Chemical analysis of glass beads from Igbo Olokun, Ile-Ife (SW Nigeria): New light on raw materials, production, and interregional interactions

Abidemi Babatunde Babalola [a, *], Laure Dussubieux [b], Susan Keech McIntosh [a], Thilo Rehren [c, d]

[a] Rice University, Houston, TX, USA
[b] Field Museum, Chicago, IL, USA
[c] Science and Technology in Archaeology Research Center, The Cyprus Institute, Nicosia, Cyprus
[d] UCL Institute of Archaeology, London, UK

ARTICLE INFO

ABSTRACT

The site of Igbo Olokun on the northern periphery of Ile-Ife has been recognized as a glass-working workshop for over a century. Its glass-encrusted crucibles and beads were viewed as evidence of secondary processing of imported glass until the high lime, high alumina (HLHA) composition of the glass was recognized as unique to the region. Archaeological excavations conducted at Igbo Olokun recovered more than twelve thousand glass beads and several kilograms of glass-working debris. Fifty-two glass beads from the excavated assemblage were analyzed by laser ablation-inductively coupled plasma-mass spectrometry (LA-ICP-MS) and scanning electron microscopy-energy-dispersive X-ray spectroscopy (SEM-EDS) to understand the chemical characteristics of the Igbo Olokun glass beads in comparison with previously analyzed beads. The analyses affirm the prevalence of HLHA glass beads, and provide firm evidence of a new compositional group characterized by low lime, high alumina (LLHA); no imported soda-lime glass beads were among the analyzed samples. The evidence from crucibles indicates that LLHA glass was worked together with HLHA glass at Igbo Olokun and may have been made locally as part of the same technological tradition. Most likely, granitic sand with or without added calcium carbonate was used to produce these two types of glass, and colorants rich in MnO, Fe_2O_3, CuO, and CoO were intentionally added. Its occurrence in other West African societies, and the presence of some soda-lime glass beads in other sites in Ile-Ife suggest that Ife was involved in regional and inter-regional networks during the early to mid 2nd millennium AD and possibly earlier.

© 2017 The Authors. Published by Elsevier Ltd. This is an open access article under the CC BY-NC-ND license (http://creativecommons.org/licenses/by-nc-nd/4.0/).

1. Introduction

Research on the chemical composition of glass beads from African archaeological sites has increased substantially over the past decade. The earliest known beads from sub-Saharan sites date to the final centuries BC (McIntosh, 1995; Ozainne, 2013). Many of the analyzed beads from pre-15th century sites align chemically with glass from known production areas in the eastern Mediterranean, the Middle East, India, or, more rarely, East Asia, informing us on far-flung trade connections (e.g., Dussubieux et al., 2008; Robertshaw et al., 2009, 2010; Wood, 2016). However, a distinctive compositional group characterized by very high lime and alumina (HLHA) concentrations has been recognized as unique to southwestern Nigeria and a likely product of local primary glass production (Lankton et al., 2006). Most of the analyzed beads in this HLHA group were recovered from various sites in and around Ile-Ife (Fig. 1), a town that traditions identify as the birthplace of the Yoruba people and their deities. In 2011-2012, excavations by Babalola (2015) at the site of Igbo Olokun on the northern periphery of Ile-Ife recovered thousands of glass beads and associated quantities of glass production debris and crucibles. These finds permit the first detailed description of a large, well-provenienced glass bead assemblage from Ile-Ife, including compositional analysis of a sample of beads and other glass using LA-ICP-MS. Here, we provide

* Corresponding author. Harvard University, Cambridge MA, USA.
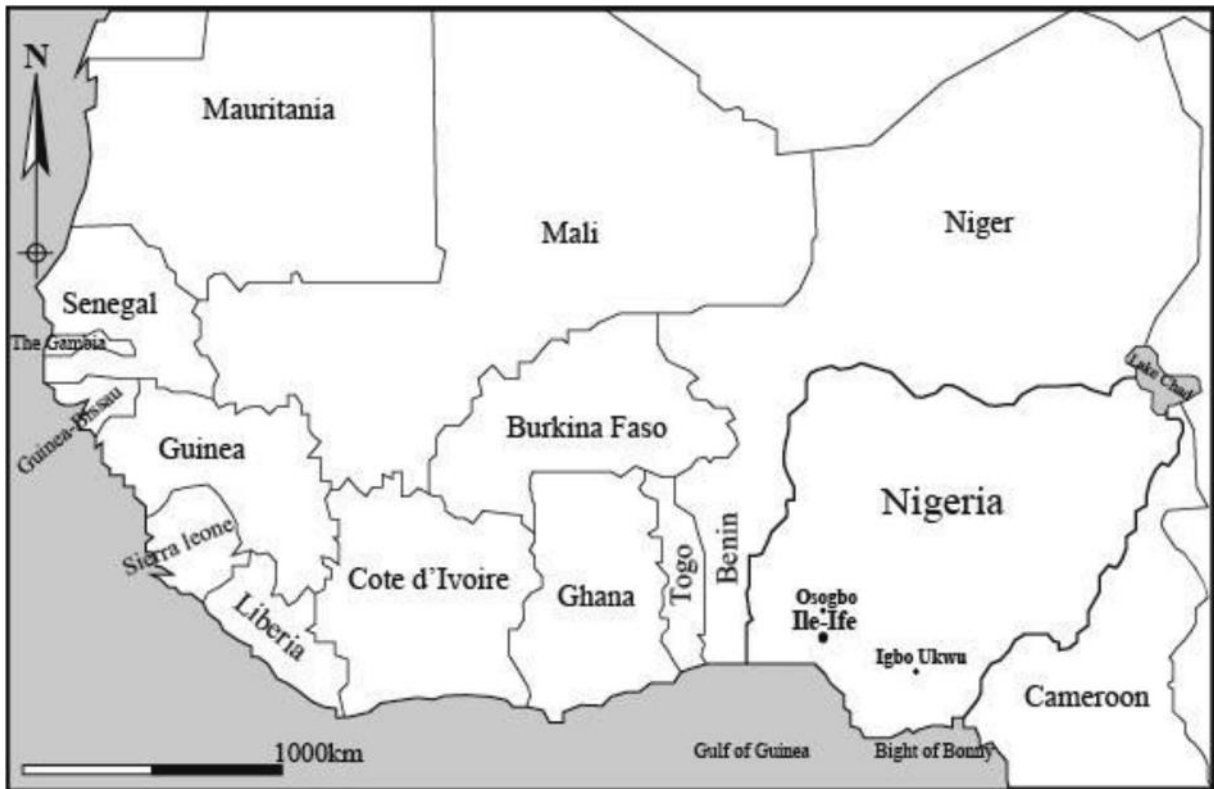E-mail address: tundebabalola@gmail.com (A.B. Babalola).

Fig. 1. Ile-Ife in the context of Nigeria.

those data. In addition, the analysis of the crucibles and other production debris has illuminated the technology involved in processing this distinctive glass to produce the beads (Babalola et al. in press). Results support the suggestion that this HLHA glass could have been made from a raw material such as pegmatite (Freestone, 2006: 140). Beads of HLHA glass have now been identified from a number of sites across West Africa. In the final section, we map their known distribution and discuss the implications for understanding trade connections.

1.1. Ile-Ife and glass in West African history

Ile-Ife has long been associated with glass processing and glass beads. Leo Frobenius (1913) described finding glass encrusted crucibles from his digging of pit shafts in the forested Olokun Grove (Igbo Olokun) (Fig. 2). He also noted that the local population mined the area extensively to locate and recover glass. Glass beads are highly valued and symbolically charged in Yoruba culture today. Beaded crowns denote political and religious authority (Blier, 2014; Adediran, 1992). A number of the famous brass and terracotta heads found or excavated in Ile-Ife wear beaded crowns, as well as bead necklaces, armlets and anklets (Willet 1967; 2004). Many of these items were almost certainly made of glass, as glass beads are found in great profusion in both surface and subsurface contexts and have been found in association with the sculptures themselves (e.g. Eluyemi, 1987). Crucibles and crucible fragments have been found in shrine contexts in Ile-Ife, which suggests their ritual potency. Garlake's (1974, 1977) careful excavation of Woye Asiri and Obalara's Land, for example, recovered glass beads and crucible fragments from well-defined contexts. Associated charcoal samples were dated to the 11th to 15th centuries. These dates are consistent with thermoluminescence dates of the 13th-15th century on fired clay cores of cast brass sculptures from Ita Yemoo, another shrine

site in Ile-Ife (Willett, 1959, 2004; Willett and Fleming, 1976). These are among the most reliable dates that place glass and beaded sculptures at Ile-Ife in the 11th-15th centuries, although Willett (1971, 2004) published several dates on charcoal from Ita Yemoo and Orun Oba Ado that are earlier than or overlap with this timeframe. All have large standard errors of 100–200 years. The Orun Oba Ado dates are particularly problematic as they were run on charcoal collected from throughout the fill of deep pits (Willett, 1971: 366). Based on available data, most scholars identify the 11th-15th centuries as the period of Ile-Ife's fluorescence, characterized by specialization and technical sophistication in the crafts of brass casting and glass working (e.g. Eyo, 1974; Ogundiran, 2005; Blier, 2014).

Few investigations to date have furnished detailed descriptions of the excavated glass beads, cullet, or crucibles found in such abundance at sites in Ile-Ife. Igbo Olokun was the focus of extensive digging by Willett, Fagg, and Murray in the 1950s in an unsuccessful attempt to find undisturbed pit deposits like those described by Frobenius (Willett, 1960). The amount of material recovered from the eighty shafts they dug must have been enormous, but it was never published. Eluyemi (1987) and Adeduntan (1985) excavated apparent glass workshop areas of Igbo Olokun and Ayelabowo, respectively, and published brief accounts. Eluyemi (1987) was the first to describe all the glass beads he recovered (n = 188). As of 2010 when this project commenced, fundamental questions remained about the production processes carried out at Igbo Olokun, the chronology of change in technology, and the kinds of glass and glass beads involved.

Compositional analysis has provided much of what is known about the glass types present in Ile-Ife. In a pioneering study, Davison (1972) used neutron activation analysis (NAA) and X-ray fluorescence (XRF) to analyze several dozen beads excavated by Willett from Orun Oba Ado, Ita Yemoo, and Igbo Olokun. She
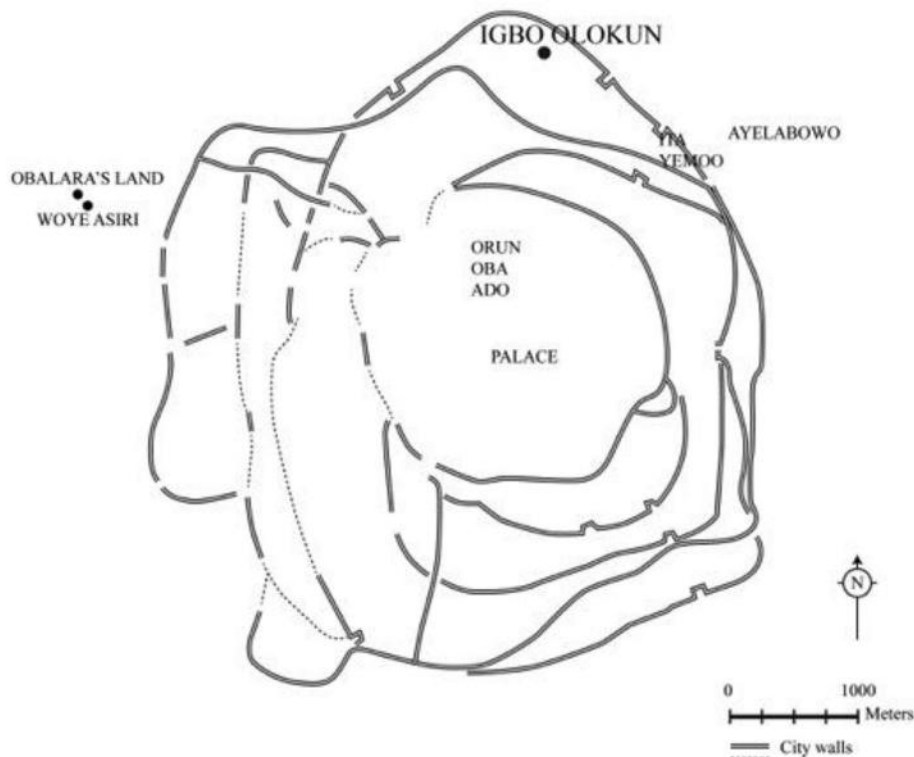
Fig. 2. The location of Igbo Olokun in Ile-Ife showing other sites mentioned in the text.

identified two major compositional groups. One group had soda content above 10 wt%, indicating that Na_2O was used as an alkali flux in the glass recipe. However, the nature of this soda source, typically thought to be either plant ash or mineral natron, remained open since Davison's (1972) analysis of the soda lime glass from Ile-Ife did not report compositional data for potash or magnesia which would have enabled this distinction to be made.

Most of the samples that she analyzed belong to a second major composition group. For Davison, the key characteristics of this group were low levels of soda and significant levels of potassium, which suggested the use of potash from wood ash as a flux (Davison et al., 1971). Wood-ash glass dominated medieval glass-making north of the Alps. Davison thus concluded that these beads most likely originated in medieval Europe (Davison et al., 1971; Davison, 1972: 255-8, 269). For three decades following her pioneering research, all Ife glass was presumed to have been imported and then remelted for local bead production. Only after the development of substantial comparative databases on glass composition (e.g., Brill, 1999; Lankton and Dussubieux, 2006) was the significance of the very high lime and alumina levels, averaging 15 wt% and 13 wt% respectively, recognized as a defining and unique signature of this Ile-Ife glass group (Lankton et al., 2006).

The work of Lankton et al. (2006) provided compositional data using SEM-EDS and electron probe microanalysis (EPMA) on fifteen glass bead samples of probable Ile-Ife origin, which were acquired from different sources including the British Museum and surface collection from Ile-Ife. Some bead samples were extracted from a glass cake (*aje ileke*) of partially melted beads that was purchased from Ife market in the 1990s (Lankton et al., 2006: 114-118). Thus, none of the samples came from known and dated archaeological contexts. Based on the concentrations of the major and minor elements, Lankton et al. identified three major groups: high lime-high alumina (HLHA), high lime-low alumina (HLLA), and soda-

lime glasses. Glass from at least two of these groups was present among the sampled beads from all Ile-Ife locales (Table 1).

Considering the uniqueness of the HLHA composition and its predominance among the samples analyzed, Lankton et al. (2006: 136) argued that this group was likely locally made in or near Ile-Ife, and certainly within southern Nigeria. In a commentary on the article, Freestone (2006: 140) suggested that the high alumina could have come from feldspar and the lime from limestone or shell. Research by Ogundiran and Ige (2015) at Osogbo, a 17th-18th century site located approximately 40 km north of Ile-Ife, recovered HLHA beads and glass production waste. They argue for a continuing technological tradition of glass manufacture using snail shells and local sand derived from pegmatite.

Beads made of distinctive HLHA glass have been identified at a growing number of archaeological sites in West Africa, shedding light on economic connections and extensive trade networks. The chronology of glass bead production in Ile-Ife needs to be more firmly established; based on current evidence, it appears that the beads began to circulate sometime after AD 900. For earlier periods, imported soda-lime glass — primarily plant-ash, but mineral soda beads have also been identified — accounts for nearly all analyzed glass beads from sub-Saharan contexts (Wood, 2016; Cissé et al., 2013; Magnavita, 2016). Questions of when and how the Ife bead industry emerged, and how the scale and technology of production changed through time are of primary interest. To investigate these questions, we undertook excavation, description, dating, and analysis of materials from likely production areas in Igbo Olokun. A general overview of the project and results have recently been published (Babalola et al., 2017; see Babalola, 2015 for details of the excavation results). Here, we report in detail the results of the LA-ICP-MS and SEM-EDS analysis of the glass materials from the 2011-2012 excavations.

Table 1
Major glass composition groups by site within Ile-Ife. Parentheses indicate the number of samples analyzed.

Site	HLHA glass	HLLA glass	Soda-lime glass
Ita Yemoo	Beads (23)		Beads (8)
(Davison, 1972)	Crucible glass (6)		Crucible glass (3)
	Cullet (7)		
Orun Oba Ado	Beads (6)		Beads (5)
(Davison, 1972)			
Ife − unknown provenience	Beads (7)	Beads (2)	Beads (6)
(Lankton et al., 2006)	Crucible glass (2)	Bead roughouts (2)	Cullet (7)
	Cullet (14)	Cullet (11)	
Igbo Olokun	Crucible glass (2)		Bead (1)
(Davison, 1972)	Cullet (5)		(Lankton et al., 2006)

2. The Igbo Olokun excavations

Frobenius (1913) described Olokun Grove as a vast, forested expanse. Willett (1960: 241) estimated its size as three-quarters of a mile by half a mile. Today, owing to land speculation and residential expansion, only a small 21 × 48 m plot remains enclosed and protected by the National Commission for Museums and Monuments (NCMM). During our 2011/2012 field season, four 1 × 3 m units (IO-A, IO-B, IO-C, IO-D) were excavated within or adjacent to the fenced Olokun reserve. These proved to be extremely rich in glass beads and debris, including crucibles. Almost 13,000 beads were recovered, using 1.2 mm mesh for screening. In addition, the excavations yielded over 800 crucible fragments, almost three kilograms of glass waste and cullet, and approximately 14,000 potsherds. Two other units, both measuring 1 × 2 m, were dug at some distance from the NCMM enclosure. Unit IO-E, located 200 m to the south, yielded 101 glass beads and 54 crucible fragments, but virtually no glass waste. Unit OO-A was 1.5 km southwest of the enclosure and produced only 13 beads and five crucible fragments. Units IO-B, IO-C, and IO-D, which provided nearly all the glass described in this article, all showed evidence of pits dug into the deposits at various times (Babalola, 2015). Recent trash pits originated at or near the surface. Other pits began deeper and often penetrated the sterile, compact lateritic clay that underlay the culture-bearing deposits at depths ranging from 0.5 to 1.2 m (Figs. 3–5). These deep pits were filled with moist, clay-rich, dark-brown deposits that contrasted starkly with the reddish-orange, sterile clay.

Three pits with dark fill were encountered in adjoining units IO-B and D (Figs. 3 and 4); the northernmost pit was excavated to a depth of 2.2 m 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 four of the NCMM enclosure units, it contained crucible fragments, pottery, glass beads and glass debris, but not in any notable concentrations, or associated with any elements that would identify the original function of the pit. Willett (1960) believed that these ubiquitous pits at Igbo Olokun were for burials, but if they once contained human bones, those have been destroyed by the moist, acidic soils.

The lack of any spatial focus or notable artifact concentrations, together with the high degree of comminution of both potsherds and crucible fragments throughout these units suggests that the deposits may have been subject to a substantial amount of churning. This is consistent with Frobenius' (1913) account of widespread local quarrying of Igbo Olokun for glass. No features such as ash deposits or intact furnace linings were found in the excavated deposits. However, the abundance of glass production debris and vitrified clay fragments indicate that the excavated areas are in or very near a zone of glass workshops.

The prevalence of disturbed deposits complicates the assessment of chronology. The inverted radiocarbon dates from the deep pit in unit IO-B/D illustrate this. Charcoal from the fill at the top of the pit (0.60 m depth) produced an AMS date of 840 ± 30 BP (1058-1264 cal AD), while charcoal from 1.43 m depth in the same pit dates to 70 ± 30 BP. We believe this latter date comes from the emptying and

Fig. 3. Units IO-B and IO-D (left) and IO-C (right) showing pits and channels dug into the sterile, reddish orange lateritic clay. Dark fill has been removed. (For interpretation of the references to color in this figure legend, the reader is referred to the Web version of this article.)
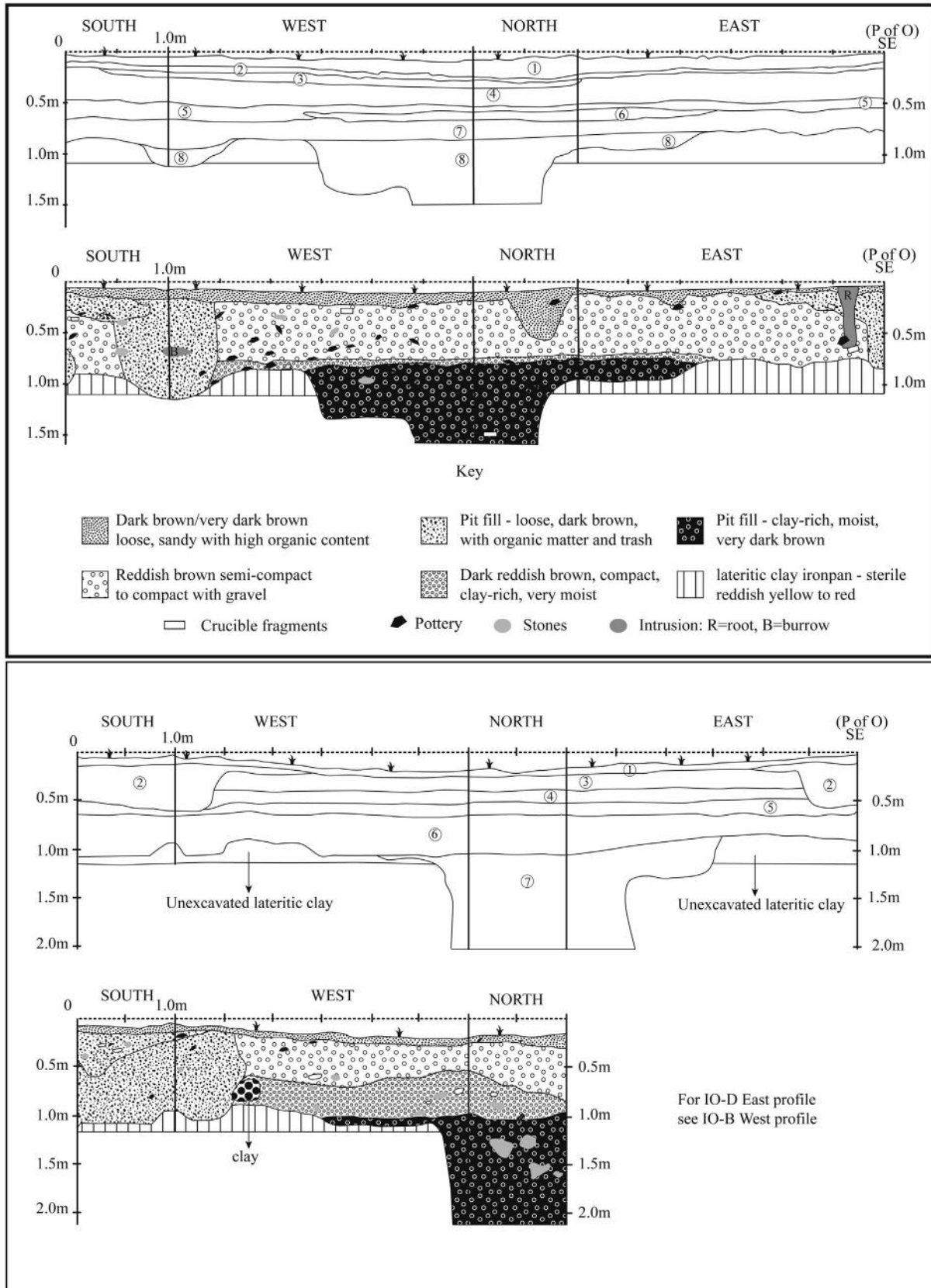
Fig. 4. Relation of excavated levels (numbers) to natural stratigraphy of deposits in profile for unit IO-B (top) and adjoining unit IO-D (bottom).
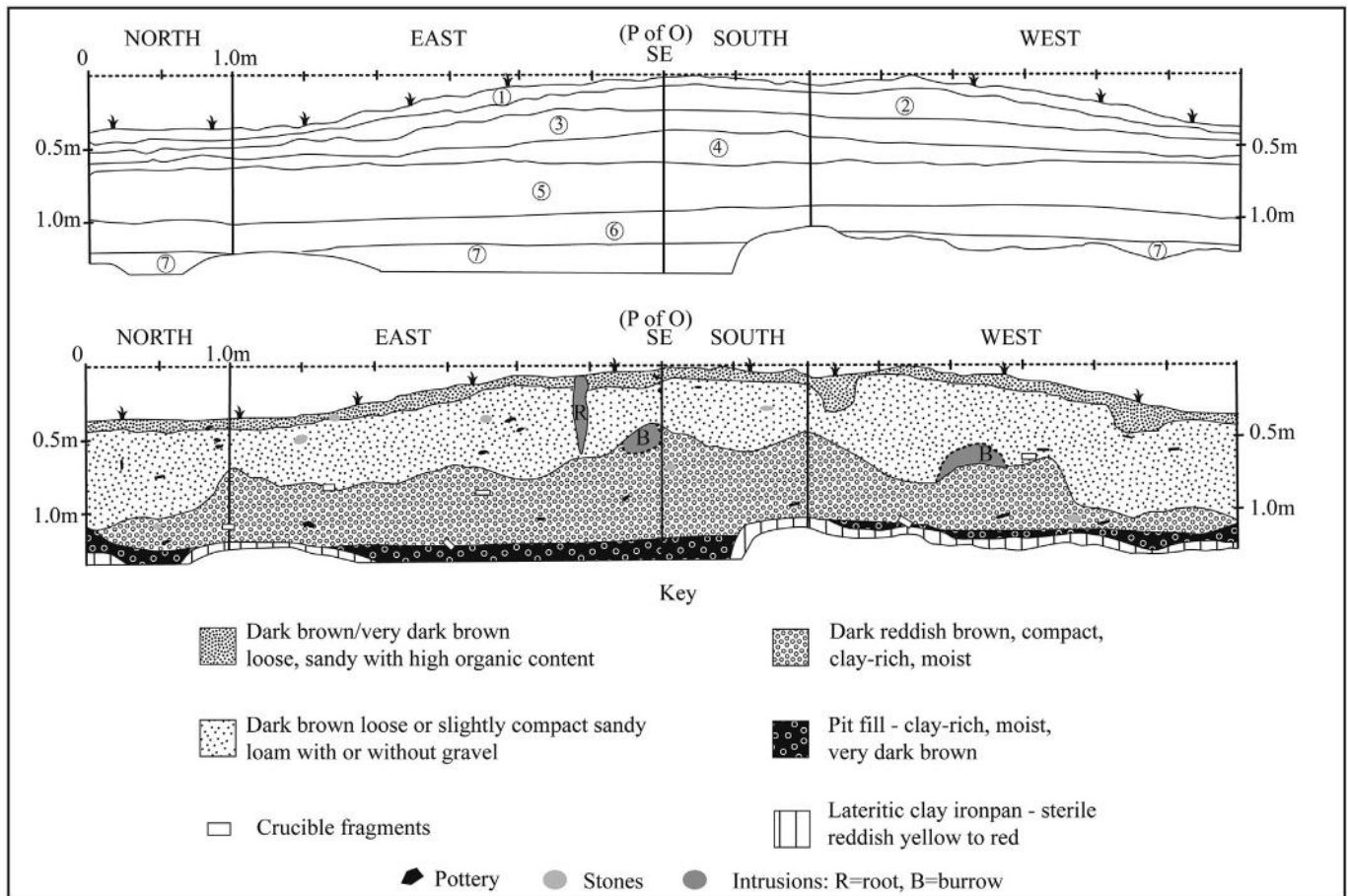
Fig. 5. Relation of excavated levels to natural stratigraphy of deposits in profile for unit IO-C.

refilling of the pit in the 18th-19th century. Dates of 570 ± 30 BP (1304-1423 cal AD) on basal deposits (Level 6) in unit IO-C, and 610 ± 30 BP (1295-1404 cal AD) from the lowest levels of unit OO-A, which contained 4 crucible fragments, combine to support a chronology of the 11th to 15th centuries for the glass beads and workshop deposits. This is consistent with the pottery assemblages from all units, which are dominated by the rim forms and decorative motifs that Garlake (1974, 1977) identified as characteristic of Ile-Ife pottery from the 11th-15th centuries (Babalola, 2015; Babalola et al., 2017). Just east of Igbo Olokun, at Ayelabowo, excavations recovered 300 crucible fragments from a possible glass workshop also dated to this same period (Adeduntan, 1985).

2.1. Overview of the glass assemblage

The glass bead assemblages from units IO-B, IO-C, and IO-D, totaling almost 10,600 beads, were studied in full. All were created by drawing a long tube of glass and cutting it into beads (Fig. 6). The surface of most of the glass beads, especially the unfinished products, is characterized by striations parallel to the perforation, consistent with drawing a glass tube. However, these striations are less visible on well-reheated, finished glass beads. No grinding of the cut ends was detected. Seventy percent of the beads are short cylinders or oblates (following Wood's (2011) descriptive categories); the remainder are tubes, most with cut ends that have not been smoothed by heating. Ninety percent of the beads have diameters under five millimeters. The bead colors include various shades of blue or blue-green (75%), colorless glass or colorless

coated with a reddish-brown glass (10%), green (5%) and yellow or multicolored (2-3%) (Fig. 6). Monochrome, transparent or translucent blue is the dominant color category in all units. Only a few beads in the assemblage are opaque, usually occurring in the infrequent yellow, dark red, and dark gray/black colors. Entirely red glass beads rarely occur; rather red appears as coating on the outside of a colorless core. Striped beads likewise have a colorless or blue core. Dichroic beads, which appear blue in reflected light and yellow-green in transmitted light, constituted only 2.2% of the assemblage. These beads were more prominent in the samples analyzed by Davison (Davison et al., 1971). Corrosion was noted on only 10% of the beads.

Of the 812 crucible fragments recovered, two-thirds have a layer 1-10 mm thick of melted glass on their interior surfaces; in over 90% of these, the glass color is blue, green, or blue-green. Only a handful of rim and base fragments were identified, but these confirmed that the crucibles were of similar shape (ovoid, with a restricted simple rim with a mouth diameter of 8–12 cm, and flat base) to the eight complete crucibles illustrated by Willett (2004). Those crucibles ranged in height from 16 to 35 cm and would have held 2.5–17.5 kg of melted glass (Babalola et al. in press).

We also recovered three kilograms of glass waste that included collapsed tubes, tube ends, and droplets, indicative of the drawing of molten glass. Cut tube ends and miscut bead discs indicate the process of cutting glass canes into beads (Francis, 1991). Overheated and fused beads and finished beads with well-rounded ends are the result of reheating to smooth cut ends.
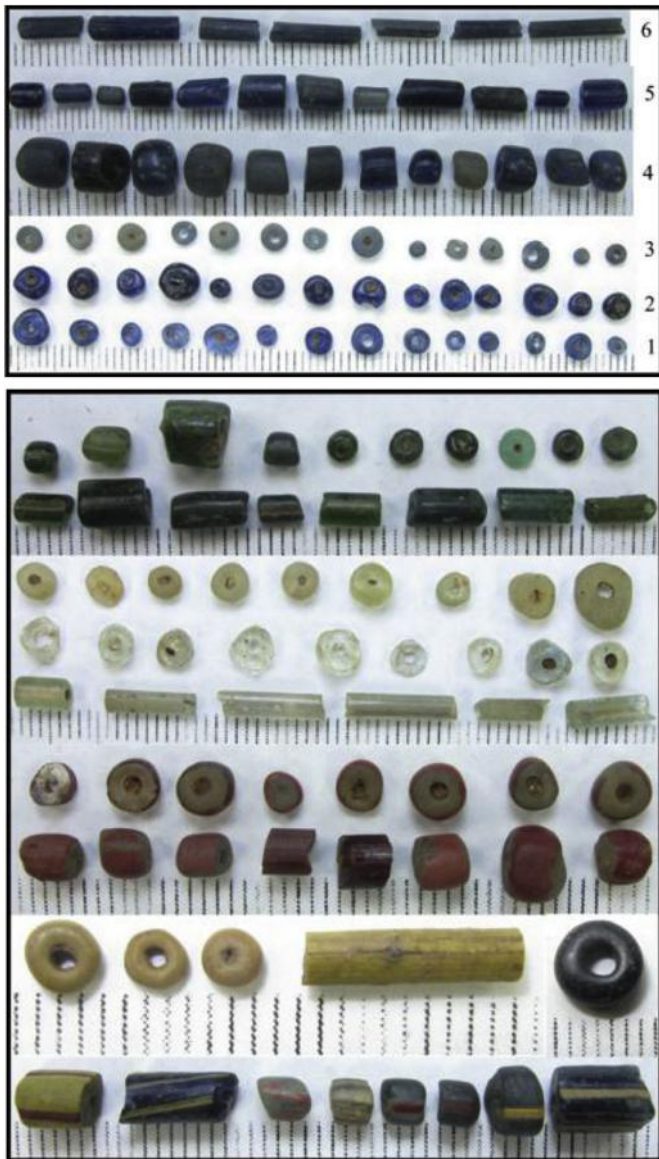
Fig. 6. Glass beads from excavations at Igbo Olokun by color and shape (1, 2, & 3 = oblates [3 = all dichroic]; 4 = cylindrical; 5 = partially or heat treated tubular; 6 = untreated tubular). All beads are monochrome or red on a colorless core except the bottom row of striped beads. (For interpretation of the references to color in this figure legend, the reader is referred to the Web version of this article.)

3. Compositional analysis of the glass

Laser ablation-inductively coupled plasma-mass spectrometry (LA-ICP-MS) was used to analyze forty-nine beads, four unperforated glass canes, ten wasters, and seven samples of glass adhering to crucibles. Results of analyses on the 21 samples of production glass have been published elsewhere (Babalola et al. in press). The bead samples include all shape groups and color categories and are well preserved with a smooth and shiny surface and no evident signs of corrosion (Fig. 7). Those with corroded surfaces were deliberately avoided because corrosion may influence the composition (Dussubieux et al., 2009: 160). Multiple analyses were carried out on samples with multiple colors, for example, those with a clear core and brownish-red coating on the exterior. On striped beads, each stripe color was analyzed separately (Supplementary Tables S1 and S2).

The analyses were done at the Elemental Analysis Facility (EAF) at the Field Museum (Chicago) using an Analytik Jena Inductively Coupled Plasma - Mass Spectrometer (ICP-MS) connected to a New Wave UP213 laser with helium. Two different series of standards were used to measure major, minor and trace elements: the NIST SRM 610 and 612 and the Corning B and D glasses. The detection limits range from 10 ppb to 1 ppm for most of the elements. Accuracy ranges from 5 to 10% depending on the elements and their concentrations. More details on the analytical protocol and the performance of the method used at the EAF are available in Dussubieux et al. (2009).

An additional three beads, all with white patination attributed initially to corrosion, were mounted as polished sections and analyzed by SEM-EDS at the Archaeological Materials Science Laboratory of UCL Qatar. The samples were mounted in epoxy resin, polished (Fig. 8), carbon-coated, and then examined in a JEOL JSM6610LV scanning electron microscope with an Oxford Instrument AZtec Energy Dispersive Spectrometer.

3.1. Description of the glass groups

Based on the results obtained with LA-ICP-MS and SEM-EDS (Supplementary Tables S1 and S2), three different glass groups were identified (Fig. 9). All but three of the samples are distinctively high in alumina with concentrations ranging from 12 to 18 wt%, and can be divided into two sub-groups based on lime concentrations. The HLHA group (following Lankton et al. 's (2006) naming conventions) has high lime concentrations of 12−20 wt%; the LLHA group has lime levels of less than 8 wt%. Three beads have intermediate concentrations of alumina and are assigned to a new, LLMA group. Table 2 reports the average compositions of the three groups.

In the HLHA glass samples, the soda content is low, ranging from 1.7 to 6.4 wt%, while the K_2O concentration significantly varies from 0.7 to 8.5%. The LLHA group has similar potash concentrations compared to the HLHA glass but higher soda concentrations ranging from 4 to 8 wt% (Fig. 10). In both cases, the two constituents seem to be negatively correlated. In the LLHA glass higher P_2O_5 (typically 0.2 to 0.4 wt%), and between 0.5 and 1.3 wt% of MgO and Fe_2O_3 were measured. Interestingly, the content of MgO and P_2O_5 is still below the level expected in regular plant ash or wood ash glass.

Three of the analyzed beads (Fig. 9) have medium alumina (4−<10 wt%) and low lime (≤5 wt%) concentration (LLMA). So far, this compositional type only occurs in yellow and red (IF0048) and black (IF0039) stripes on bead samples with clear and pale blue cores. When other major and minor elements are considered, this group seems to be a sub-group of LLHA with which they share more characteristics than with the HLHA, such as relatively elevated magnesia, phosphate, and iron oxide. However, it is difficult to conclude, at the moment, whether or not the LLMA should be classified as another major group. Perhaps, it could be a variant of either of the two major groups resulting from technical failure, human error, and natural variation/anomalies in the constituents of the raw materials.

3.2. Dichroic glass

One dichroic bead was among the analyzed samples. In ancient glass, dichroism is produced when light is scattered by the presence of colloidal gold and/or silver (Freestone et al., 2007) or by the presence of small immiscible glass droplets in the glass matrix (Kingery et al., 1983; Li et al. 2001). These glass droplets have a different composition (and therefore different refractive index) compared to the surrounding glass and are created by a liquid-liquid micro-phase separation. The size and the refractive indexes
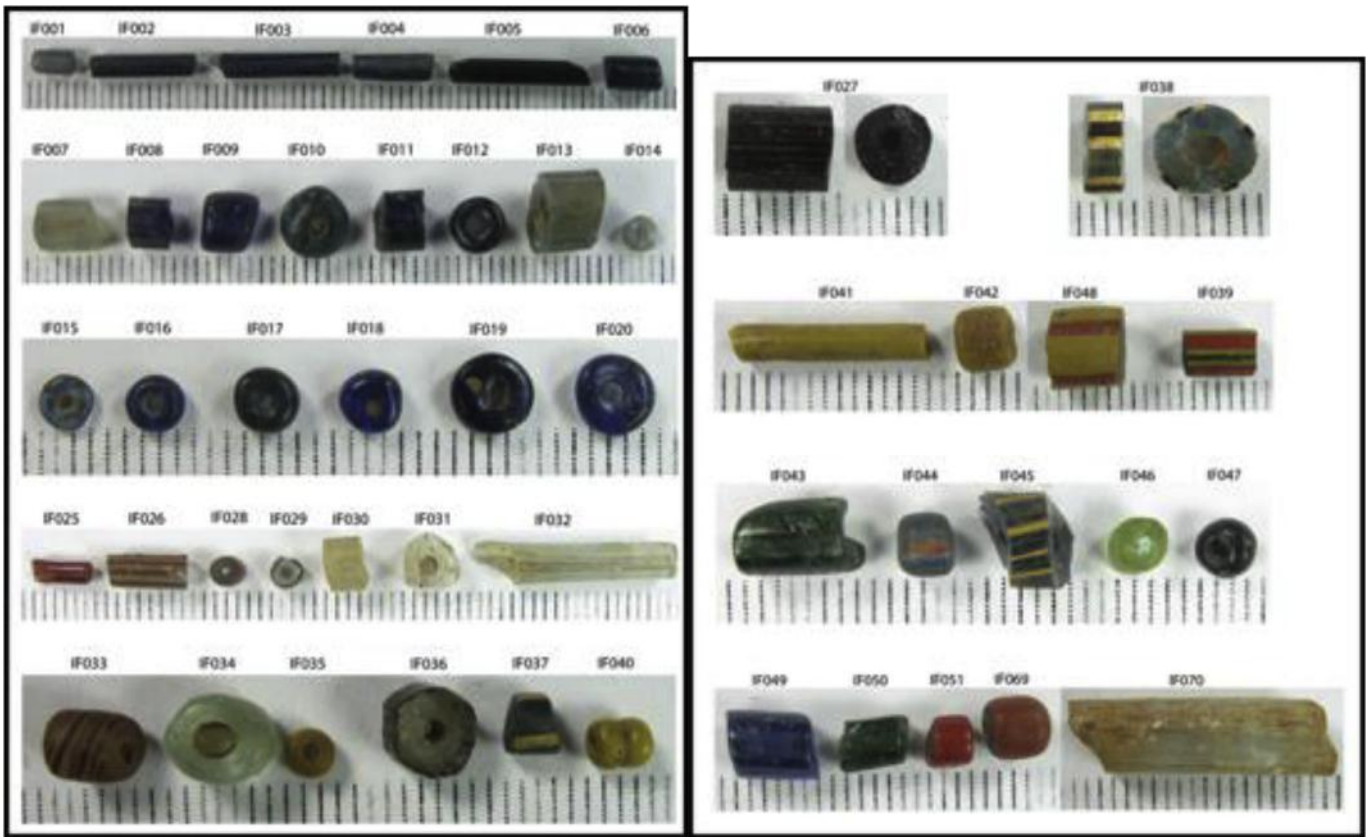
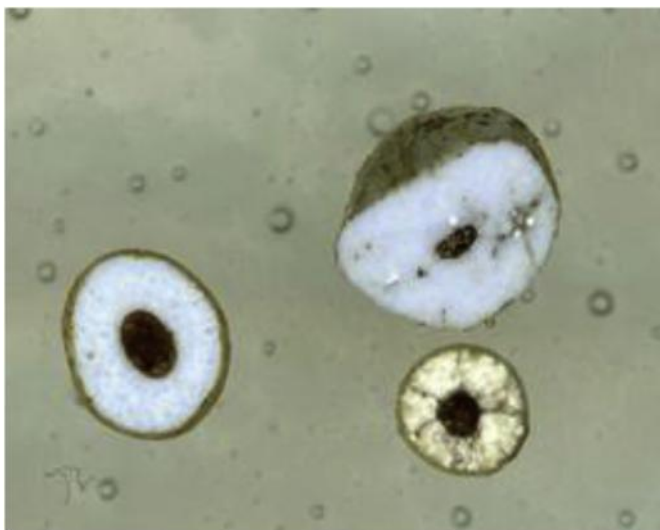**Fig. 7.** Igbo Olokun bead samples analyzed by LA-ICP-MS.

Fig. 8. Resin embedded corroded white glass beads from Igbo Olokun analyzed by SEM-EDS. Left (IF0084), right (top – IF0083A, bottom – IF0083B).
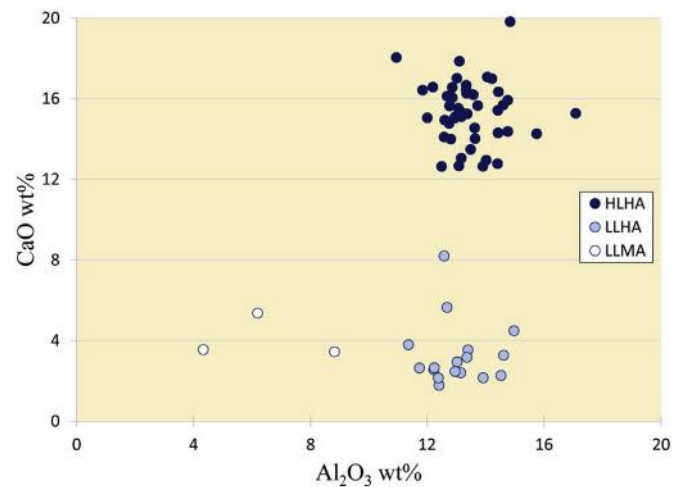

Fig. 9. Alumina vs lime content of the glass beads from Igbo Olokun showing the two major groups and possibly a third group or sub-group with low lime and medium alumina.

of the droplets and glass matrix are two important factors to control dichroism. Kingery et al. (1983) showed that high lime and low alkali glasses were prone to liquid-liquid immiscibility. The dichroic bead from Ile-Ife that was analyzed (IF007) belongs to the HLHA glass group (with a concentration of lime of 16.4 wt% and of alkali of 8.1 wt%). It does not contain any specifically high concentrations of

gold or silver and does not exhibit any differences in its composition compared to the other beads in the same group. It seems that maybe different conditions during the production of the glass produced the dichroism. Hopefully more research will be possible on the Ile-Ife dichroic glass to understand better how this very special glass was produced.

Table 2
Average concentrations (wt%) with standard deviations for the HLHA, LLHA, and LLMA glass groups.

	HLHA	SD	LLHA	SD	LLMA	SD
SiO$_2$	62.3	1.9	68.7	2.6	74.3	1.4
Na$_2$O	3.9	1.2	6.5	1.2	5.6	0.6
MgO	0.06	0.03	0.9	0.2	0.9	0.2
Al$_2$O$_3$	13.5	1.0	12.4	2.4	6.5	2.3
K$_2$O	3.81	2.1	5.1	2.2	4.01	1.4
CaO	15.2	1.5	3.3	1.4	4.1	1.1
Fe$_2$O$_3$	0.6	0.3	2.2	1.1	3.4	0.7
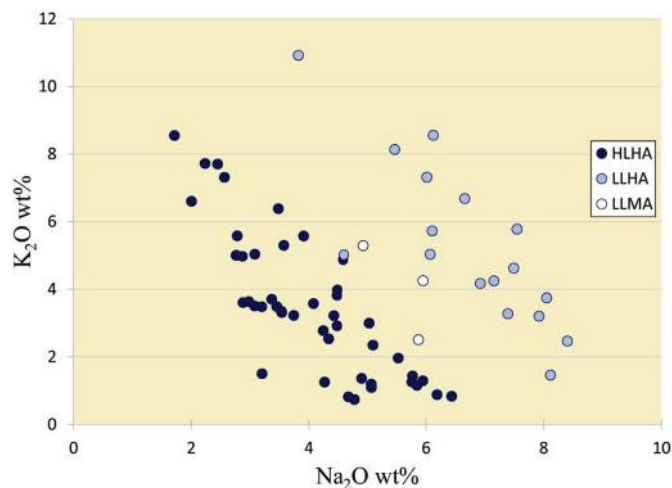
Fig. 10. Soda vs potash concentration of Igbo Olokun glass beads showing a difference in the soda content of the two major groups.

3.3. Coloring and opacifying agents

More than half of the HLHA glass samples contain a significant content of cobalt that produces different shades of dark blue glass. The concentrations for this element range from 200 to 1000 ppm, with a few samples containing between 1600 and 1900 ppm. Higher concentrations of cobalt are connected to higher concentrations of manganese as already noticed by Davison (1972: 255) (Fig. 11) but also to higher concentrations of other trace elements such as lithium, vanadium, nickel, zinc and copper.

The cobalt occurs together with about ten times as much manganese oxide, and about one tenth of the cobalt concentration in zinc and nickel. The good correlation of these elements suggests that they co-occur in the colorant used (Figs. 12 and 13). The correlation of various transition metals such as Mn, Zn, and Ni with Co is known from the cobalt alum used in Late Bronze Age Egyptian glass coloring (Kaczmarczyk, 1986; Abe et al., 2012; Smirniou and Rehren, 2013), but also from many other cobaltiferous minerals, notably wad, a manganese hydroxide that often contains other transition metals as well (e.g. Burlet and Vanbrabant, 2015). The low levels of the transition metal in other HLHA beads, especially the colorless ones, can be used as indicators for the local geological background concentrations of these elements (Smirniou and Rehren, 2013), of about 1000 ppm for MnO and <10 ppm for Co. Ajayi and Suh (1999) discuss transition metal concentrations near Ife, but no cobalt rich deposits have been identified so far.

Other colors in the HLHA glass group are: translucent or transparent pale blue, emerald green, greenish, transparent red, and colorless. Aside from manganese and iron, which can be natural constituents of the sand or purposefully added as a colorant, no other coloring element was identified in those colored glass
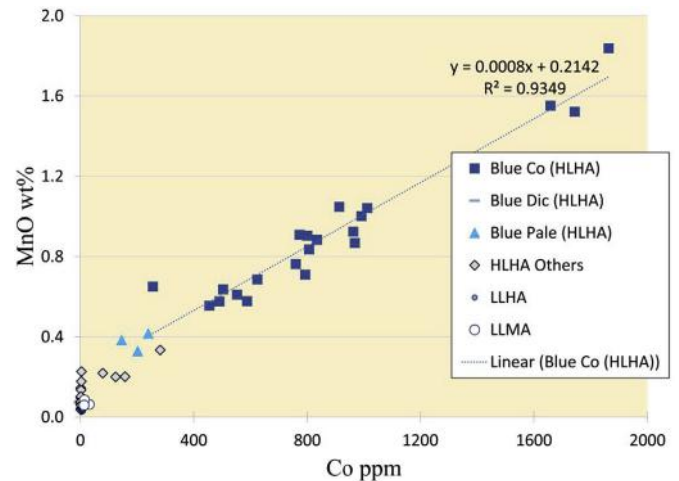

Fig. 11. Manganese oxide vs cobalt content in Igbo Olokun glass beads. The positive correlation of the two constituents in the blue beads indicates that cobalt was added as part of a manganese-rich mineral. (For interpretation of the references to color in this figure legend, the reader is referred to the Web version of this article.)
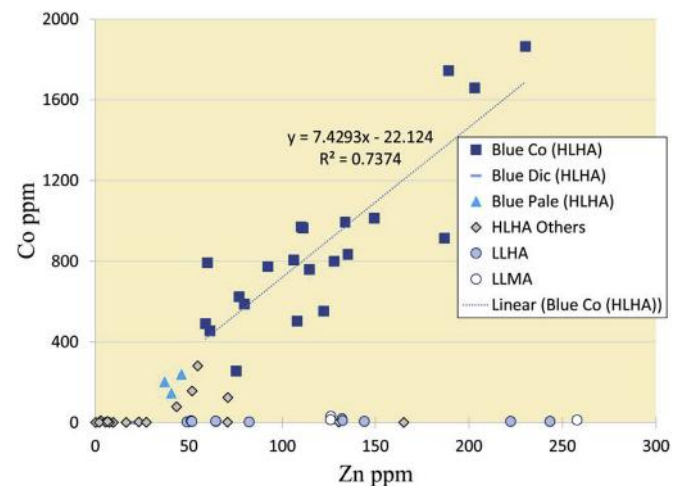

Fig. 12. Cobalt vs zinc content in Igbo Olokun glass beads. The moderate positive correlation suggests that the zinc is entering the glass together with the cobalt source.

samples. Concentrations of iron and manganese oxides in these HLHA glass samples vary, and can be similar in glasses of different colors (Fig. 14). Changes in the glass melting atmosphere might have played a major role in producing the different colors, producing different iron and manganese oxidation states.

In the LLHA glass group, the range of colors is very different with a majority of opaque glass: red, yellow and white. Some samples are not totally opaque, but very dark. When a light is shone through those objects they appear mostly brown. In the LLHA glass, manganese is low (<0.1%) and therefore might not have been added intentionally to modify the color of the glass.

Copper oxide is 0.2 wt% or more in all the LLHA samples with one exception (Fig. 15). Indeed, IF038B only contains 0.05 wt% of copper oxide. It seems that increased amounts of copper in the red glass are accompanied with increased amounts of iron. The presence of iron in copper-rich glass can facilitate the red color by acting as an internal reducing agent for copper (Brill and Cahill, 1988). The presence of copper-rich particles in the red glass adhering to a crucible fragment from Igbo Olokun indicates that metallic copper is the colorant used to make red color (Fig. 16). The shape of the
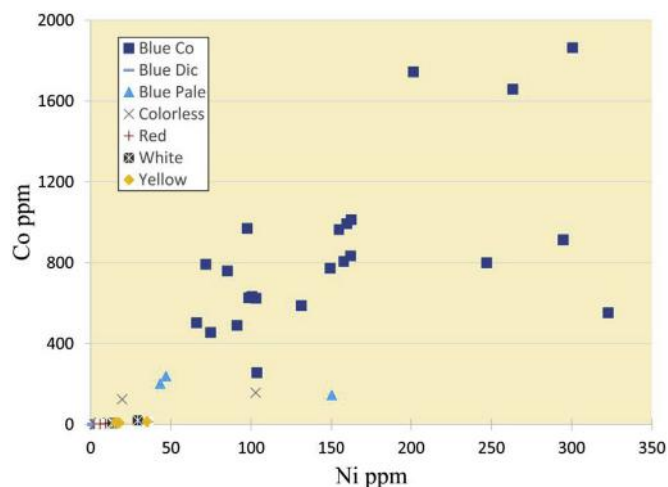
Fig. 13. Cobalt vs nickel content in Igbo Olokun glass beads by color groups. Like zinc, nickel appears to enter the glass together with the cobalt source. (For interpretation of the references to color in this figure legend, the reader is referred to the Web version of this article.)
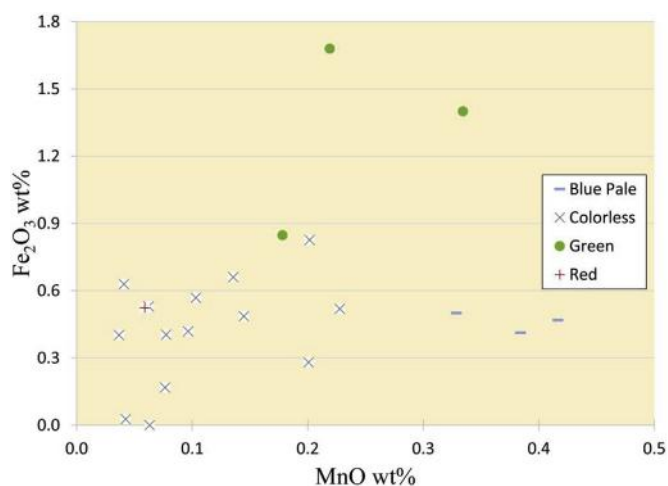

Fig. 15. Scatter plot representing Fe_2O_3 and CuO concentrations in the LLHA dark, opaque red, white and yellow glass samples. (For interpretation of the references to color in this figure legend, the reader is referred to the Web version of this article.)

Fig. 14. Scatter plot presenting the MnO and Fe_2O_3 concentrations in the HLHA translucent or transparent pale blue, green, red and colorless glass samples. (For interpretation of the references to color in this figure legend, the reader is referred to the Web version of this article.)

particles (round drops rather than cubic dendrites) and their reflective behavior (metallic, not transparent) clearly indicate that these are prills of metallic copper, and not cuprite.

In order to determine the opacifying agent, further investigations were conducted on a few beads that appear opaque white. The white color was initially thought to be corrosion, but SEM-EDS analysis of three of these beads (Fig. 8) showed them to be partially weathered HLHA glass that was originally opaque white. Interestingly, their preserved cores showed no noticeable compositional difference compared to the less corroded beads, indicating that the corrosion is due to the particularly moist nature of the context they were found in (Babalola, 2015), and not due to a chemically different, less stable composition. No colorant or opacifier was immediately identified; detailed investigation using high-contrast SEM imaging revealed that the matrix of these beads consists of a dense network of plate-like calcium silicate crystals in a glassy matrix, visible particularly in the heavily corroded bead and at higher magnification (Fig. 17 a & b). Incompletely dissolved
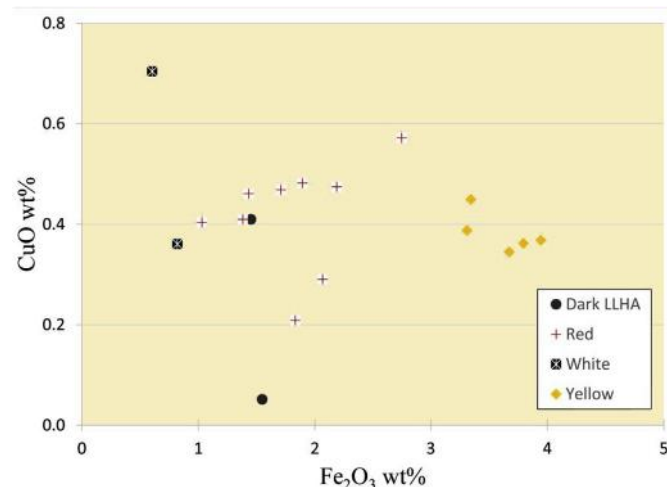
quartz grains, probably relics of the raw materials, and recrystallized dendrites of leucite ($KAlSi_2O_6$) are present (Fig. 17c), but are too rare to be the main source of opacity.

From the analyses, CoO, Fe_2O_3, and CuO seem to have been the major coloring agents used at the Igbo Olokun glass factory. This leaves us with some questions. For example, was cobalt added deliberately as colorant? Was the colorless glass achieved by accident due to less impurity in the raw materials or was it intentionally decolorized? And what ingredients were used as opacifiers? Future investigations targeting specifically the coloring technique for the Igbo Olokun glass beads might give new insights about this very particular glass technology.

3.4. Raw materials

Here, we discuss only the raw materials for the HLHA and LLHA glasses, omitting the small and uncertainly defined LLMA group. Based on archaeological finds, Ogundiran and Ige (2015) suggest pegmatite and snail shells as raw materials for the manufacture of HLHA glass. The snail shell compositions published in their paper are pure calcium carbonate, while the sample of pegmatite they found at Osun Grove contains less than 1 wt% of calcium oxide. Pegmatite is an igneous rock made of large crystals that have a dimension of more than 1 cm and that are very close in composition to granite. Both rocks include quartz, mica and feldspar as main minerals. With an assumption that the HLHA glass is a binary mix of pegmatite and snail shell we calculated from the average composition of the HLHA glass from Ile-Ife what would be the average composition of the silica rich material used as a glass former by removing lime and strontium oxide from the analyzed compositions and re-calculating the remaining components to 100 wt%. Results are presented in Table 3. We compare this composition to the compositional range of pegmatite found approximately 50 km away from Ile-Ife (Akintola et al. 2011). We notice the same high alumina concentrations with extremely low magnesia and phosphorus oxide found in the recalculated composition of the HLHA glass. Similarly, also soda, potash, iron and most of the trace elements present overlapping concentration ranges, supporting the argument first made by Freestone (2006) and later elaborated upon by Ogundiran and Ige (2015).

The LLHA glass contains less lime but some elements were detected with higher concentrations than in the HLHA glass. One of
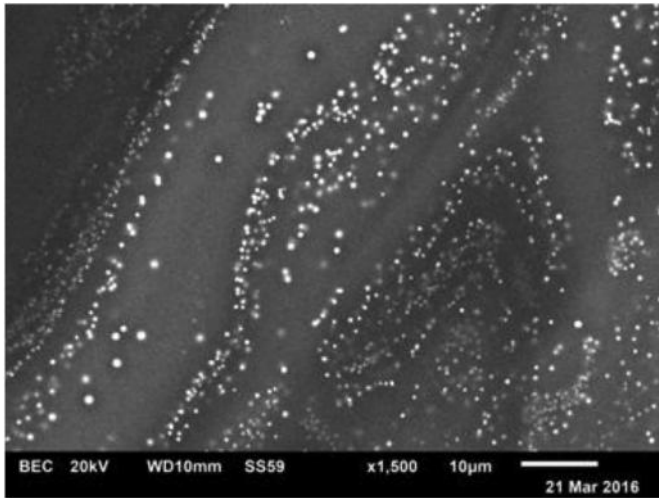
Fig. 16. Backscattered electron (BSE) image of copper particles in the inner red glass of a crucible from Igbo Olokun. The lighter area in form of swirls is indicative of incomplete mixing of colorant, in this case, metallic copper.

these elements is copper. It is possible that the same silica rich material (pegmatite) was used to manufacture this glass but instead of adding snail shells to it, a different ingredient containing copper and other elements such as magnesia, phosphorous and manganese could have been used. This ingredient would have contained some lime too as indicated in the figure below (Fig. 18) where from a general point of view (with one exception) higher concentrations of copper are correlated to higher concentrations of lime. The different ways lime and strontium correlate in the HLHA and LLHA glasses also suggest a different source of lime in each glass (Fig. 19).

4. Discussion

4.1. Ile-Ife glass groups compared to other known compositional groups

The HLHA glass beads from Igbo Olokun are consistent in their composition with those from elsewhere within Ile-Ife (Fig. 20). Lankton et al. (2006) have extensively discussed the uniqueness of the Ile-Ife HLHA glass among other compositional groups from
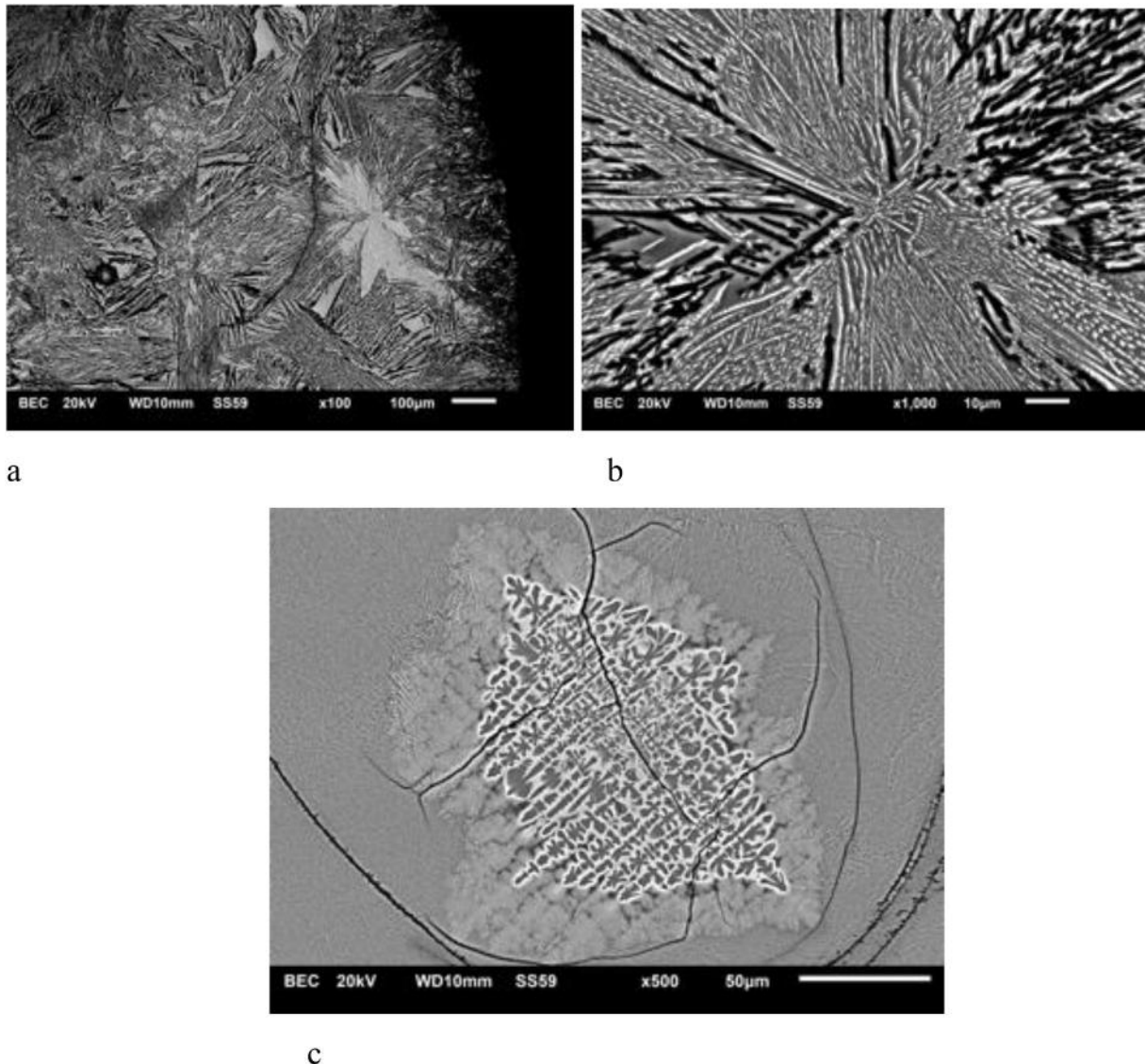

Fig. 17. BSE images of the corroded white beads from Igbo Olokun showing the network of plate-like calcium silicate crystals (a & b), and an incompletely dissolved and recrystallized dendritic structure (c).

Table 3

Comparison of the major, minor, and trace elements of the Ile-Ife HLHA glass with the pegmatite source near Ile-Ife. The composition of the glass is recalculated to a lime- and strontium-free composition in order to facilitate comparison with the pegmatite. The major and minor elements ($SiO_2 - Fe_2O_3$) are reported in wt% while the trace elements (Be $-$ Th) are in ppm.

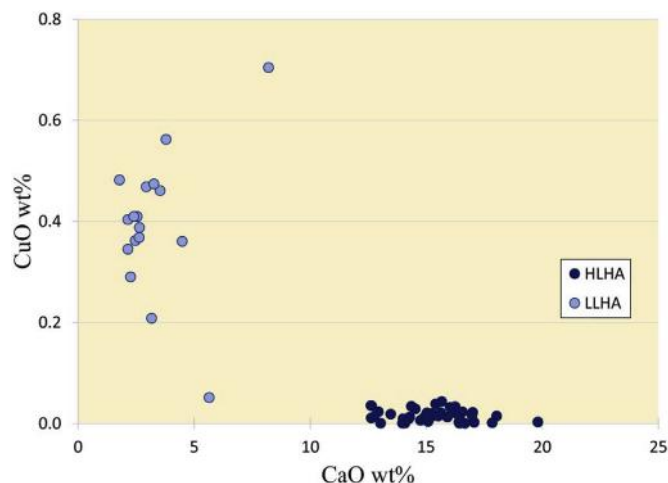|	HLHA glass composition re-calculated	Awo Pegmatite
SiO_2	68.5–79.3	44.8–70.4
Na_2O	2.0–7.7	0.09–4.8
MgO	0.03–0.26	0.01–0.14
Al_2O_3	13.4–20.1	14.7–33.5
P_2O_5	0.09–0.60	0.016–0.199
K_2O	0.88–9.9	1.47–6.95
CaO	–	0.03–1.74
MnO	0.04–3.00	0.008–8.41
Fe_2O_3	0.00–1.98	0.25–7.22
Be	1–149	2.0–82
Ti	83–720	0.1–22.6
Zn	0.2–230	30–210
Rb	39–636	58–1000
Sr	–	22–278
Zr	10.0–48	4–152
Nb	1.0–77	4–390
Cs	1.6–35	2.5–526
Ba	82–604	29–442
Y	2.0–40	1.0–10
W	0.3–2.3	0.5–8
Hf	0.3–6	0.2–8.4
Th	0.3–4.7	0.2–12.9

Fig. 19. Scatter plot showing the concentrations of lime and strontium in the LLHA and HLHA glasses.

Fig. 18. Scatter plot showing the concentration of lime and copper oxide in the LLHA and HLHA glasses.

Fig. 20. Alumina vs lime concentration of the glass beads from Igbo Olokun and other beads from elsewhere in Ile-Ife, showing the prevalence of HLHA (top ellipse) and the cluster of the LLHA at Igbo Olokun. The left bottom ellipse indicates LLMA beads.

such as very low levels (~1 ppm) of uranium, which distinguish Ile-Ife LLHA glass from Indian high alumina glass (U > 11 ppm in average). We conclude that the LLHA glass was also locally made.

4.2. Raw material and production

Earlier work identified the HLHA glass from Ile-Ife as an indigenous African product (Lankton et al., 2006), probably based on pegmatitic sands sourced locally (Ige, 2010; Ogundiran and Ige, 2015). Our comparison with pegmatite available close to Ile-Ife confirms the compatibility of the HLHA composition with the use of pegmatite as a silica source. This raw material would have provided the high alumina content in the glass and explain the very low magnesia and phosphate levels.

An additional lime source, such as limestone or shell, is needed to explain the high lime concentration in the HLHA glass (Freestone, 2006). Snails are common in the rainforest zone where Ife is located, and are a popular staple among the Yoruba, which would have made the shell readily available, even in the large quantities that would have been required to achieve such high lime levels. The use of snail shells in the glass-making recipe is therefore

around the globe. In addition to their discussion, we would reiterate that the alumina content in excess of 10 wt% sets the Ile-Ife beads apart from European and Middle Eastern glasses, which usually have alumina levels below c 3.5–4.0 wt% (Brill, 1987; Wedepohl et al., 2011). The corresponding high lime content differentiates Ile-Ife HLHA glass from South Asian high alumina glass (Dussubieux et al., 2010). In the HLHA glass, soda and potash levels occur at broadly similar concentration ranges, classifying it as mixed-alkali glass. MgO and P_2O_5 are exceptionally low averaging 0.06 wt% and 0.15 wt% respectively (Supplementary Table S1), which indicates that the glass was not made using plant ash as an alkali flux. The low level of MgO and P_2O_5 also separates the group from the Medieval European wood ash glass (Wedepohl et al., 2011).

The LLHA group is much less common among the samples (Fig. 20) and seems to be closely related to HLHA glass. Both have consistently high Al_2O_3 level and similar levels of trace elements,
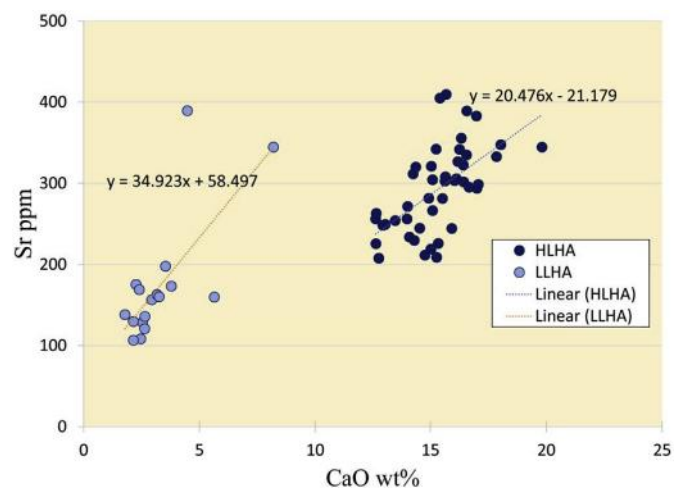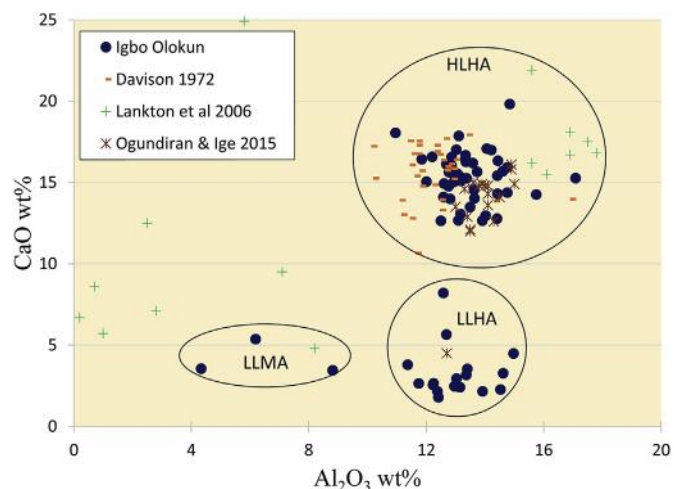
not a farfetched idea. The LLHA glass did not involve the addition of lime and is consistent with a pure granitic/pegmatite raw material with perhaps the addition of a copper rich ingredient containing also lime, magnesia and small quantities of phosphorus.

Among the 52 glass beads analyzed for this study and several other materials analyzed from Igbo Olokun (Babalola, 2015), the three compositional groups presented here are the only ones identified, in contrast to the samples analyzed by Davison (1972) and Lankton et al. (2006) (Table 1). Although the samples chemically analyzed so far are only a very small fraction (about 0.5%) of the assemblage excavated at Igbo Olokun, the careful selection of the samples representing the entire assemblage in terms of shape, decoration, color, and diaphaneity suggest that these results are likely to be reliable. We therefore argue that Igbo Olokun was primarily a production site specialized in making glass beads from HLHA and LLHA glass.

The scale of glass production at Igbo Olokun is unique in West Africa (Babalola et al. in press). Within the Yoruba region, only one other site, Osogbo, has evidence for the production of HLHA glass. It dates to the 17th-18th century. The strong resemblance in the composition of the glass beads from early Osogbo and Ile-Ife glass beads suggests a continuing glass-making tradition (Ogundiran and Ige, 2015), which perhaps originated in Ile-Ife and shifted to Osogbo in later centuries.

4.3. Regional and interregional distribution of Ife type glass beads

Most of the analyzed glass beads from pre-Atlantic era archaeological sites in West Africa have soda-lime compositions that match production areas in the Middle East, Egypt, or Syria-Palestine. These identifications provide valuable clues to the networks that linked disparate sites in West Africa via exchange or organized trade over long distances during the first and early second millennia AD. Soda-lime beads may have been reaching Ile-Ife as early as the 8th-9th century at Orun Oba Ado, but those radiocarbon dates are problematic, as mentioned earlier. The presence of soda-lime beads at Orun Oba Ado and Ita Yemoo indicates that Ife participated in far-reaching networks (Davison, 1972; Willett, 1977). At these sites, soda-lime glass co-occurs with locally produced HLHA glass. Although the chronology of the beginnings and development of HLHA glass production is presently uncertain, available compositional and archaeological data strongly indicate that at least by the 11th century AD HLHA glass beads were being made and used in Ile-Ife. At this time, if not earlier, HLHA beads circulated widely in West Africa. They have been identified in archaeological deposits at Igbo-Ukwu (9th-12th century context – Lankton et al., 2006), Essouk (12th-14th century context – Lankton, 2008), Gao Ancien (10th-12th century (Cissé et al., 2013; Dussubieux, pers. comm.), Kissi (uncertain date – Wood, 2016), Bura (uncertain date – Magnavita, 2016), Diouboye (11th-14th centuries – Gokee, 2016; Dussubieux, pers. comm.) and Kumbi Saleh (uncertain date – Davison, 1972: 261). In all cases, the HLHA beads are part of assemblages that include beads from at least one other glass production area. The distribution of HLHA glass within the Yoruba-Edo area of southwest Nigeria is poorly known, since few excavated glass beads have been chemically analyzed. However, historical sources point to the supply of glass beads from Ile-Ife to other communities within the region (Egharevba, 1968; Ogundiran, 2002). The early circulation of glass beads more widely in southern Nigeria reached impressive levels, as attested by the recovery of more than almost 150,000 beads at Igbo Ukwu (Shaw, 1970), representing multiple composition groups (Brill, 1999; Wood, 2016).

5. Conclusion

No analyses of glass beads from Ile-Ife had been published since Davison (1972), until Lankton et al. (2006) recognized Ife HLHA glass as unique among compositional groups known in the Old World. This study provides for the first time compositional data for beads from controlled excavations in Ile-Ife, including trace elements. These new data significantly strengthen the argument by Lankton et al. (2006) and Freestone (2006) of the importance and uniqueness of HLHA glass in Ile-Ife, and more generally in southern Nigeria, and enable us to define a related glass group with lower lime levels, dubbed LLHA. We are now confident beyond reasonable doubt that both the HLHA and LLHA groups of glass represent a glass produced in early Ile-Ife using local recipes, raw materials and technology.

Compositional analysis by LA-ICP-MS has proved highly useful for better understanding the chemical characteristics of the Ile-Ife HLHA and the LLHA glass beads. They are distinctive from the soda-lime glass of the Islamic world with the wood ash or potash glass from medieval Europe, or the Indian high alumina glass. Based on the presence of glass-melting crucibles and working waste at Igbo Olokun, it is certain that glass was colored and worked into beads at the site. The prevalence of HLHA and LLHA glass at Igbo Olokun and the availability of suitable raw materials nearby suggest a possible proximity of the workshop to a primary source. HLHA glass beads have been found at several sites across West Africa, indicating wider significance of this glass production and the involvement of Ile-Ife in early trade and exchange networks. Finally, this paper has opened a new pathway in our knowledge of early technology in global perspective. David Killick (2016: 62) recently argued that rather than focusing on iron as a single "pyrotechnology in sub-Saharan Africa, much more attention needs to be paid to other branches of pyrotechnology." This paper, and its companion paper on the crucibles used in this glass industry (Babalola et al. in press), both aim to contribute to such a wider view of early African pyrotechnologies.

Appendix A. Supplementary data

Supplementary data related to this article can be found at https://doi.org/10.1016/j.jas.2017.12.005.

References

Abe, Y., Harimoto, R., Kikugawa, T., Yazawa, K., Nishisaka, A., Kawai, N., Yoshimura, S., Nakai, I., 2012. Transition in the use of cobalt-blue colorant in the New Kingdom of Egypt. J. Archaeol. Sci. 39, 1793—1808.

Adediran, B., 1992. The Early Beginnings of the Ife State. The Cradle of a Race: Ife from the Beginning to 1980. Port Harcourt. Sunray Publications, pp. 77—95.

Adeduntan, J., 1985. Early glass bead technology of Ile-Ife. West Afr. J. Archaeol. 15, 165—171.

Ajayi, T.R., Suh, C.E., 1999. Partially extractable metals in the amphibolites of Ife-Ilesa Area: a lithogeochemical approach for gold exploration. J. Min. Geol. 35, 103—116.

Akintola, A.I., Omosanya, K.O., Ajibade, O.M., Okunlola, O.A., Kehinde-Philips, O.O., 2011. Petrographic and geochemical evaluations of rare — metal (Ta-Nb) potentials of precambrian pegmatites of AWO area, southwestern Nigeria. Int. J. Basic Appl. Sci. 11 (4), 57—70.

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

Babalola, A.B., McIntosh, S.K., Dussubieux, L., Rehren, Th, 2017. Ile-Ife and Igbo Olokun in the history of glass in West Africa. Antiquity 91 (357), 732—750.

Babalola, A. B., Rehren, Th, Ige, A. and McIntosh, S.K., in press. The glass crucibles from Ile-Ife, Nigeria. *J. Afr. Archaeol.*

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

Brill, R.H., 1987. Chemical analyses of some early Indian glasses. In: Archaeometry of Glass, XIV International Congress on Glass, pp. 1—25.

Brill, R.H., 1999. Chemical Analyses of Early Glasses. Vol. 1: Catalogue of Samples. Vol. 2: Tables. Corning Museum of Glass.

Brill, R.H., Cahill, N.D., 1988. A red opaques glass from Sardis and some thoughts on red opaques in general. J. Glass Stud. 30, 16—27.

Burlet, C., Vanbrabant, Y., 2015. Study of the spectro-chemical signatures of cobalt-manganese layered oxides (asbolan-lithiophorite and their intermediates) by Raman spectroscopy. J. Raman Spectrosc. 46, 941—952.

Cissé, M., McIntosh, S.K., Dussubieux, L., Fenn, T., Gallagher, D., 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. J. Afr. Archaeol. 11, 9—37.

Davison, C.C., 1972. Glass Beads in African Archaeology: Results of Neutron Activation Analysis, Supplemented by Results of X-Ray Fluorescence Analysis. Ph.D. University of California, Berkeley, United States.

Davison, C.C., Giauque, R.D., Clark, J.D., 1971. Two chemical groups of dichroic glass beads from West Africa. Man, New Series 6, 645—659.

Dussubieux, L., Kusimba, C.M., Gogte, V., Kusimba, S.B., Gratuze, B., Oka, R., 2008. The trading of ancient glass beads: new analytical data from South Asian and East African soda—alumina glass beads. Archaeometry 50, 797—821.

Dussubieux, L., Robertshaw, P., Glascock, M.D., 2009. LA-ICP-MS analysis of African glass beads: Laboratory inter-comparison with an emphasis on the impact of corrosion on data interpretation. Int. J. Mass Spectrom. 284, 152—161.

Dussubieux, L., Gratuze, B., Blet-Lemarquand, M., 2010. Mineral soda alumina glass: occurrence and meaning. J. Archaeol. Sci. 37, 1646—1655.

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

Eluyemi, O., 1987. The technology of the Ife glass beads: evidence from Igbo-Olokun. ODU 32, 200—216.

Eyo, E., 1974. Excavations at Odo Ogbe street and Lafogido, Ife, Nigeria. W. Afr. J. Archaeol. 4, 99—109.

Francis, P., 1991. Beadmaking at Arikamedu and beyond. World Archaeol. 23, 28—43.

Freestone, I.C., 2006. An indigenous technology? A commentary on Lankton et al. "Early primary glass production in southern Nigeria". J. Afr. Archaeol. 4, 139—141.

Freestone, I.C., Meeks, N., Sax, M., Higgitt, C., 2007. The Lycurgus cup — a Roman nanotechnology. Gold Bull. 40 (4), 270—277.

Frobenius, L., 1913. [1968]. Voice of Africa; being an account of the travels of the German Inner-African Exploration Expedition in the years 1910-1912. In: Deutsche Inner-afrikanische Forschungs Expedition. Blom.

Garlake, P., 1974. Excavations at Obalara's land, Ife: an interim report. W. Afr. J. Archaeol 4, 111—148.

Garlake, P., 1977. Excavation at the Woye Asiri family land in Ife, Western Nigeria. W. Afr. J. Archaeol 7, 57—96.

Gokee, C.D., 2016. Assembling the Village in Medieval Bambuk: an Archaeology of Interaction at Diouboye. Equinox Publishing Limited, Senegal.

Ige, O.A., 2010. The Science behind the Stories: the Adventure of a Museum Geologist. Inaugural Lecture Delivered at the Obafemi Awolowo University Ile-Ife, Nigeria.

Kaczmarczyk, A., 1986. The source of cobalt in ancient Egyptian pigments. In: Proceedings of the 24th International Archaeometry Symposium. Smithsonian Institution Press, pp. 369—376.

Killick, D., 2016. A global perspective on the pyrotechnologies of Sub-Saharan Africa. Azania 51, 62—87.

Kingery, W.D., Vandiver, P.B., Huang, I.-W., Chiang, Y.-M., 1983. Liquid-liquid immiscibility and phase separation in the quaternary systems $K_2O-Al_2O_3$-CaO-SiO_2 and $Na_2O-Al_2O_3$-CaO-SiO_2. J. Non-Cryst. Solids 54, 163—171.

Lankton, J.W., 2008. The glass from Tadmakka: chemical compositional analysis and interpretation. In: Nixon, S. (Ed.), The Archaeology of Early Islamic Trans-saharan Trading Towns in West Africa: a Comparative View and Progressive Methodology from the Entrepot of Essouk-tadmekka. University of London, pp. 443—455. PhD thesis.

Lankton, J.W., Dussubieux, L., 2006. Early glass in Asian maritime trade: a review and an interpretation of compositional analyses. J. Glass Stud. 48, 121—144.

Lankton, J.W., Ige, O.A., Rehren, Th, 2006. Early primary glass production in Southern Nigeria. J. Afr. Archaeol. 4, 111—138.

Li, Weidong, Li, Jiazhi, Wu, Jun, Guo, Jingkun, 2003. Study on the phase-separated opaque glaze in ancient China from Qionglai kiln. Ceram. Int. 29, 933—937.

Magnavita, S., 2016. Early trans-Saharan trade in the light of archaeometric research. In: Paper Presented at the Society of Africanist Archaeologists Meetings, Toulouse, France.

McIntosh, S.K., 1995. Excavations at Jenné-Jeno, Hambarketolo, and Kaniana (Inland Niger Delta, Mali), the 1981 Season. University of California Press.

Ogundiran, A., 2002. Of small things remembered: beads, cowries, and cultural translations of the Atlantic experience in Yorubaland. Int. J. Afr. Hist. Stud. 35, 427—457.

Ogundiran, A., 2005. Four millennia of cultural history in Nigeria (ca. 2000 BC—AD 1900): archaeological perspectives. J. World Prehistory 19, 133—168.

Ogundiran, A., Ige, O.A., 2015. "Our ancestors were material scientists" archaeological and geochemical evidence for indigenous Yoruba glass technology. J. Black Stud. 46, 751—772.

Ozainne, S., 2013. Un Néolithique Ouest-Africain: cadre chrono-culturel, économique et environnemental de l'Holocène récent en Pays dogon (Mali), vol. 8. Africa Magna Verlag.

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: Crossroads: Cultural and technological developments in 1st millennium BC/AD West Africa. Journal of African Archaeology Monograph Series 2, 105—118.

Robertshaw, P., Wood, M., Melchiorre, E., Popelka-Filcoff, R.S., Glascock, M.D., 2010. Southern African glass beads: chemistry, glass sources and patterns of trade. J. Archaeol. Sci. 37, 1898—1912.

Shaw, C.T., 1970. Igbo Ukwu. Faber, London.

Smirniou, M., Rehren, Th, 2013. Shades of blue — cobalt-copper coloured blue glass from New Kingdom Egypt and the Mycenaean world: a matter of production or colourant source? J. Archaeol. Sci. 40, 4731—4743.

Wedepohl, K.H., Simon, K., Kronz, A., 2011. Data on 61 chemical elements for the characterization of three major glass compositions in Late Antiquity and the Middle Ages. Archaeometry 53, 81—102.

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

Willett, F., 1959. Bronze figures from Ita Yemoo, Ife, Nigeria. Man 49 (308), 189—193.

Willett, F., 1960. Ife and its archaeology. J. Afr. Hist. 1, 231—248.

Willett, F., 1971. A survey of recent results in the radiocarbon chronology of Western and Northern Africa. J. Afr. Hist. 12, 339—370.

Willett, F., 1977. Baubles, Bangles, and Beads: Trade Contracts of Medieval Ife. University of Edinburgh for the Centre of African Studies, Edinburgh.

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

Willett, F., Fleming, S.J., 1976. A catalogue of important Nigerian copper-alloy castings dated by thermoluminescence. Archaeometry 18, 135—146.

Wood, M., 2011. Interconnections: Glass Beads and Trade in Southern and Eastern Africa and the Indian Ocean - 7th to 16th Centuries AD. Uppsala University, Uppsala.

Wood, M., 2016. Glass beads from pre-European contact sub-Saharan Africa: Peter Francis's work revisited and updated. Archaeological Research in Asia 6, 65—80.