface		IFE CC4	1.9	Body	Light Green	Yes	White	Interior glass: blue tint, smooth surface. Exterior glaze: thin layer
IO-B/D	7	IF0072	1.2	Body	Red	Yes	White	Interior glass: black mixed with red. Exterior glaze very thin layer, almost indistinct
IO-A	4	IF0073	3.7	Body	Blue	Yes	Dark gray	Thick exterior glaze, ranging from pale to dark blue in cross-section.
IO-C	7	IF0074	1.9	Body	Blue	Yes	White	Exterior glass: thin layer
IO-B/D	7	IF0075	2.2	Rim	Blue	Yes	White	Exterior glass: fuel ash or smoke appears to have added greenish tint to original blue glass.
IO-D	2	IF0076	1.8	Body	Light Green	Yes	Light gray	Interior glass: off-white surface color, possibly due to corrosion,

								light green in cross-section. Exterior glaze: thin layer.
OO-A	2	IF0077	2.1	Body	Red	Yes	off-white	Interior glass: blue mixed with red.
IO-B/D	7	IF0078	3.8	Body	White	Yes	off-white	Interior glass: chalky due to corrosion.
IO-C	7	IF0079	1.9	Body	Black/dark gray	Yes	off-white	Interior and exterior glass: thin layer

Table 3: Provenience and description of the crucible samples analyzed by SEM-EDS

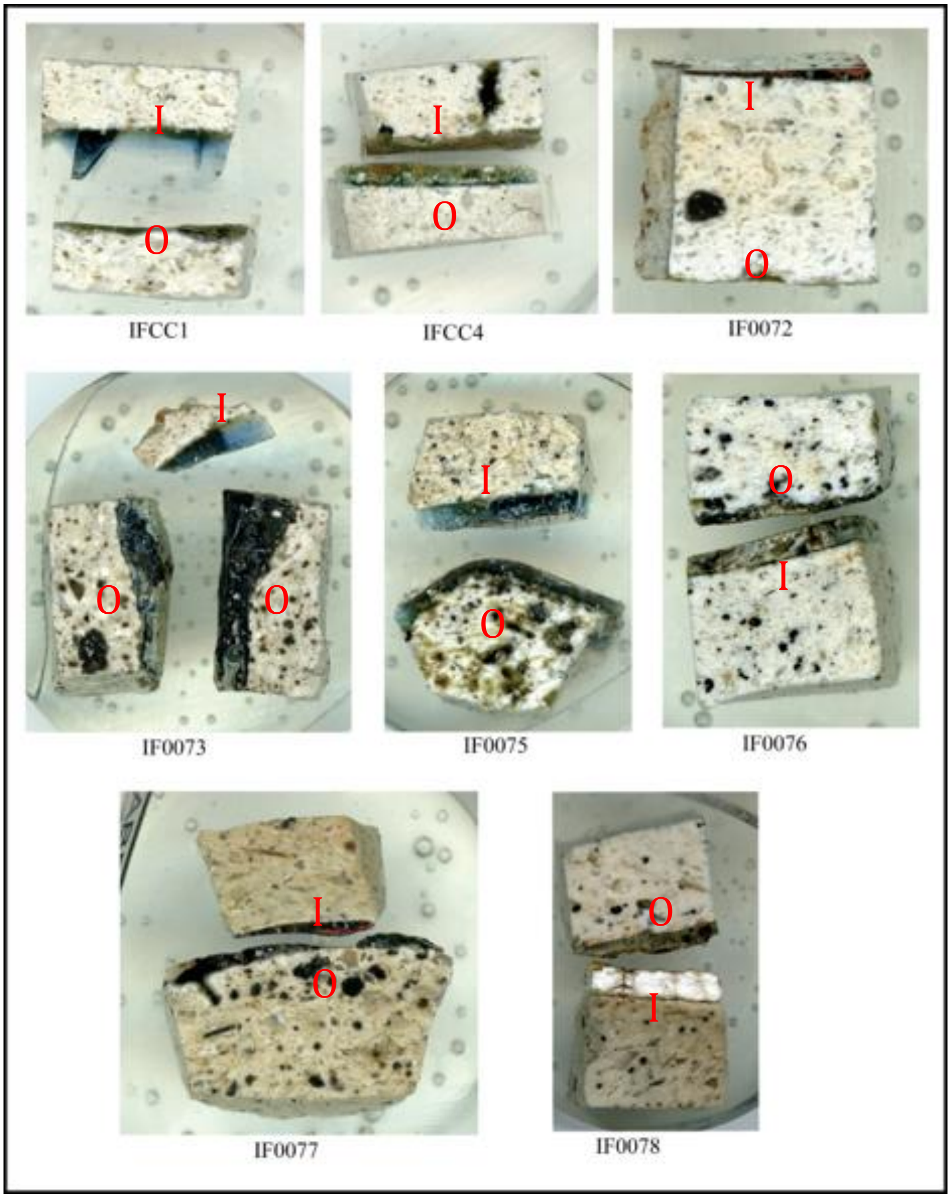


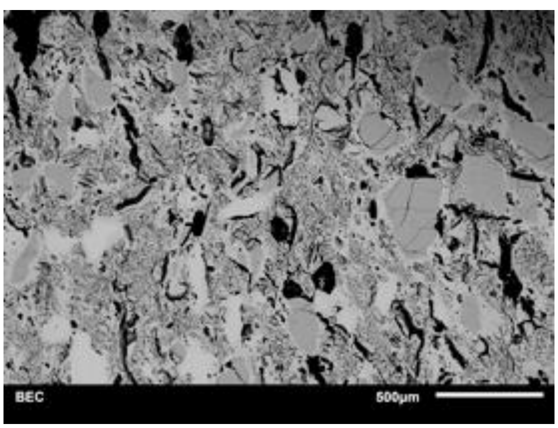
Figure 12: Prepared samples for SEM/EDS analysis (see details in Table 3 above). For practical reasons each sample was cut in half and mounted so that the inner glass layer (labeled ‘I’) and the outer glaze layer (‘O’) face each other.

Results of the Analysis

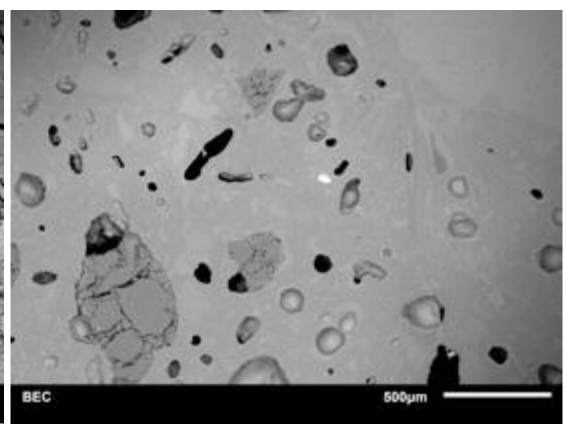
Microstructure and Composition (Table 5a-d)

Fabric

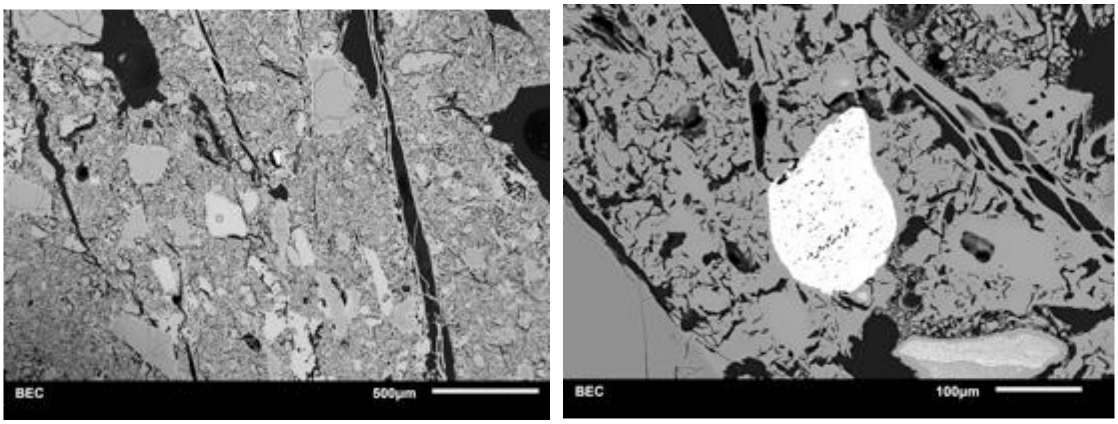
The fabric appears highly vitrified, with greater vitrification near the outer surface compared to the inner fabric (Figs. 13a-b). Quartz grains appear as a common constituent of the fabric with evidence of thermal fracturing (Fig. 13a-c). Based on the volume, rounded nature, and the consistent size of the quartz grains in the Ile-Ife crucibles, Lankton *et al*. (2006: 119) have suggested that they are natural sand components in the crucible clay. Both elongated and more equant voids were observed in the fabric (Fig. 13c). While the elongated voids may indicate the former presence of organic materials, the more equant voids may represent natural spaces in the clay matrix. Titania rich inclusions such as ilmenite and leucoxene were also present in the clay matrix (Fig 13d).



a



b



c d

Figure 13. BSE images of the matrix of the crucible fabric. a. Fabric near the center of the fragment (sample IF0074). b. Fabric near the outer glaze. Note the higher degree of vitrification compared to the fabric near the center (Sample IF0073). c. Elongated voids suggesting burned off organic temper (Sample IF0078). d. Titanium rich inclusion (bright, center) and organic remains (upper right corner)(Sample IF0077). Also note the quartz grains in Figure 13 a-c (darker gray, often fractured).

The composition of the ceramic is dominated by silica (57 – 68 wt% SiO_2) and alumina (26 – 35 wt% Al_2O_3), with minor amounts of potash (average 3 wt% K_2O), iron oxide (average 2.3 wt% Fe_2O_3) and soda (average 0.8 wt% Na_2O). Titania is present in concentrations around 0.5 wt% or less, at the lower end of reported concentrations from other kaolinite ceramics (e.g. Rehren and Papachristou 2003: 396; Martinón-Torres *et al.* 2008: 2072; Yin *et al.* 2011: 2355). All other oxides are present only at concentrations below 0.5 wt% (Table 4). Darker and lighter gray areas were noticed in the SEM-BSE images, indicating compositional heterogeneity. Compared to the lighter area, the darker areas have higher Al_2O_3. There are also more vitrified areas within the crucible fabric with elevation of Na_2O-Al_2O_3-CaO, which may be due to melted glass leaked through a crack into the fabric matrix, or melted plagioclase feldspar. Overall, the clay was not particularly homogenized. The composition of the ceramic near the inner surface has slightly elevated soda content, averaging 2 wt%, indicating a limited amount of penetration of soda from the glass melt (Table 5; Fig 14).

Body fabric	**Na_2O**	**MgO**	**Al_2O_3**	**SiO_2**	**P_2O_5**	**K_2O**	**CaO**	**TiO_2**	**MnO**	**FeO**
IFCC 1	0.8	0.1	32.5	59.9	<dl	4.7	0.1	0.1	<dl	1.8
IFCC4	0.7	0.2	34.9	58.5	<dl	2.4	0.1	0.6	<dl	2.5
IF0072	0.7	0.2	26.1	68.3	<dl	2.7	0.2	0.1	<dl	1.5
IF0073	1.3	0.1	33.0	59.0	<dl	3.6	0.1	0.3	0.1	2.4
IF0074	0.5	0.2	34.5	59.5	<dl	3.0	0.2	0.2	<dl	1.8
IF0075	0.6	0.1	28.7	62.2	0.1	4.5	0.1	0.3	<dl	3.2
IF0076	0.8	0.1	27.5	66.2	<dl	2.7	0.1	0.5	<dl	2.0
IF0077	0.4	0.2	35.1	57.2	0.2	3.5	0.1	0.1	<dl	3.0
IF0078	0.3	0.1	35.1	59.6	<dl	3.0	0.1	0.2	<dl	1.4
IF0079	0.9	0.1	27.8	63.8	<dl	4.8	0.7	0.2	0.1	1.6
Body near glass	**Na_2O**	**MgO**	**Al_2O_3**	**SiO_2**	**P_2O_5**	**K_2O**	**CaO**	**TiO_2**	**MnO**	**FeO**
IFCC 1	1.6	0.4	33.3	56.9	<dl	4.6	0.2	0.2	0.1	2.6
IFCC4	2.6	0.2	37.6	54.1	0.1	2.7	0.3	0.5	<dl	1.7
IF0072	1.8	0.2	38.6	54.0	0.1	3.6	0.2	0.1	0.1	1.3
IF0073	1.8	<dl	36.9	54.3	<dl	4.0	0.2	0.2	0.2	2.4
IF0074	1.4	0.2	36.6	57.0	0.1	2.7	0.2	0.2	<dl	1.6
IF0075	2.3	0.1	32.2	59.5	<dl	2.8	0.2	0.3	<dl	2.4
IF0076	4.5	<dl	33.0	55.9	<dl	4.7	1.2	0.1	0.1	0.5
IF0077	1.1	0.1	36.9	55.4	0.3	2.6	0.1	0.2	<dl	3.2
IF0078	2.0	0.1	40.8	51.3	<dl	4.4	0.3	0.0	0.1	0.8
IF0079	1.3	0.1	35.2	55.5	0.1	5.5	0.4	0.3	<dl	1.6

Table 4: Chemical composition of the fabric of the crucibles (SEM-EDS area analyses, reported as weight% oxides, normalized to 100 wt%). Upper part: fabric in the center of crucibles; lower part: fabric near the inner glass layers.

Outer glaze	**Na_2O**	**MgO**	**Al_2O_3**	**SiO_2**	**P_2O_5**	**Cl**	**K_2O**	**CaO**	**TiO_2**	**MnO**	**FeO**
IFCC 1	0.2	3.8	17.5	45.3	0.6	<dl	9.6	20.0	0.7	0.3	2.0
IF CC4	1.0	2.3	15.4	50.5	1.3	<dl	5.0	20.3	0.8	0.2	3.1
IF0072	0.6	3.1	16.5	47.9	1.0	<dl	7.3	20.2	0.7	0.3	2.6
IF0073	0.2	3.2	14.1	54.8	1.0	<dl	6.2	12.1	1.7	0.3	6.4
IF0074	2.1	1.0	15.2	61.2	2.8	<dl	8.9	4.5	1.4	0.2	2.7
IF0075	3.9	0.2	11.9	64.9	0.2	<dl	3.2	14.2	0.2	0.9	0.5
IF0076	0.2	0.2	26.0	55.2	0.8	<dl	15.4	0.7	0.3	0.1	1.1

IF0077	0.5	0.6	18.0	61.2	0.7	<dl	3.8	4.3	3.3	0.1	7.5
IF0078	0.5	2.7	18.1	52.2	1.5	0.1	13.3	7.7	1.1	0.5	2.4
IF0079	1.2	3.1	11.0	54.6	1.5	<dl	9.1	12.1	2.0	0.5	4.9

Table 5: Chemical composition of the outer glaze of the crucibles (SEM-EDS area analyses, reported as weight% oxides, normalized to 100 wt%).

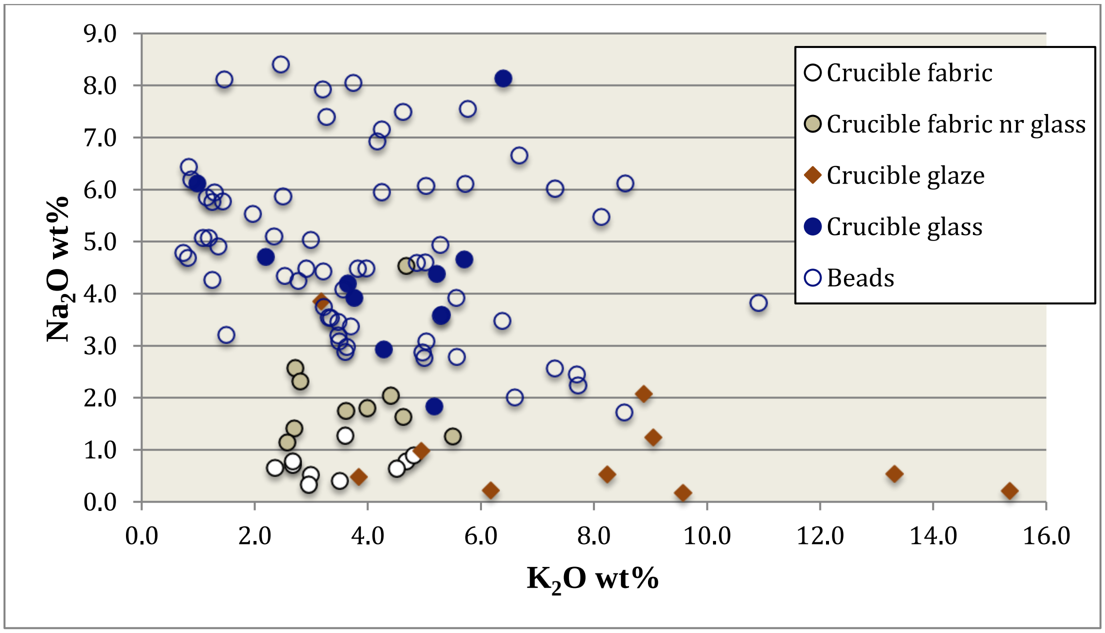
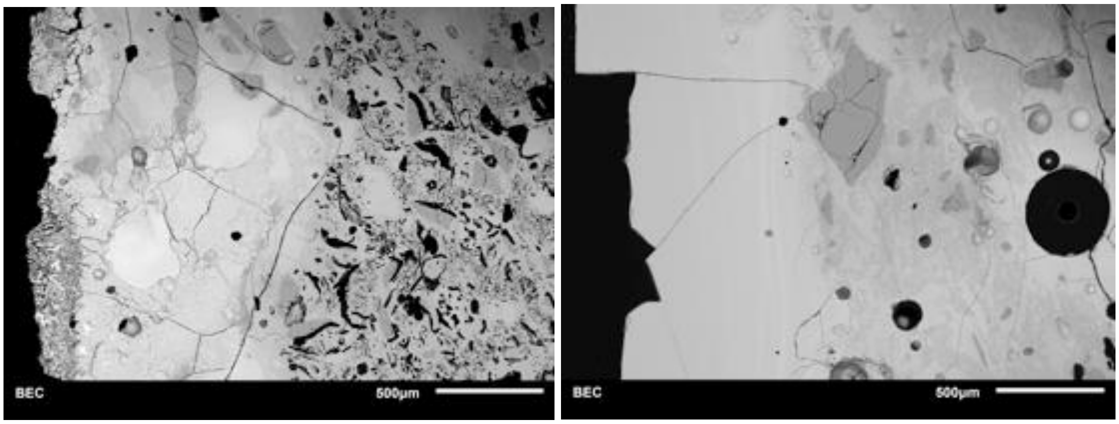


Figure 14. Comparing the soda vs potash contents in the major segments of the crucibles. Note how the crucible glass matches the glass beads, while the outer glaze is much richer in potash and lower in soda. The crucible fabric is low in both alkalis, indicating that it is not the source of the alkalis in either the glass or the glaze. Bead data from Babalola *et al.* (in press).

Outer glaze

The outer glaze differs strongly from the crucible fabric, with greater vitrification and fewer quartz grains in the glaze compared to the fabric (Fig. 15a & b) and significant compositional differences, too (Table 5).



a b

Figure 15. BSE image of examples of the matrix near the outer surface. a. Thin outer corroded glaze. Note the difference between the highly vitrified area of the fabric next to the glaze and the area farther from the glaze (sample F0074).
b. Compositional analysis shows that the outer encrustation in this case is glass rather than glaze, indicating spillage of melted glass. Also note the highly vitrified area fronting the glaze (Sample IF0075).

Compositionally, the outer glaze is very heterogeneous, with high lime (up to 20 wt% CaO) and potash (up to 15 wt% K_2O), and elevated levels of magnesia (up to 3.8 wt% MgO) and phosphate (up to 2.8 wt% P_2O_5) (Fig 16; Table 5). This high level of CaO and the elevated concentrations of MgO, P_2O_5, and K_2O indicate that the glaze is formed by interaction of fuel ash with the crucible ceramic (see below).

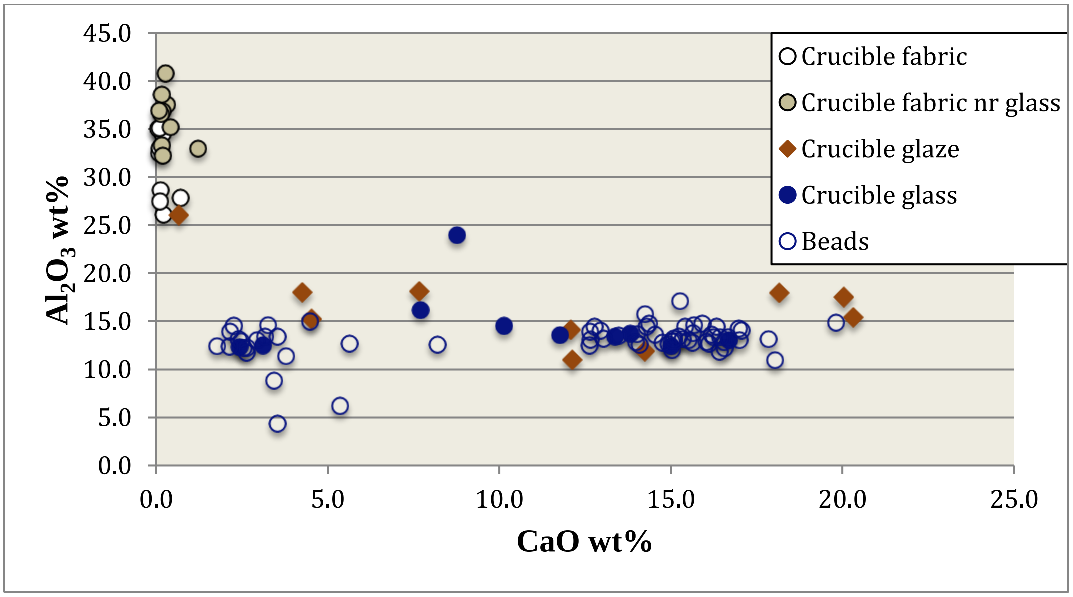


Figure 16. Alumina vs lime contents in analyzed crucible segments. The lime and alumina content of the crucible glaze matches the composition of the glass within the crucibles and also the glass beads.

Inner glass

The interior glass is mostly homogeneous, even though sometimes unmelted quartz grains and *schlieren* (swirls) are present (Fig. 17). The glass has typically around 12 to 16 wt% alumina, up to 17 wt% lime, and in the order of 3 to 8 wt% each soda and potash (Table 6). This is consistent with the known composition of Ile-Ife glass, which includes both a high lime high alumina (HLHA) group (Lankton *et al.* 2006; Ige 2010a, b) and a low lime high alumina group (LLHA) (Babalola 2015; Babalola *et al*. in press; Fig 17; Table 6 lower part). The HLHA glass has lime concentrations between 8 and 17 wt% CaO and is low in magnesium oxide (<0.2 wt% MgO), while the two LLHA samples have only around 3 wt% CaO but elevated magnesia exceeding 1 wt% MgO, and higher phosphate than the HLHA glass. The concentration of soda and potash varies significantly (Fig 14). Chlorine is present in the glass only at concentrations below the detection limit of the SEM-EDS, estimated to about 0.1 wt%, matching earlier observations for the Ile-Ife glass beads (Lankton *et al*. 2006; Babalola *et al*. in press). Since chlorine is an important element associated with both mineral natron and plant ash glass where it routinely reaches between 0.5 and 1.3 wt% (Freestone 2006: 140; Tanimoto & Rehren 2008; Rehren 2008), its absence here indicates a different source of alkalis for Ile-Ife glass.

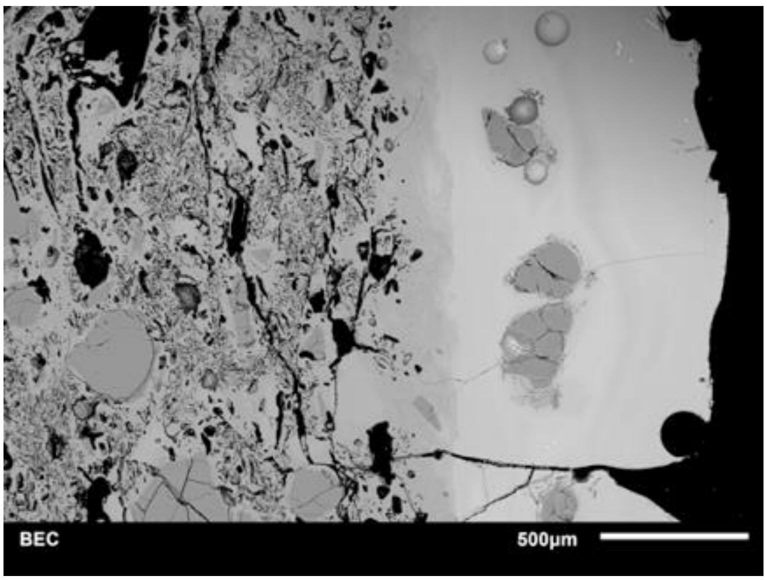


Figure 17. BSE image of the inner glass with quartz grains preserved in the glass matrix (light gray layer on the right). This, and the presence of swirls of different bulk composition (different gray shades in the glass) indicate that glass may have been made in these crucibles from raw materials, resulting in incomplete reaction and heterogeneous glass melt forming (sample IF0074).

In glass	Glass Color	Na_2O	MgO	Al_2O_3	SiO_2	P_2O_5	Cl	K_2O	CaO	TiO_2	MnO	FeO	CuO
IFCC 1	Blue	2.9	0.1	13.1	61.1	0.1	<dl	4.3	16.7	0.1	1.0	0.8	<dl
IFCC4	Yellowish-green	6.1	<dl	12.4	64.0	0.1	<dl	1.0	15.0	<dl	0.1	1.1	<dl
IF0073	Blue	3.6	0.1	14.5	64.0	<dl	<dl	5.3	10.1	0.1	0.6	1.7	<dl
IF0074	Blue	3.9	0.1	13.7	63.4	0.1	<dl	3.8	13.8	<dl	0.7	0.5	<dl
IF0075	Pale blue	4.7	<dl	24.0	58.9	0.1	<dl	2.2	8.8	0.3	0.3	0.7	<dl
IF0076	Yellowish-green	4.2	0.1	13.6	65.1	0.1	<dl	3.7	11.8	0.1	0.9	0.6	<dl

IF0078	White (corroded)	4.4	<dl	16.2	65.3	0.3	<dl	5.2	7.7	<dl	0.1	0.7	<dl
IF0079	Blue	1.8	0.1	13.4	63.7	0.1	<dl	5.2	13.4	0.2	1.1	1.1	<dl
IF0072	Red	4.7	1.4	12.5	66.6	0.4	0.2	5.7	3.1	0.2	0.1	5.1	0.2
IF0077	Red	8.1	1.1	12.3	65.2	0.4	0.4	6.4	2.4	0.1	1.3	1.5	0.7
HLHA	Average	3.9	0.1	13.5	62.3	<dl	<dl	3.8	15.2	0.2	0.5	0.5	0.02
LLHA	Average	6.5	0.9	12.4	68.7	<dl	<dl	5.1	3.3	0.6	0.05	2.0	0.4

Table 6: Chemical composition of the glass inside the crucibles (The first 8 are HLHA glass, the next 2 are LLHA glass; SEM-EDS area analyses, reported as weight% oxides, normalized to 100 wt%). HLHA and LLHA average data are for glass beads; see Babalola *et al*. (in press) for full data.

Discussion

Crucibles as vessels used in high temperature production

Analysis of the crucible fragments from our excavations has confirmed their suitability for and use in glass-related high temperature activities in Ile-Ife. The crucibles are highly refractory and were easily able to withstand the temperatures of 900–1150 ºC needed to melt glass without softening or contaminating the glass charge. They are made from kaolinitic clay with high alumina content and numerous quartz grains, which provided good high-temperature stability and resistance to thermal shock (Martinón-Torres & Rehren 2014: 15; Ige 2010b). Ige (2010b: 9) suggests that the locally abundant kaolin clays with high alumina content (>20%) could have provided a good clay source for the Ile-Ife crucibles. Significantly, this clay composition is not seen in regular domestic pottery (Table 7; Fig 18), suggesting that the crucible clay was specially selected for this property. Due to their much higher alumina content and lower iron oxide content than the domestic pottery, the Ife crucibles would have been able to resist much higher temperatures, well in excess of 1300 °C, (Fig. 19 a,b). Such highly refractory crucibles are unknown elsewhere in Sub-Saharan Africa (Chirikure *et al.* 2015; Killick

2016; Bandama *et al.* 2016), and are only found in highly specialized medieval Central Asian and post-medieval European crucibles (e.g. Rehren & Papachristou 2003; Martinón-Torres & Rehren 2002, 2009, 2014) (Fig 20).

Sample #	Na_2O	MgO	Al_2O_3	SiO_2	P_2O_5	Cl	K_2O	CaO	TiO_2	MnO	FeO
IF0086	0.4	0.6	13.2	73.3	1.9	0.2	0.6	1.2	1.9	0.1	6.6
IF0087	1.7	1.3	23.6	55.0	1.3	0.1	3.4	1.8	1.5	0.0	10.3
IF0088	2.6	2.8	21.1	56.4	1.2	0.1	1.5	3.5	1.3	0.1	9.4
IF0090	1.5	2.7	18.1	64.5	1.6	0.2	0.6	3.2	1.3	0.1	6.2
IF0092	2.7	1.4	18.5	63.5	0.1	0.3	2.1	3.2	1.0	0.0	7.3

Table 7: Chemical composition of the domestic pottery from Igbo Olokun (SEM-EDS area analyses, reported as weight% oxides, normalized to 100 wt%).

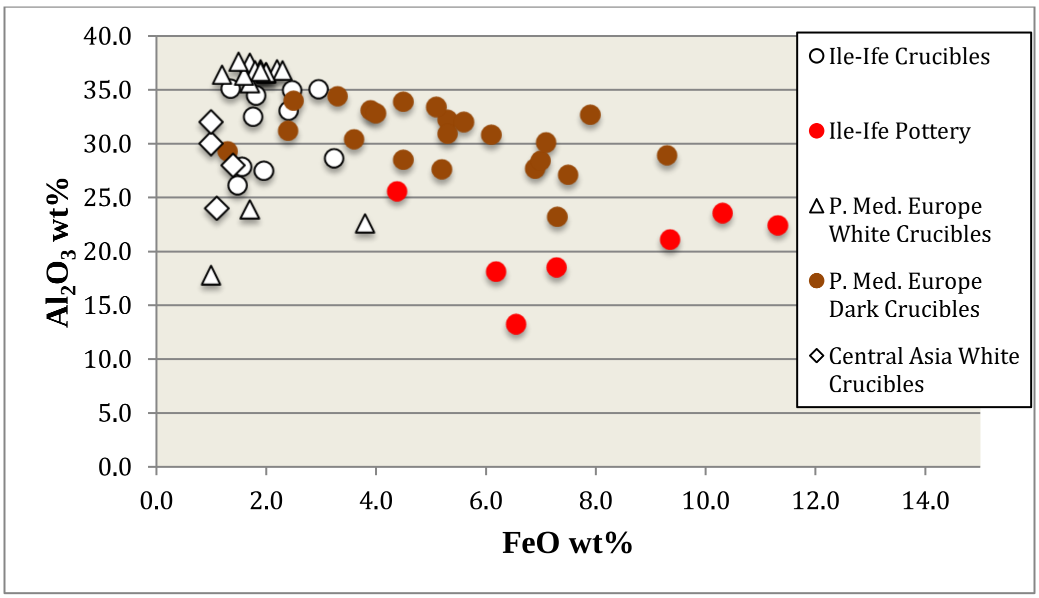


Figure 18. Comparison of alumina vs FeO content of Ile-Ife crucibles with the Ile-Ife domestic pottery and the post-medieval European crucibles (post-medieval European data from Martinón-Torres & Rehren 2009; Central Asian data from Rehren & Papachristou 2003).

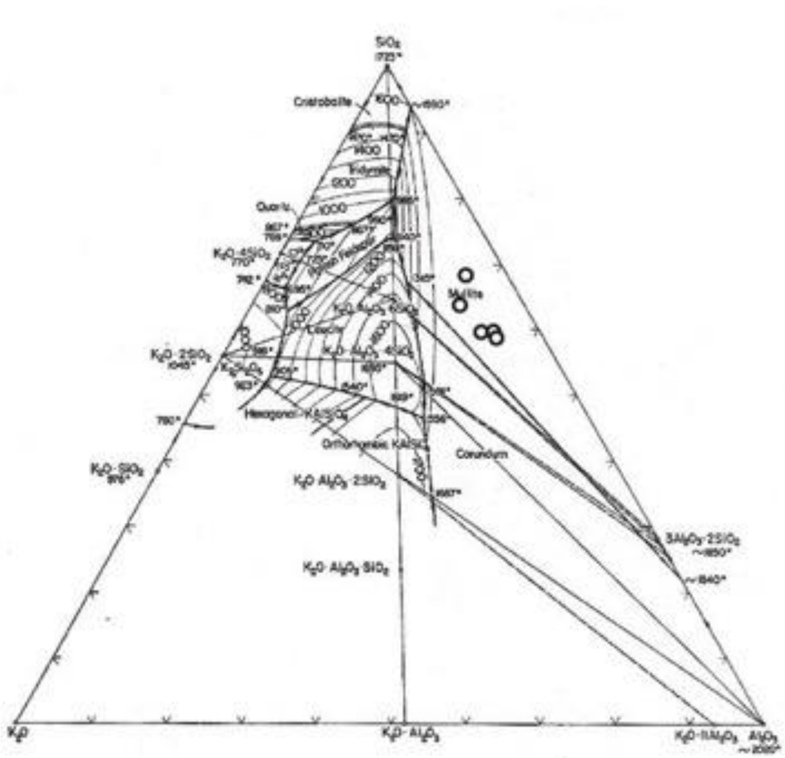


Figure 19a. Projection of the composition of Ile-Ife crucible fabrics (circles) into the ternary phase diagram for kaolinitic ceramic. The composition falls into an area with very high theoretical melting temperatures. Actual melting temperatures would be somewhat lower due to the presence of minor oxides, primarily iron oxide, working as fluxes. See table 5a for actual data.

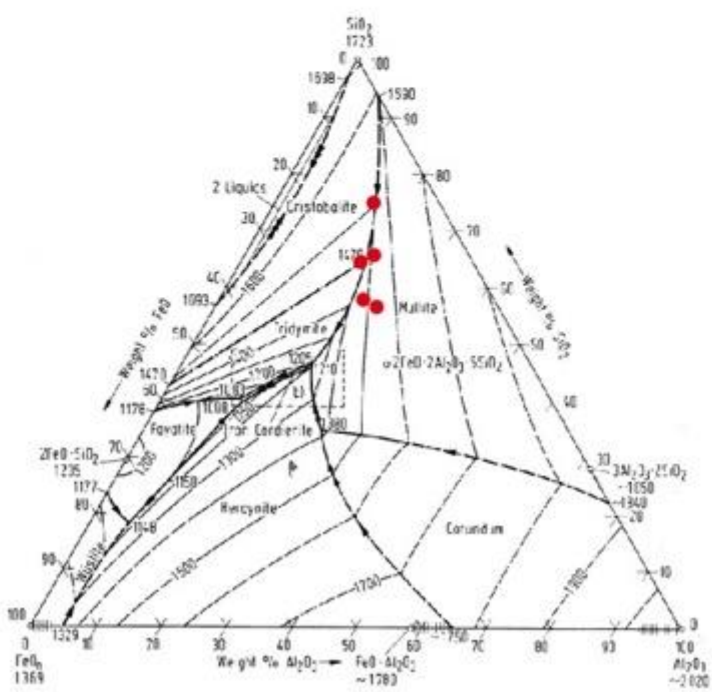


Figure 19b. Projection of the composition of Ile-Ife domestic pottery (red circles) into the ternary phase diagram for ferruginous ceramic. The composition falls near a cotectic trough indicating relatively low theoretical melting temperatures. Actual melting temperatures would be even lower due to the presence of minor oxides such as potash and others working as fluxes.

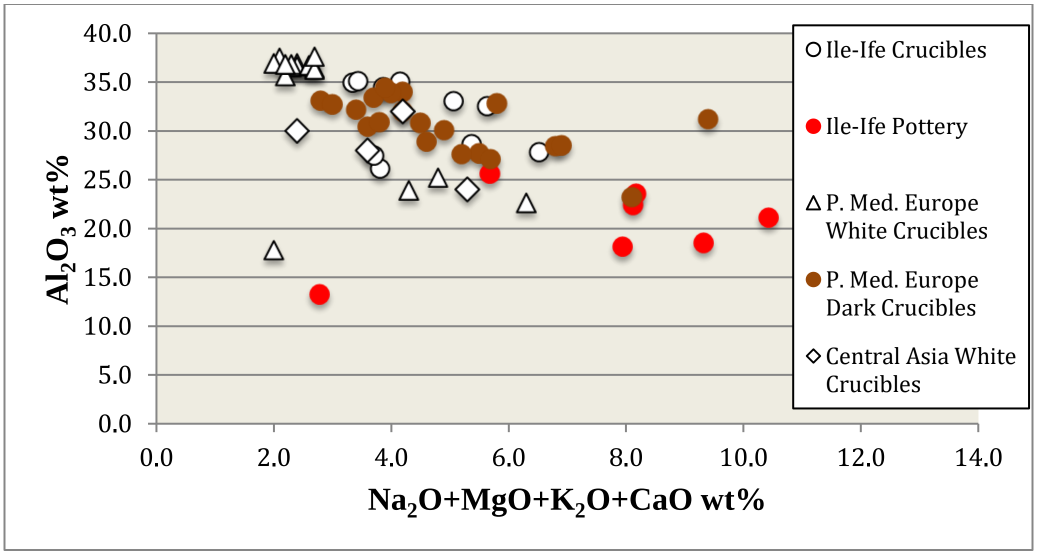


Figure 20. Comparison of alumina vs alkalis of Ile-Ife crucibles and Central Asian and post-medieval European crucibles (data from Martinón-Torres and Rehren 2009; Central Asian data from Rehren and Papachristou 2003).

Crushed quartz and/or organic materials may have been added as temper to the crucible clay to enhance its resistance to thermal shock when used in high temperature operations, preventing cracking of the crucibles during use. Despite this, cracks formed in some of the crucible fragments, and glass seepage observed in some of the cracks suggests that this happened during use.

Outer glaze and inner glass layers

The formation on the outside of the crucibles of a glaze rich in lime, magnesia, potash and phosphorus oxide (Fig 21), and the increased vitrification of the outer layer of crucible fabric is due to the fluxing effect of fuel ash, similar to the reactions between calcareous wood ash and kaolinitic ceramic discussed in detail in Rehren & Yin (2012). Together with the impression of fuel particles in the vitrified outer surfaces (see above) the evidence indicates that the crucibles were embedded in a charcoal bed in the furnace.

The crucible morphology appears to have served a functional purpose. Wall thickness of between 2cm and 4cm and a restricted orifice were designed to provide mechanical stability and to contain the heat in the crucible, respectively. The evidence from the glass layer on the inner surface of the majority of the crucible fragments clearly demonstrates their function to melt glass in preparation for hot working and bead production, indicated also from the substantial quantity of bead working waste associated with the crucible fragments. This does not, however, answer the question whether the crucibles were also used to produce the glass from its raw materials, and at what stage of the process the colorants were added.

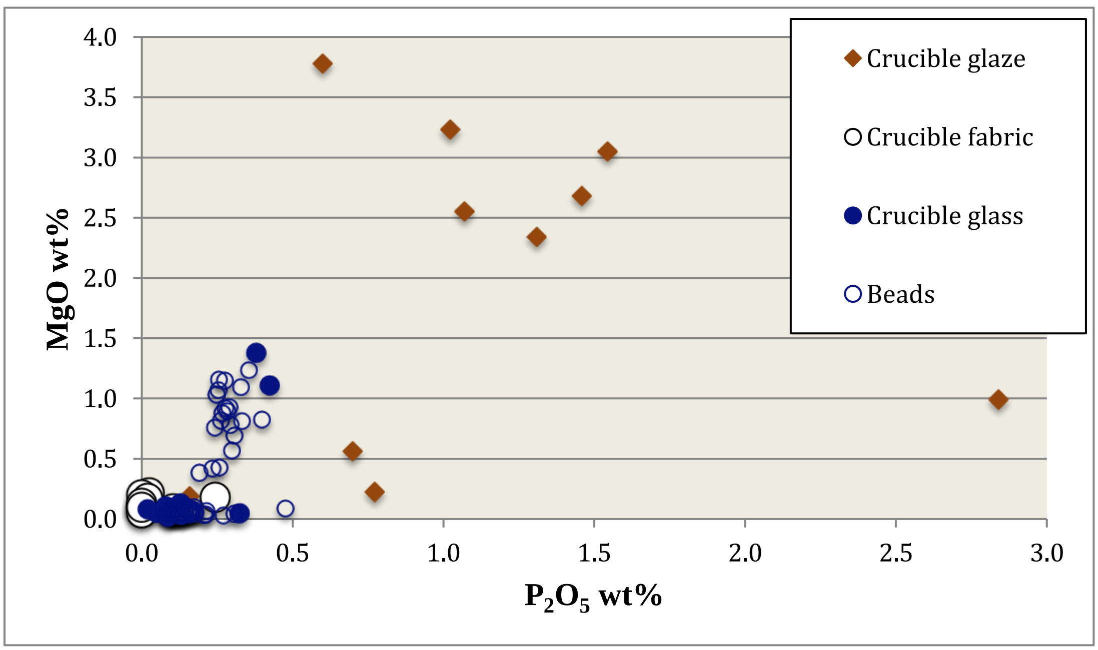


Figure 21. Comparing the magnesium vs phosphorus content of the areas in the crucibles with the glass beads, showing much higher values of both oxides in the outer glaze, typical for fuel ash.

A case for primary glass production?

Primary glass making leaves very few traces in the archaeological record, unless large-scale furnaces are involved, e.g. tank or dome furnaces (Kock & Sode 1994; Gorin-

Rosen 2000). In the absence of *in-situ* furnaces, evidence of semi-finished glass, compositional variation across the crucible fabric, and thorough microstructural examination of the matrix could provide some clues (Jackson et al. 2003; Rehren et al. 2005; Smirniou & Rehren 2011). Results of the analysis here potentially show that some form of primary production took place at or near the site, based primarily on the relatively frequent presence of quartz grains in the matrix of the glass attached to the inner surfaces of the crucibles (Fig.17). Given the refractoriness of the crucible fabric, it is unlikely that the quartz grains are remains of partly absorbed crucible material. Instead, we believe that the quartz grains are residue from unmelted raw material that was fed into the crucible at an early stage of production. As the quartz grains were only observed in about 30% of the samples analyzed, it is possible that some selected crucibles were used for primary glass production and other were not.

Another indication for primary glassmaking in these crucibles is the elevated level of soda in the crucible fabric near the contact to the glass melt, increasing from an average of less than 1 wt% in the center of the wall fragments, to more than 2 wt% near the glass-coated inner surface. Significantly, the lime content remains unchanged, at around 0.1 to 0.2 wt% CaO; given that the actual glass has in the order of 8 to 17 wt% CaO but only a fraction of this in soda, it is clear that this elevated soda content in the crucible fabric is not due to mechanical seepage of molten glass into the ceramic, but a selective penetration of soda. Detailed studies on LBA glassmaking and glass-working crucibles have shown that the selective migration of soda from the glass melt into the crucible fabric is a good indicator for primary glassmaking (Merkel & Rehren 2007; Smirniou & Rehren 2016). This increased soda content is seen in all analyzed crucible fragments, potentially indicating that all crucibles were used for glass making as well as glass working. Alternatively, primary production may have taken place in or near Ile-Ife but not necessarily at Igbo Olokun; however, we have no indication for this, and there is no technical reason to separate glassmaking from glass-working. If there was primary glassmaking in early Ile-Ife, then what were the raw materials used and where was the source(s)?

Considering the *metaluminous* composition of the Ile-Ife glass, Freestone (2006: 140) has suggested “an immature granitic sand, or granitic/syenitic rock or pegmatite” material would have been used with the lime possibly derived from the addition of calcium-rich material – e.g. shell. The experimental work by Akin Ige (2010) and the analysis of HLHA glass beads from Osogbo-Ile (an archaeological site approximately 45 km north of Ile-Ife) have demonstrated that pegmatite and snail shell were the primary raw materials for Ile-Ife glass (Ogundiran and Ige 2015). Elsewhere, Babalola et al (in press) compare the concentration of the elements in Ile-Ife glass with the pegmatite source available near Ile-Ife (Akintola *et al.* 2011, 2012). The comparison is fully consistent with the hypothesis that the Ile-Ife HLHA glass was made with pegmatite as the source of all oxides apart from lime and the colorants. This also explains the low MgO, P_2O_3 and Cl content of the glass.

Coloration of glass in the crucibles

The presence of swirls or *schlieren* of different color in the glass matrix is good evidence that these crucibles were used to add colorants to the glass (Rehren *et al*. 2012: 82). These swirls, as seen in BSE images of the inner glass of sample IF0074 (Fig. 17) and IF0077 (Fig. 22) indicate incomplete mixing of colorants and glass.

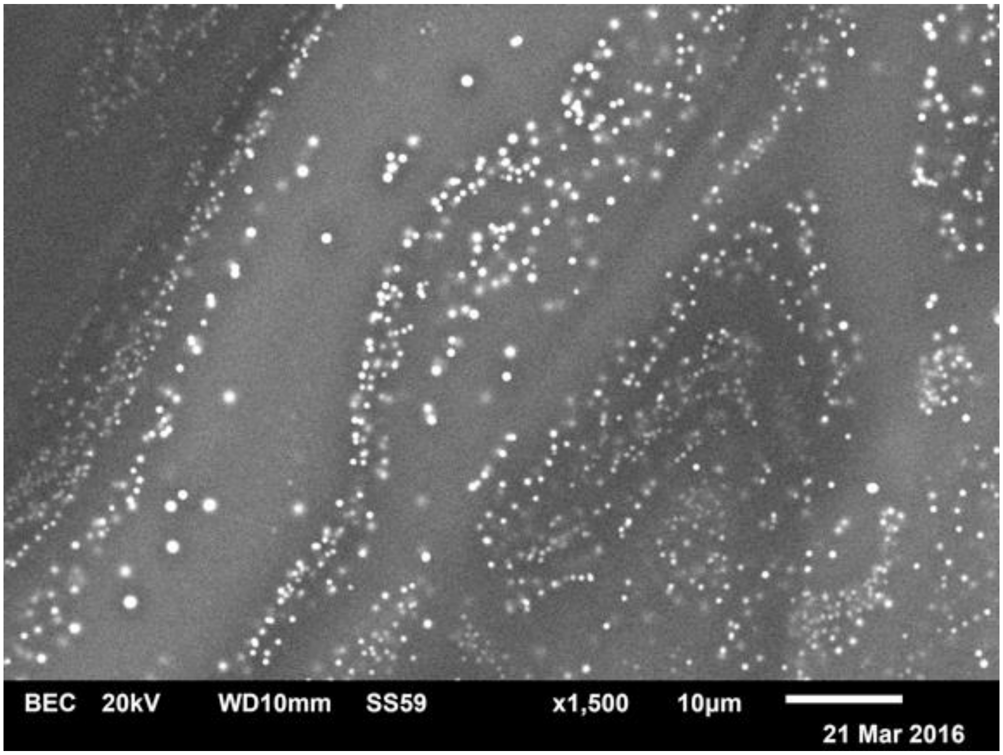


Figure 22. BSE image of copper particles in the inner red glass of a crucible from Igbo Olokun (sample IF0077). The lighter area in form of swirls is an indication of incomplete mixing of the colorant, in this case, metallic copper, into the glass matrix.

Furthermore, the presence of multiple colors, typically shades of the same color, in the inner glass of the crucible may have resulted from the reaction of colorant oxides under different furnace conditions (Goffer 2007). For example, the presence of copper oxide in a glass batch gives a blue color in oxidizing conditions and red under reducing conditions. Similarly, iron oxide in glass melt under reducing conditions can result in either black or green glass. Regardless of whether the multiple colors found in these crucibles resulted from incomplete mixing or variable furnace conditions, it suggests that colorants were being added into the glass melt at Igbo Olokun. Although colorants can be added at different stages during the glass production process, we assume that at Igbo Olokun they were added to freshly-made raw glass rather than recycled cullet, since very little uncolored glass is known from the site.

Crucible typology, scale of production, and the trade of the HLHA glass

With their ovoid- or barrel-shaped form and narrow opening, the Ile-Ife glass crucibles are distinctive from other known glass-melting crucibles, such as the cylindrical glass crucibles from LBA Egypt (Turner 1954; Rehren & Pusch 1997; Rehren 1997; Pusch and Rehren 2007), the wide-mouthed vessels used in Roman glass-working (Cool *et al.* 1999; Cholakova & Rehren 2014), and the early modern glass crucibles from Central Europe (Eramo 2006). The early modern brass cementation crucibles from SE Germany seem to be the closest in shape to the Ife crucibles, yet they are much bigger with distinct lids and not related to glass working (Martinón-Torres & Rehren 2002). Guided primarily by the more complete vessels described above and the wall thicknesses of the fragments from Igbo Olokun, we loosely classify the Ile-Ife crucibles into two types based on their size (Fig. 23, see Table 1).

Type A consists of larger vessels with heights varying from 25 to 35 cm. They are ovoid in shape, with a very restricted orifice that in some cases slopes outward to an expanded shoulder. Wall thickness is mostly 2cm and above. The wear marks on the rim indicate that they were most likely lidded. The average useable capacity of type A is estimated to between 3 and 7 liters translating to about 7.5 to 17.5 kg of finished glass. Four out of the ten complete and near complete examples (Table 1) belong to this type.

Type B is smaller, with a height between 16 cm and 22 cm, and a barrel shaped body. The wall thickness is usually 2cm or less. The opening is less restricted than in type A. It is uncertain whether crucibles of this type were lidded. The useable volume of type B crucibles is estimated to vary from half a liter to a maximum of about 3 liters, equivalent to 1.2 to nearly 8 kg finished glass. There are five examples of type B among those studied from Willett's catalogue and the British Museum collection. The volume of one of the samples could not be estimated because of incomplete information; however, the shape and the available information suggest that it most likely belongs to type B.

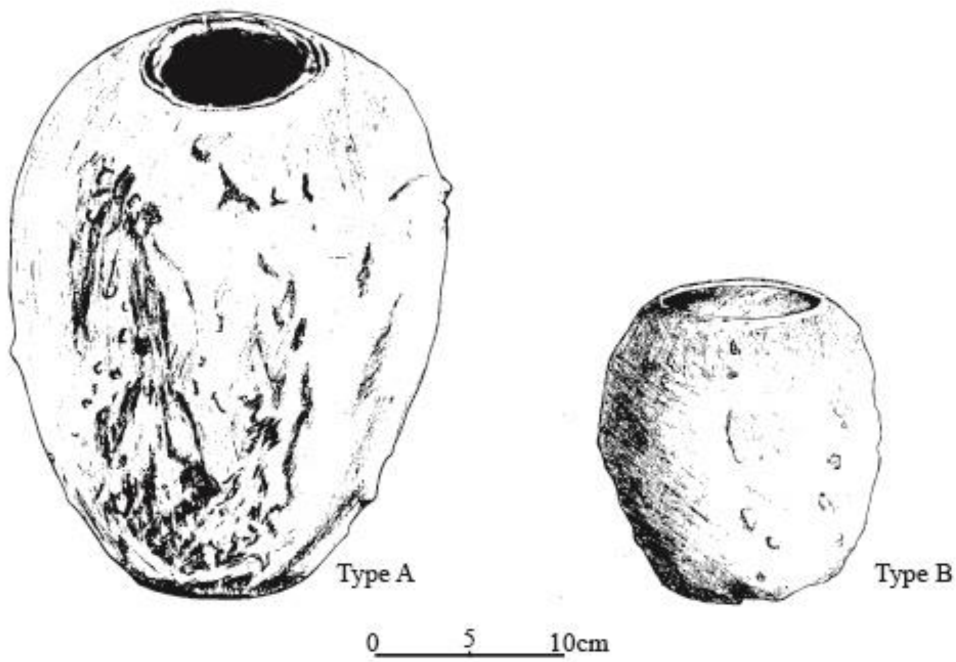


Figure 23. Schematic drawing of the recognized Ile-Ife glass crucible types, showing the difference in size. There is variation in the overall morphology of the crucible beyond what is represented in these drawings. The scale is estimated based on images in Willett (2004). (Drawing by Oluseyi Agbelusi).

The less common glass colors such as black and red rarely occur on thick-walled fragments (see Fig. 11). This may suggest that smaller crucibles were more often used to produce black and red glass than the larger vessels. However, this is not a strict relationship, and blue to green glass dominates in both crucible types. From the ten complete and near complete crucible examined, there is one example of type A (T829) with red glass and another (T834) of type B with blue glass. This situation makes it challenging to decipher a "color-size" dichotomy. Further study of assemblages with larger and near complete fragments would help to determine if there is a significant correlation of glass color and crucible type. It is also possible that crucible morphology changed over time. To assess this, we need more extensive excavations that recover larger crucible fragments from clearly stratified and datable contexts.

The estimated usable volumes of the two crucible types indicate a very significant scale of bead production. The most common beads from the excavations at Igbo Olokun are <5mm in diameter and weigh approximately 0.1 gram; thus, a crucible with 1 liter of usable volume would have held 2.5 kg of glass, equal to 25,000 finished beads. The larger crucibles had total volumes exceeding 5 liters; if half of this volume were filled with liquid glass this would equal between 6 and 6.5 kg glass, enough to produce 60,000 to 65,000 small beads of 0.1 gram each. To estimate the scale of production of glass beads at Igbo Olokun, two extreme scenarios are considered with the assumption that the excavated crucible vessels were used in this same time period. If all 36 crucibles represented in the assemblage from Igbo Olokun belong to type B crucible (small) with useable capacity between half a liter and 3 liters, and were only used once, the craftsmen would have produced a minimum of 18 kg and maximum of 108 kg of finished glass. From this, between 180,000 and 1,080,000 <5mm beads could have been made. If, however, the 36 crucible vessels were all type A, between 1.4 and 6.1 million small glass beads could have been made, and potentially a multiple of this if the crucibles were used for multiple melting episodes. This, together with the about 13,000 beads recovered from the excavations and the fact that only a very small proportion of the entire workshop area has been excavated shows the truly industrial scale of glass bead making at Igbo Olokun, even allowing for the production remains having accumulated over several centuries. No other archaeological site in sub-Saharan Africa has produced evidence of early primary glass bead production on this scale.

We have preliminary indications from early to mid-2nd millennium AD West African sites that HLHA glass beads were traded widely. They have been found in Mali (Davison 1972:272; Nixon 2017; Lankton 2017), Mauritania (Davison 1972:268), Senegal (Dussubieux, pers. comm.), Burkina Faso (Magnavita 2009), Niger (Magnavita 2016), and Igbo Ukwu in southeastern Nigeria (Fenn et al. 2009) (Fig. 24). How much of the Ile-Ife HLHA glass reached these centers is still unclear as only a few HLHA beads have been identified from most of these sites, with the exception of Igbo Ukwu (Lankton *et al.* 2006; Robertshaw, *pers. comm*.).

Although there is so far only limited compositional evidence to demonstrate the circulation and/or trade of the Ile-Ife glass beads within the Yoruba-Edo speaking region, historical and oral traditional evidence unequivocally state Ile-Ife as a major, if not the sole, supplier of beads across the region (Egharevba 1968; Horton 1979; Ogundiran 2002). In addition, there is no archaeological evidence of glass-working technology outside Ile-Ife prior to the 15th century. The recently discovered glass-working site in Osogbo, a town about 45 km north of Ile-Ife, dated to the 17th–18th century provided the first evidence of HLHA glass-working beyond the Ife territory (Ogundiran 2014). The composition of the glass debris and glass beads from Osogbo closely matches the Ife glass (Ogundiran & Ige 2015), firmly linking its glass-working technology to that of Ile-Ife.

Fig. 24. West African sites with HLHA glass beads. 1. Diouboye (Dussubieux pers comm); 2. Kumbi Saleh (Davison 1972); 3. Essouk (Nixon 2016); 4. Gao (Robertshaw *pers comm*); 5. Kissi (Robertshaw *et al* 2009); 6. Elmina (Brill & Stapleton 2012); 7. Igbo Ukwu (Lankton *et al*. 2006; Robertshaw in prep): 8. Bura (Magnavita 2016). The star indicates the production site of Ile-Ife.

Conclusion

The detailed study of crucible fragments from a known archaeological context, several complete crucibles earlier reported from Ife and currently stored in museums, and samples collected from the surface at Ife has demonstrated that all glass crucibles from Ile-Ife belong to the same Ile-Ife glassmaking tradition. This tradition is dated to Ife's "Classical" Period, 11th - 15th century AD. The crucibles were made with highly refractory clay to meet their functional requirements, including resistance to thermal stress during use and little if any contamination of the glass melt. The use of such highly refractory kaolinitic ceramic is known from contemporary Central Asia and Central

Europe, but is without parallel in sub-Saharan Africa. A tentative typology for the crucibles is proposed, differentiating them by their size and shape. However, more finds, especially larger fragments with complete profiles, are necessary to refine this typology.

The analysis of the crucible glass, wasters, and beads from Igbo Olokun has demonstrated that they all belong to the same glass technology, which further strengthens the argument that the crucibles were used in glass-working, and possibly glassmaking. The occurrence of quartz grains in the matrix of the inner glass in about 1/3 of all analyzed crucibles and the elevated soda content of the ceramic fabric near the contact to the glass melt indicate that raw materials were been fed into the crucible for glassmaking. The *schlieren* and swirls observed in several cases are direct evidence that colorants were added to the melt in the crucibles.

This study provides unprecedented evidence of pre-15th century production of glass beads from locally made glass in West Africa, using technologically sophisticated crucibles and a unique glass recipe. The estimated production volume for the Igbo Olokun workshop is consistent with the emerging picture that its products circulated both regionally and trans-regionally, firmly establishing Ife-Ile as a major glassmaking center of international significance.

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References

Adeduntan, J. 1985. "Early Glass Bead Technology of Ile-Ife." *West African Journal of Archaeology* 15: 165–71.

Akintola, A. I., Omosanya, K. O., Ajibade, O. M., Okunlola, O. A., and Kehinde-Philips, O. O. 2011. Petrographic and Geochemical Evaluations of Rare-Metal (Ta-Nb) Potentials of Precambrian Pegmatities of Awo Area Southwestern, Nigeria. *International Journal of Basic & Applied Sciences IJBAS-IJENS, 11 (04), 57-70.*

Akintola, A. L., Ikhane, P. R., Okunola, O. A., Akintola, G. O., and Oyebolu, O. O. 2012. Compositional Features of Precambrian Pegmatites of Ago-Iwoye Area South West, Nigeria. *Journal of Ecology and the Natural Environment,* 4(3), 71-87.

Babalola, A.B. 2015. Archaeological Investigations of Early Glass Production at Igbo Olokun, Ile-Ife (Nigeria). PhD. Dissertation, Rice University.

Babalola, A.B., McIntosh, S.K., Dussubieux, L., and Rehren, Th. 2017. Ile-Ife and Igbo Olokun in the history of glass in West Africa. *Antiquity* 91, 732-750.

Babalola, A.B., Dussubieux, L., McIntosh, S.K., and Rehren, Th. (in press). Chemical analysis of glass beads from Igbo Olokun, Ile-Ife (SW Nigeria): New light on raw materials, production, and interregional interactions. *Journal of Archaeological Science.*

Bandama, F., Moffett, A.J., Thondhlana, T.P., and Chirikure, Sh., 2016. The production, distribution and consumption of metals and alloys at Great Zimbabwe. *Archaeometry* 58, Suppl. 1, 164-181.

Blier, S.P. 2014. *Art and Risk in Ancient Yoruba: Ife History, Power, and Identity, c.1300*. Cambridge University Press.

Brill, R. and Stapleton, C., 2012, *Chemical Analyses of Early Glasses*. Volume 3, Corning Museum of Glass.

Chirikure, Sh., Hall, S. and Rehren, Th. 2015: When ceramic sociology meets materials science: Sociological and technological aspects of crucibles and pottery from Mapungubwe, southern Africa. *Journal of Anthropological Archaeology* 40, 23-32.

Cholakova, A. and Rehren, Th. 2014: Producing black glass during the Roman period – notes on a crucible fragment from Serdica, Bulgaria. In: *Proceedings of the 39th International Symposium for Archaeometry*, Leuven 2012, 261-267.

Cisse, M. 2011. Archaeological Investigations of Early Trade and Urbanism at Gao Saney (Mali). Ph.D., Rice University.

Cissé, M., McIntosh, S. K., Dussubieux, L., Fenn, T., Gallagher, D., and Smith, A. C. 2013. Excavations at Gao Saney: New Evidence for Settlement Growth, Trade, and Interaction on the Niger Bend in the First Millennium CE. *Journal of African Archaeology* 11(1), 9–37.

Cool, H.E.M., Jackson, C.M. and Monaghan, J. 1999. Glass-making and the Sixth Legion in York. *Britannia* 30, 147-162.

Davison, C. C., Giauque, R. D., and Clark, J. D. 1971. Two Chemical Groups of Dichroic Glass Beads from West Africa. *Man*, New Series, 6, (4), 645–659.

Davison, C. 1972. Glass Beads in African Archaeology: Results of Neutron Activation Analysis, Supplemented by Results of X-ray Fluorescence Analysis. Unpublished PhD dissertation, University of California, Berkeley.

Drewal, H.J. 1989. “Ife: Origins of Art and Civilization.” In: *Yoruba: Nine Centuries of African Art and Thought*, ed. R. Abiodun, H.J. Drewal, and J. Pemberton III, pp. 45–74. New York: The Center for African Art.

Egharevba, J.U. 1968. *A Short History of Benin*. Ibadan: Ibadan University Press.

Eluyemi, O. 1975. “New Terracotta Finds at Oko-Eso, Ife.” *African Arts*, 32–92.

Eluyemi, O. 1977. “Excavations at Isoya, near Ile-Ife (Nigeria) in 1972.” *West African Journal of Archaeology* 7: 97–116.

Eluyemi, O. 1986. “Technology of Ife Glass Beads: Excavations at Igbo-Olokun, Ile-Ife, Nigeria,” in *The Social and Economic Contexts of Technological Change*, The World Archaeological Congress, London: Allen and Unwin.

Eluyemi, O. 1987. “The Technology of the Ife Glass Beads: Evidence from Igbo-Olokun.” *Odu* 32: 200–216.

Eramo, G. 2006. The glass-melting crucibles of Derrière Sairoche (1699-1714 AD, Ct. Bern, Switzerland): a petrological approach. *Journal of Archaeological Science* 33, 440-452.

Eyo, E. 1970. “1969 Excavations at Ile-Ife.” *African Arts*, 45–87.

Eyo, E. 1974a. “Excavations at Odo Ogbe Street and Lafogido, Ife, Nigeria.” *West African Journal of Archaeology* 4: 99–109.

Eyo, E. 1974b. “Recent Excavations at Ile-Ife and Owo, and their Implications for Ife, Owo, and Benin Studies.” Ibadan, Nigeria: University of Ibadan.

Fagg, W.B. 1953. “Some Archaeological Problems at Ile-Ife.” Conference Internationale des Africanistes de l’ Ouest. Reunion, Abidjan: Compte rendu, pp. 125-6.

Fagg, W.B., and Willett, F. 1960. “Ancient Ife. An Ethnographic Summary.” *Odu* 8: 21–35.

Fenn,, T. R.., Killick, D. I., Chesley, J., Magnavita, S,, and Ruiz, J. 2009. Contacts Between West Africa and Roman North Africa: Archaeometallurgical Results from Kissi, Northeastern Burkina Faso.” *Crossroads/Carrefour Sahel: Cultural and Technological Developments in First Millennium BC/AD West Africa*,, 119.

Freestone, I. C. 2006. “An Indigenous Technology? A Commentary on Lankton *et al*. ‘Early Primary Glass Production in Southern Nigeria.’” *Journal of African Archaeology* 4 (1): 139–41.

Frobenius, L. 1968 (1913). *Voice of Africa; being an account of the travels of the German Inner-African Exploration Expedition in the years 1910-1912*. First Published 1913. Deutsche Inner-Afrikanische Forschungs Expedition. Blom.

Garlake, P. 1974. "Excavations at Obalara's Land, Ife: An Interim Report." *West African Journal of Archaeology* 4: 111–48.

Garlake, P. 1977. "Excavation at the Woye Asiri Family Land in Ife, Western Nigeria." *West African Journal of Archaeology* 7: 57–96.

Goffer, Z. 2007. *Archaeological Chemistry*. 2nd ed. Hoboken, New Jersey: Wiley.

Gorin-Rosen, Y. 2000. The ancient glass industry in Israel: summary of finds and new discoveries, in: Nenna, M.-D. (Ed.), *La Route du verre. Ateliers primaires et secondaires du second millenaire av. J.-C. au Moyen Age*, Travaux de la Maison de l'Orient 33, Lyon, pp. 49-63.

Horton, R. 1979. "Ancient Ife: A Reassessment." *Journal of the Historical Society of Nigeria* 9 (4): 69–149.

Ige, O. A. 2010a. "Classification and Preservation of Ancient Glass Beads from Ile-Ife, Southwestern Nigeria." presented at the Glass and Ceramics Conservation 2010, Interim Meeting of the ICOM-CC Working Group, Corning, New York.

Ige, O.A. 2010b. "The Science Behind the Stories: The Adventure of a Museum Geologist." presented at the Inaugural Lecture Series 236, Obafemi Awolowo University, Ile-Ife.

Jackson, C.M., Joyner, L., Booth, C.A., Day, P.M. and Wager, E. 2003. Roman glass-making at Coppergate, York? Analytical evidence for the nature of production. *Archaeometry* 45, 435-456.

Killick, D., 2016. A global perspective on the pyrotechnologies of Sub-Saharan Africa. *Azania: Archaeological Research in Africa*, *51*(1), pp.62-87.

Kock, J. and Sode, T. 1994. *Glass, Glassbeads and Glassmakers in Northern India*. THOT Print Publisher, Vanlose, Denmark.

Lankton, J., Ige, O.A. & Rehren, Th. (2006): Early primary glass production in southern Nigeria. *Journal of African Archaeology* 4, 111-138.

Lankton, J. 2017 in Nixon 2017 forthcoming JAA Monograph

Magnavita, S. 2009. Sahelian crossroads: some aspects on the Iron Age sites of Kissi, Burkina Faso. In S. Magnavita, L. Kote, P. Breunig & OA Ide (eds), *Crossroads. Cultural and technological developments in first millennium BC/AD West Africa*. Journal of African Archaelogy Monograph Series, 2, pp.79-104.

Magnavita, S. 2016. Early trans-Saharan trade in the light of archaeometric research. Paper delivered at Society for Africanist Archaeologists (SAFA) biennial meeting, Toulouse, France, 28th June.

Martinón-Torres, M. & Rehren, Th. 2002. Agricola and Zwickau: theory and practice of Renaissance brass production in SE Germany. *Historical Metallurgy* 36, 95-111.

Martinón-Torres, M., and Rehren, Th. 2009. "Post-Medieval Crucible Production and

Distribution: A Study of Materials and Materialities." *Archaeometry* 51: 49–74.

Martinón-Torres, M., and Rehren, Th. 2014. "Technical Ceramics." In B.W. Roberts and Ch. Thornton (eds) *Archaeometallurgy in Global Perspective*, 107–31. Springer, New York.

Martinón-Torres, M., Freestone, I.C., Hunt, A. and Rehren, Th., 2008. Mass-produced mullite crucibles in medieval Europe: manufacture and material properties. *Journal of the American Ceramic Society*, *91*(6), pp. 2071-2074.

Merkel, S. and Rehren, Th, 2007. Parting layers, ash trays and Ramesside glassmaking: an experimental study. In: Pusch, E.B. and Rehren, Th. (eds) *Hochtemperatur-Technologie in der Ramses-Stadt Rubinglass fuer den Pharao* Teil 1 Text, p. 205-225.

Myers, Oliver. 1967. "Excavations at Ife." *West African Archaeological Newsletter* 6: 6–11.

Nixon, S. 2017; forthcoming JAA *Monograph on Essouk excavations*

Ogundiran, A. O. 2002. "Of Small Things Remembered: Beads, Cowries, and Cultural Translations of the Atlantic Experience in Yorubaland." *The International Journal of African Historical Studies* 35 (2/3): 427–57.

———. 2003. "Chronology, Material Culture and Pathways to the Cultural History of the Yorùbá-Ẹ̀dó Region, 500 BC-AD 1800." *Sources & Methods in African History; Spoken, Written, Unearthed*, 33–79.

———. 2014. "The Making of an Internal Frontier Settlement: Archaeology and Historical Process in Osun Grove (Nigeria), Seventeenth to Eighteenth Centuries." *African Archaeological Review* 31 (1): 1–24.

Ogundiran, A.O. and Ige, O.A. 2015. "'Our Ancestors Were Material Scientists' Archaeological and Geochemical Evidence for Indigenous Yoruba Glass Technology." *Journal of Black Studies* 46 (8): 751–72.

Ògúnfólákan, A. 1990. "Luwo Potsherd Pavements in Ile-Ife." *Нигерия: тридцать лет независимости. М*, 87–93.

———. 1994. "Archaeological Investigation of Osun North-East, Osun State." MA Thesis, University of Ibadan.

Ozanne, P. 1969. "A New Archaeological Survey of Ife." *Odu New Series* 1: 28–45.

Pusch, E.B., and Rehren, Th. 2007. *Hochtemperatur-Technologie in der Ramses-Stadt Rubinglass für den Pharao - Teil 1 Text*. Vol. 1. Hildesheim: Gerstenberg.

Rehren, Th. 1997. Ramesside glass colouring crucibles. *Archaeometry* 39, 355-368.

Rehren, Th. 2008: A review of factors affecting the composition of early Egyptian glasses and faience: alkali and alkali earth oxides. *Journal of Archaeological Science* 35, 1345-1354.

Rehren, Th. and Freestone, I.C. 2015: Ancient glass: from kaleidoscope to crystal ball. *Journal of Archaeological Science* 56, 233-241.

Rehren, Th. and Pusch, E.B. 1997. “New Kingdom Glass-Melting Crucibles from Qantir-Piramesses.” *The Journal of Egyptian Archaeology*, 127–41.

Rehren, Th. and Papachristou, O. 2003: Similar like white and black: a comparison of steel-making crucibles from Central Asia and the Indian subcontinent. In: Th. Stoellner, G. Körlin, G. Steffens & J. Cierny (eds), *Man and Mining*, (=Der Anschnitt, Beiheft 16, Bochum), 393-404.

Rehren, Th. and Yin, M. 2012: Melt formation in lime-rich proto-porcelain glazes. *Journal of Archaeological Science* 39, 2969-2983.

Rehren, Th., Spencer, L. & Triantafyllidis, P. 2005: The primary production of glass at Hellenistic Rhodes. In: H. Cool, ed, *Annales du 16e Congres de l'Association Internationale pour l'Histoire du Verre*, Nottingham, 39-43.

Rehren, Th., Cholakova, A. and Zivanovic, M., 2012: The making of black glass in Late Roman Doclea, Montenegro. *New Antique Doclea* 3, 75-90.

Robertshaw, P., Magnavita, S., Wood, M., Melchiorre, E., Popelka-Filcoff, R.S., Glascock, M.D., 2009. Glass beads from Kissi (Burkina Faso): chemical analysis and archaeological interpretation. In: Magnavita, S., Koté, L., Breunig, P, Idé, O.A. (Eds.), *Crossroads: Cultural and Technological Developments in 1st Millennium BC/AD West Africa*. Journal of African Archaeology Monograph Series 2. Africa Magna Verlag, Frankfurt a. M, pp. 105-118.

Smirniou, M. and Rehren, Th. 2011. Direct evidence of primary glass production in Late Bronze Age Amarna, Egypt. *Archaeometry* 53, 58-80.

Smirniou, M. and Rehren, Th. 2016: The use of technical ceramics in early Egyptian glass-making. *Journal of Archaeological Science* 67, 52-63.

Tanimoto, S. and Rehren, Th. 2008. Interactions between silicate and salt melts in LBA glassmaking. *Journal of Archaeological Science* 35, 2566-2573.

Turner, W.E.S. 1954. “Studies of Ancient Glass and Glassmaking Processes. I. Crucibles and Melting Temperatures Employed in Ancient Egypt at about 1370 BC.” *J. Soc. Glass Technol* 38: 436–44.

Willett, F. 1960. “Ife and Its Archaeology.” *The Journal of African History* 1 (2): 231–48.

Willett, F. 1967. *Ife in the History of West African Sculpture*. McGraw-Hill Book Company.

Willett, F. 1977. *Baubles, Bangles and Beads: Trade Contracts of Mediaeval Ife*. Melville J. Herskovits Memorial Lecture 13. Edinburgh: Printed by the University of Edinburgh for the Centre of African Studies.

Willett, F. 2004. *The Art of Ife a Descriptive Catalogue and Database*. Glasgow: Hunterian Museum and Art Gallery.

Wood, M., Dussubieux, L., and Robertshaw, P. 2012. “Glass Finds from Chibuene, a 6th to 17th Century AD Port in Southern Mozambique.” *South African Archaeological Bulletin* 67 (195): 87–99.

Wood, M., Panighello, S., Orsega, E., Robertshaw, P., van Elteren, J., Crowther, A., Horton, M. and Boivin, N. 2016. Zanzibar and Indian Ocean trade in the first millennium CE: the glass bead evidence. *Archaeol Anthropol Sci* online DOI 10.1007/s12520-015-0310-z

Yin, M., Rehren, Th. and Zheng, J., 2011. The earliest high-fired glazed ceramics in China: the composition of the proto-porcelain from Zhejiang during the Shang and Zhou periods (c. 1700–221 BC). *Journal of Archaeological Science* 38, pp.2352-2365.