Composition data of a large collection of black-appearing Roman glass

Simone Cagno,[1] Peter Cosyns,[2]
Veerle Van der Linden,[1] Olivier Schalm,[3]
Andrei Izmer,[4] Isolde Deconinck,[4]
Frank Vanhaecke,[4] Anna Nowak,[5]
Barbara Wagner,[5] Ewa Bulska,[5]
Karin Nys,[2] Koen Janssens[1]

[1]Department of Chemistry, University of Antwerp, Antwerp; [2]Mediterranean Archaeological Research Institute, Vrije Universiteit Brussel, Brussels; [3]Faculty of Design Sciences, University of Antwerp, Antwerp; [4]Department of Analytical Chemistry, University of Ghent, Ghent, Belgium; [5]Faculty of Chemistry, University of Warsaw, Warsaw, Poland

Abstract

Jewels and tableware made of black-appearing glass were popular in the Roman Empire. Compositional changes (due to modifications in glassmaking technology and use of raw materials) over the period considered (1st-5th century AD) have been investigated on a large number of samples originating from various archaeological excavations in Europe, Northern Africa and the Near East. In the course of this work, over 400 samples of Roman glass, the greatest part of them deeply coloured glass fragments, were embedded into acrylic resin and mechanically ground and polished in order to obtain flat surfaces of unaltered glass. The samples were analysed with scanning electron microscopy/energy dispersive X-ray spectroscopy and the quantification was performed by using a standard-less ZAF software. The trace elements contained in a selection of glass samples were determined via laser ablation-inductively coupled plasma-mass spectrometry. The data collected in this study show that from about 150 AD a change in the black glass production process occurred, involving coloration of raw glass made with iron in the secondary workshops. Furthermore, from the 4th century AD on we can observe a change in the type of raw glass used, while the colouring process was maintained. The main aim of this paper is to provide glass scholars with the analysis results, as reference and comparison for further studies.

Introduction

The introduction of glassblowing halfway through the 1st century BC turned glass from an expensive luxury material into an affordable commodity: the glass market expanded and large amounts of glass were produced in order to satisfy the needs of the consumers in Rome and its provinces. In this context it is interesting to explore the dynamics and locations of glassmaking and glassworking.

Between the Hellenistic and the Byzantine period the technological level and conditions required to control glassmaking were significantly more elevated than those needed for glassworking (Stern and Schlick-Nolte, 1994; Stern, 1999). In order to manufacture glass objects, glassworkers in secondary glass workshops melted and shaped raw glass obtained from primary workshops. It is generally accepted that during the Roman imperial period glass objects were manufactured in secondary glass workshops all over the Empire by re-melting and shaping raw glass imported from primary workshops which are located in the south-eastern Mediterranean with the Egyptian and the Syro-Palestinian coast as key areas (Foy, 2000; Nenna, 2008; Jackson et al., 2009). These primary workshops were located close to the raw material supplies, using lime-rich coastal sand and natron to produce large slabs of glass. From the primary centers the raw glass was then shipped (in large chunks) along the trade routes to the secondary workshops throughout the Roman Empire (Nenna, 2008; Henderson, 1985; Velde, 1990; Freestone et al., 2000; Freestone, 2006; Picon and Vichy, 2003; Fontaine and Foy, 2007). However, it has also been suggested that in the Roman period primary glassmaking might have occurred in the Italian peninsula as well as in Spain and Gaul likewise Pliny the Elder's statement in his Naturalis Historia (Silvestri et al., 2006; Degryse and Schneider, 2008; Foster and Jackson, 2009).

Previously we proposed that, in a first phase, the craftsmen's knowledge in the western provinces was limited to the production of objects by re-melting imported *naturally* colored, deeply colored and decolorised raw glass and cullet. In a later phase at about the mid 2nd century AD the expertise got enriched with the acquaintance in how to obtain independently the color of the (remelted) glass batch (Van der Linden et al., 2009). A logical deduction is that a final stage in the accumulation of glass craftsmen's knowledge in the western part of the Roman Empire comprises the know-how to produce raw glass locally by means of imported natron may have been reached in some regions during the late Roman period. This evolution in knowledge in glass production technology is one of two possible models for

glass production in the late Roman period (Shortland et al., 2006; Wedepohl and Baumann, 2000). A second model suggests the continuation of large primary glass centers in the eastern Mediterranean (Foy et al., 2000; Freestone et al., 2005).

In a first period, from Tiberian to Flavian times, so-called black glass was one of the hues used to produce the fashionable deeply colored monochrome glass vessels which received an empire wide distribution (Cosyns and Fontaine, 2006). The subsequent production period of black appearing glass vessels lasted from the mid 2nd to the mid 3rd century AD and had only a regional impact on the consumption as it was limited to the north-western provinces Gallia Belgica, Germania inferior and Germania superior (Cosyns and Hanut, 2005; Cosyns et al., 2006). Influenced by the success of black appearing jewelry in jet and other materials there is a huge increase from the 3rd century AD onwards in using black glass to produce jewelry – bangles, finger-rings, beads, pendants, hairpins, and gems. Affected

by the invasions of the Migration People this production gradually disappeared during the 5th century AD in the western part with solely black glass beads in combination with trailed decoration in vivid colored opaque glass remaining in use up to the mid 6th century AD. Conversely, a continuity of a wide range of jewelry production in black glass can be observed within the Byzantine Empire (Cosyns, 2011).

Because of its unusual time- and location-specific distribution, black glass is a useful type of archaeological find to be studied in order to explore in a more detailed way the relation between primary and secondary glass workshops and the trade among them.

The aim of this paper is to provide scholars with a substantial number of analysis results of Roman black glass fragments, as a reference and comparison for further studies. Data relative to the over 400 analysed samples are presented here. A basic interpretation of the data is provided in the present paper, whereas the statistical reduction of the data and a wide descriptive interpretation is provided elsewhere (Van der Linden et al., 2009). Not many analyses of black glass have been published up to now. The earliest analytical results of European black glass are those of a set of European glass beads originating from France and Switzerland (Gratuze, 2009; Cagno et al. 2014). The Bronze Age samples in this series are ash-fluxed, while the Iron Age ones are natron-fluxed, (low levels of MgO and K_2O) and containing low amounts of CaO. In both cases, the black color is obtained by a notably high amount of Fe_2O_3, comprised between 10 and 25%.

Among the published analytical results of Bronze and Iron Age glass material from Pella, Jordan (Reade et al., 2009), the black glass, natron fluxed, is rather different from the glass produced in that same period. The composition is similar to the aforementioned Iron Age glass and the color of the glass is determined by the high percentage of iron present.

Besides that, Pre-Roman dark glass beads from Belgium, dated to the 2nd quarter of the 3rd century BC (Cosyns and Hurt, 2007), analysed via laser ablation-inductively coupled plasma-mass spectrometry (LA-ICP-MS) show two different compositions. The first one (black/green glass) can be confidently attributed to the Syro-Levantine glass type, with a significant voluntary addition of iron in order to render the glass black, while the second (brown glass) has a more difficult interpretation. Compositions of more samples of dark glass from the Iron Age to the late antiquity are also reported; these are all of the Syro-Levantine high-iron type (Cosyns and Gratuze, 2007).

In the Low Countries, a precursor of the black-appearing Roman glass is the La Tène dark purple glass. Large quantities of purple glass jewelry dated to the late La Tène period (2nd-1st century BC) have been found in the central area of the Netherlands. The composition of only a few fragments of such glass is published (together with others results) by Van der Linden et al. (2009). This glass has a composition similar to naturally-colored natron-based glass, but with a high content of manganese. Presumably a significant amount of a Mn mineral was used in order to obtain the desired dark purple/black hue. For what concerns black-appearing Roman glass, a first compilation of data is presented in Van der Linden et al. (2009). The most abundant and widespread type of coloration is the black/green one. Two different compositions could be distinguished among the black/green sample set: a low iron group (1-2% Fe_2O_3), associated with the Early Roman imperial period up to about 150 AD, and a high iron one, containing 4-10% Fe_2O_3, typical of the period after 150 AD. Those data are also implemented in this paper, for comparison purposes.

Materials and Methods

Over 400 archaeological samples have been selected for analysis including black-appearing glass, deeply colored glass and colorless glass. The two latter color groups only represent a minor set of samples which have been chosen for comparison. The samples cover the whole area of the Roman Empire and the period 1st-5th century AD.

Small glass fragments were removed from the archaeological finds and embedded into acrylic resin. The resin blocks were mechanically ground with silicon carbide paper and polished with diamond paste up to 0.25 μm in order to obtain a smooth surface of unaltered glass. Finally the resin blocks were coated with a thin carbon layer and scanning electron microscope–energy-dispersive X-ray spectroscopy (SEM-EDX) measurements were performed. After the removal of the carbon layer, the same sets of resin blocks were used for LA-ICP-MS analyses.

SEM-EDX measurements were performed in Antwerp, Belgium, with a JEOL 6300 SEM equipped with an energy-dispersive X-ray detector. The spectra were collected for 100 seconds by using a 2 nA electron beam current, an accelerating voltage of 20 kV and a microscope magnification of 500. The net intensities were calculated with the program AXIL and quantified by means of a standardless ZAF programme (Schalm and Janssens, 2003).

The LA-ICP-MS measurements were performed on selected samples in two laboratories in Warsaw, Poland, and Ghent, Belgium.

In Warsaw an ELAN 9000 ICP-MS equipped with the LA system LSX-200+ was used. The LSX-200+ contains a stable, environmentally sealed 266 nm UV Nd:YAG laser with a high sampling efficiency, variable pulse repetition rate and maximum energy up to 6 mJ per pulse. More details on the instrumental conditions can be found in literature (Van der Linden et al., 2009). Line-scan measurements were performed on each sample: a measurement cycle consisted of pre-cleaning the sample surface with a laser beam (no signal was registered during this step) followed by a second ablation pass accompanied by registering of mass signals. Before ablation, blank signals were always registered within the first 18 s of argon flow only. Before quantification the blank signal was subtracted for each element individually. 43Ca was used as an internal standard, while the Corning Museum of Glass (CMG) standard B was employed as an external standard; the quantification was validated by using NIST and CMG glass standards.

In Ghent the PerkinElmer Sciex DRCplus and Thermo Corporation X-Series 2 ICP-MS instruments were used. The measurements were carried out using a GeoLas 193 nm ArF excimer-laser ablation system. A pre-cleaning of the surface was performed with a wider laser-beam than for ablation (120 vs 80 μm diameter). A laser output energy of 100 mJ, generating a fluence of ~6 J.cm^{-2} on the sample surface, was used throughout all of the measurements. The measurements were performed with a repetition rate of 10 Hz along a 1.2 mm long line, with a speed of 0.2 mm/s for pre-cleaning and 0.02 mm/s for ablation. The most important ICP-MS instrument settings (carrier gas flow rate and RF power) were optimised, aiming at maximum signal intensities, minimal background intensity and low oxide ion and doubly charged ion formation, based upon the signal intensities obtained when ablating NIST SRM 610. The signal intensities, obtained during 60 sec of the signal profile obtained during the line scan, were averaged so that for each nuclide of interest, an intensity value proportional to the concentration of the isotope in the sample was obtained. The average of the blank signal, monitored during twenty seconds at the beginning of each LA-ICP-MS measurement, was used for blank correction. NIST SRM 610 was analysed in the same way as the Roman black glass samples to act as external standard. During each measurement, 43Ca was measured as internal standard. The method was validated via the analysis of several NIST standard glasses.

Results

The obtained glass compositions are reported in Supplementary Table 1 (SEM-EDX data) and Supplementary Table 2 (LA-ICP-MS data).

The data are expressed in oxide weight percentages (Supplementary Table 1) and μg/g (Supplementary Table 2).

In the following paragraphs a number of general considerations about the structure of this dataset are provided with the help of oxide bi-plots. For a more detailed description and interpretation of the reported data, we refer the reader to other papers (Van der Linden *et al*., 2009; Cagno *et al*. 2014).

All the glass fragments analysed show a silica-soda-lime composition. As shown in Figure 1, when applying the classification proposed by Henderson (1985), relative to the fluxing agent, the samples mostly fall in the low magnesium glass (LMG) group (K_2O and $MgO<1.5\%$), and partly in the high magnesium glass (HMG) group (K_2O and $MgO>1.5\%$). The LMG-group is totally made of 1st century AD samples.

For what concerns the colouring agents used to obtain a black appearing hue, two main groups are found (Figure 2): i) glass with a dark green hue, colored with iron; ii) glass with a dark purple hue, coloured with manganese.

Next to these, rare samples of glass with a dark blue hue (cobalt and/or copper) and dark brown glass (Fe-S complex) are also present.

The dark green group is by far the largest, represented by many object shapes that were typically only produced in black glass (*e.g.* bracelets, beads). This group covers all the periods and areas studied.

The black/purple glass samples represent a characteristic production mainly related to pre-Roman bracelets dated 1st century BC and 1st century AD and early Roman counters of the 1st-2nd century AD.

Within the dark green group, which represents about 80% of the analysed samples, three distinct compositions can be identified: i) Syro-Levantine glass (LMG), plus a variable percentage of Fe_2O_3 (4-16%); ii) high manganese, iron, titanium (HIMT) glass (LMG), plus a variable percentage of Fe_2O_3 (4-11%); iii) HMG glass, with a low percentage of Fe_2O_3 (1-3%).

Iron is responsible for the green hue; because of the high Fe concentration, this results in a very dark color. For the first two groups, the introduction of iron in the glass batch is unlikely to have taken place in the primary Levantine workshops, devoted to the production and trade of widely marketable raw glass, but was probably done in a secondary step, coincident with the reworking and shaping of the raw glass in Western workshops (Van der Linden *et al*., 2009).

The Syro-Levantine naturally-colored group of glass is the most common in the analysed set (about 50%) and it is spread throughout almost all the considered sites, from the ones closest to the raw glass workshops (*i.e.* in Israel, Cyprus) to the farthest ones. The hypothesised raw materials used for its production are lime-rich sand (*e.g.* from the Belus river, as cited by Pliny) and mineral soda (natron) obtained from inland lakes, (*e.g.* in Wadi al Natrun, Egypt). When considering the dates attributed to the analysed samples, this composition appears to be typical for the glass from mid 2nd century AD to the mid 4th century AD, agreeing with the Syro-Levantine (naturally colored) glass time span proposed in the literature (Foster and Jackson, 2009).

The HIMT glass group is comprised of glass from the 4th century onwards and its composition is similar to the reported analyses of HIMT glass in the literature (Foster and Jackson, 2009; Freestone *et al*., 2005), except for their extremely high iron content. HIMT glass is a late Roman glass composition originating from the Eastern Mediterranean (presumably not from Syro-Palestine) that is richer in Na, Mn, Ti, Fe and is of overall lower quality than the Syro-Levantine glass. The dark green color is generated, as before, by the addition of iron to raw glass in secondary workshops.

The HMG glass is produced by using ash of halophytic plants, (*e.g.* Salsola Soda) as a fluxing agent. This composition is not typical of Roman glass, but of both earlier and later glass. However, it is reported that during the Roman period in areas not under Roman domination, glass was produced in this manner (Mirti *et al*., 2008). A few samples of this group are on a line connecting HMG and LMG samples (Figure 1). Thus, it is possible that a mixing process of vegetable and mineral soda was in use at the time, or that recycled glass (cullet) with an LMG composition was added to a batch containing vegetable ash and sand. The HMG samples contain a lower amount of iron oxide than the other groups; iron is also correlated to other Fe-tracers present in the sand, suggesting that no iron was separately added to the batch. Since in most cases the Fe-content in this group is not high enough to give rise to the dark color alone, we must assume that at this stage the thickness and shape of the objects played an important role in the perception of color. Only one HMG sample, originating from Trier, presents a high content of iron (6.9% Fe_2O_3).

The trace element contents can help in identifying specific silica sources: when considering the Ti and Zr contents (Figure 3) a distinct difference in sand source is evidenced by the Zr/Ti ratio of the HMG-HIMT glass that is different from that of the Syro-Levantine glass. The sand used to produce the HIMT glass appears to be similar to that of the earlier HMG glass, with higher average Ti-content.

Finally, the black/purple glass owes its color to high concentration levels of MnO ranging from 1.6 to 10.5%. Normally, MnO contents of up to 0.6% are commonly found in Roman (non-purple) glass (Aerts *et al*., 2003; Brill, 1999; Silvestri *et al*., 2007) and are attributed to impurities present in the siliceous sand. In the present case, it is clear that a Mn-mineral was intentionally added. Among the Mn-rich samples, a few LMG-HMG intermediate samples are found (Figure 1).

Discussion and Conclusions

The results presented in this paper on the composition of black-appearing glass from over five centuries offer the possibility of comparing a large amount of data and give a wide overview on the used raw materials and adopted production techniques of Roman glassmak-
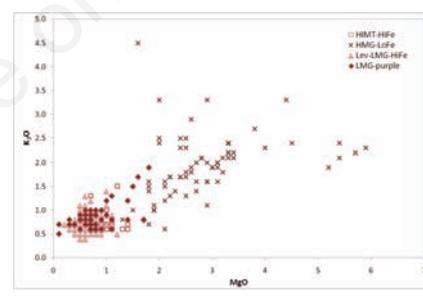

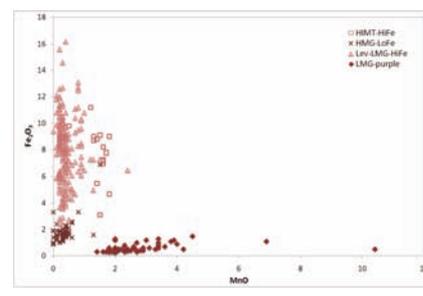**Figure 1. K_2O and MgO concentrations in the analysed samples (% w/w).**

Figure 2. MnO and Fe_2O_3 concentrations in the analysed samples (% w/w).

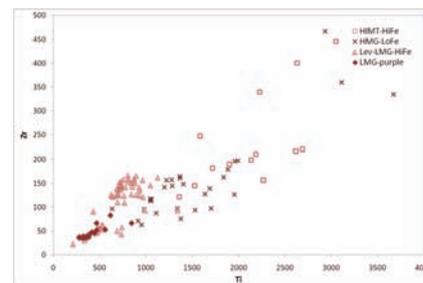**Figure 3. Ti and Zr concentrations in the analysed samples (μg/g).**

ing. Different raw materials (silica source, flux, coloring agents) have been identified on the basis of the presented data. When combining the compositions with the typological and chronological information, we obtain an account of the black Roman glass compositional history. Two co-existing recipes can be distinguished for the first period of popularity of black glass (until the mid 2nd century AD): one is the black/purple glass, made with Syro-Levantine raw glass (produced with relatively pure sand and natron), colored via the addition of manganese, while the other is the dark green glass, with a HMG composition (produced with impure Fe-rich sand and vegetable ash), without deliberate addition of iron. The black purple glass and the HMG glass are rather uniformly distributed but particularly concentrated in the Western provinces.

In the second half of the 2nd century, when black glass became fashionable for a second time, particularly within the North-Western provinces, innovations in glass technology occurred. Although artifacts continued to be made of black/purple glass with the same recipe as before, a big change is noticeable in black/green glass production: the low iron HMG glass disappeared, and got replaced by the Levantine base glass with purely mineral soda and colored with a high amount of iron. This shift in applied glass technology can be related to a higher degree of knowledge acquired in the secondary workshops in the western part of the Roman Empire.

The major change in global glass production that occurred in the Roman Empire during the 4th century is also noticeable in the black glass production. A decline in the use of the more refined Syro-Palestinian raw glass is observed in favor of a less pure, and probably cheaper, HIMT glass. Most 4th century artifacts show the use of HIMT glass and the addition of iron, confirming the hypothesis of the coloring process in secondary workshops, where iron minerals were added to the available raw glass. Considering the variable glass composition and the different hues of the glass that were employed in the five centuries and within the European-Mediterranean area covered by this study, it is possible to conclude that the production of black glass was a process driven by the availability of the starting materials (*e.g.* the type of raw glass, specific Fe/Mn-minerals) with the aim of obtaining a (macroscopically apparent) black appearance.

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