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Quantitative Analysis of Glass Artifacts from Mykolaiv and Zaporizhzhia Oblasts (southern Ukraine): a Multi-Scale Approach

Kateryna D'Ayala,[1] Dmytro Nykonenko[1,2], Anzhelika Kolesnychenko[3,4], Oleh Yatsuk[1], Patrizia Davit[1], Monica Gulmini[1]

[1]Department of Chemistry, University of Turin, Via Pietro Giuria, 7 – 10125 Torino (Italy)
[2]Khortytsia National Reserve, Vulytsya Starogo Redutu 9, – 69017 Zaporizhzhia (Ukraine)
[2]Science and Technology in Archaeology and Culture Research Centre, The Cyprus Institute, Konstantinou Kavafi Street, 20 – 212, Aglantzia, Nicosia (Cyprus)
[4]Institute of Archaeology of the National Academy of Sciences of Ukraine, Volodymyr Ivasiuk avenue, 12 – 04210 Kyiv (Ukraine)

Abstract
This study explores approaches to investigate archaeological glass artifacts and discuss the merits of a multi-scale approach integrating macroscopic characteristics with microanalysis to shed light into the routes of glass exchange in the past populations and on glass production techniques. Two case studies from southern Ukraine are considered: Hellenistic core-formed vessels from Olbia Pontica, (5th c. BCE – 1st centuries CE) and Scythian beads from Khortytsia, (5th-4th centuries BCE). SEM-EDS analysis of the Olbia glass led a preliminary classification into Types II and III, linked to a Syro-Palestinian and Egyptian productions. The Khortytsia beads underwent in-depth analysis using LA-ICP-MS, XRD, and FORS, allowing a more detailed classification based on trace elements, as well as identification of opacifiers, and colorants. The beads were divided into two groups according to TiO_2 and CaO concentrations, aligning with Iron Age production centres of glass in Egypt and the Levant.

I. INTRODUCTION

Glass is a material frequently found in archaeological contexts of the second half of the first millennium BCE, particularly in funerary assemblages, where it appears as personal ornaments and grave goods [1,2]. It was also commonly used for small, core formed containers, which likely held perfumes, ointments, or cosmetic powders [3], as well as for everyday tableware, much like today [4]. Due to its wide distribution and the relatively limited number of known production centres in the Mediterranean region [5,6], ancient glass can provide valuable information about the trade routes and cultural exchanges between past societies [7]. In the Mediterranean area, glass finds have traditionally been classified through a purely archaeological lens—based on typology, chronology, and colour [8,9]. Only later; compositional approaches were also used, which highlighted groups based on chemical analyses allowed for a more precise understanding of the origin and production techniques of the raw glass [5,10,11]. A major advancement in the study of Mediterranean glass has been recently achieved by Lü (2021), who has revised the compositional classification of a large set of glass artefacts through the adoption of a scientifically grounded, quantitatively driven compositional approach, marking a turning point in the integration of archaeometric data into the typological framework [7].

In order to build the multi-scale approach, a variety of methods and techniques can be used both in the laboratory and on-site. Among them. optical microscopy is a non-invasive method that provides preliminary information about the structure of the object and its state of preservation, both of which can already suggest manufacturing techniques or glass types through alteration processes [12]. Such preliminary observations are crucial for guiding the further steps of the analyses.

X-ray fluorescence (XRF) is widely used for elemental analysis, due to the possibility to employ non-invasive procedures with portable instruments. It allows the detection of major, minor and trace elements (although light elements cannot be included in the element list) helping to identify intentional additives or impurities in the raw materials [13]. Scanning Electron Microscopy coupled with Energy Dispersive X-ray Spectrometry (SEM-EDS) offers complementary microtextural insights in addition to quantitative elemental analyses, provided that the instrument is properly calibrated and the analyses is done on well-preserved objects or samples prepared as polished cross-sections [1,14]. Both XRF and SEM-EDS can be used to enhance the accuracy of data obtained through Laser Ablation Induced Coupled Plasma Mass

Spectrometry (LA-ICP-MS) enabling the use of the internal standard approach in the calibration of the instrument [15]. ICP-MS, particularly when coupled with a lLA microsampling, is a microinvasive technique which enable the quantification of a wide set of elements, from major constituent to trace impurities, due to its low limit of detection [16]. LA-ICP-MS has high spatial resolution with less cross-contamination. Virtually, no sample preparation is needed and many analytical spots can be explored directly on the archaeological sample leaving traces that are normally not visible to the naked eye [17].

Further contribution come from qualitative techniques such as X-ray diffraction (XRD) and Fiber Optic Reflectance Spectroscopy (FORS). XRD provides information on the crystalline phases, which can indicate residues of the raw materials, or newly formed minerals related to temperature and processing conditions [18,19] or opacifiers. FORS is used mainly to detect the chromophores in the glass, as the technique is able to reveal the oxidation states of the metallic ions dissolved in the glass matrix [20].

In this paper, we present two significant case-study to demonstrate how the integration of this set of techniques can significantly improve the classification of glass objects beyond the macroscopic observation that is proper of the archaeological approach, thus enabling a better understanding of the archaeological glass artefacts.

II. MATERIAL AND METHODS

A. Materials

Two sets of archaeological objects are considered, both from southern Ukraine. The first set includes fragments of core-formed vessel from the Greek archaeological site of Olbia Pontica, on the shore of the Southern Buh estuary in Mykolaiv Oblast. It's composed of 28 samples dated back to V – I century BC. The second set consists of twenty glass beads/beads fragments and one glass fragment of an unidentified object from the cemetery of Kanfarka and the hillfort of Sovutynske, on the Island of Khorytsia in Zaporizhzhia. Both the archaeological sites date to the late V to the early IV century BCE and are located in the Khortytsia National Reserve. The set of the beads have been divided into typological groups according to their shape (cubic, barrel, round) and the type of decorations (chevrons, stripes, spots, eye beads) as described with details in [21].

B. Methods

The analytical approach in the investigation of archaeological material shall be non-invasive as much as possible, i.e. no material shall be detached from the archaeological object. On the other hand, the possibility to detach small chips from the original archaeological fragments (i.e. through a micro-invasive approach), is nevertheless a valuable opportunity: if the samples are then analysed with non-destructive methods, they remain available for subsequent analyses even in the future, should further analytical exploration be envisaged. In the two set of samples considered here, the two approaches are represented: core-formed vessels have been studied after that small chips have been detached to represent all the glass colours in each object and then embedded in epoxy resin, whereas a non-invasive approach have been used for the Khortytsia beads. Nevertheless, the word "sample" is used here to indicate both the archaeological objects and the (representative) chips detached from them. All the samples were previously observed under a Leica MZ95 stereomicroscope at various magnification to select the regions of interest for the following analyses and to assess their state of conservation.

Then the Olbia Pontica set was analyzed by a LEO's SEM, model 50XVP coupled with an energy-dispersive microanalysis with an Oxford Silicon drift X-max detector (80 mm2) equipped with a Super ATW ©(Super Atmosphere Thin Window), which allows qualitative and quantitative point analyses to be obtained on the surface of samples.

On the other hand, the Khortytsia set was analyzed with a larger set of techniques according to a multi-analytical approach. FORS was employed to identify the dissolved colourants through an AvaLight-HAL-S-IND light source and an AvaSpec-ULS2048XL-USB2 spectrophotometer connected via a 2 m Y-shaped fiber optic probe. Measurements (350–1000 nm, 2.4 nm resolution) were taken at ~45° on 2 mm spots, averaged over 100 readings. Spectra were calibrated with a WS-2 reference and normalized; only informative data were used.

A portable X-ray fluorescence spectrometer (p-XRF) was used for all the samples to quantify the calcium concentration, used as an internal standard for LA-ICP-MS calibration [15] and to confirm the information that emerged from the LA-ICP-MS. The analysis was performed with an ELIO spectrometer, following the protocol described in [13,22]. While p-XRF results have higher bias than data from LA-ICP-MS, the results obtained on glass of know composition (two glasses specifically produced for interlaboratory testing by the Corning Museum of Glass – CMOG) calcium concentration were accurate enough to serve as internal standards (e.g., relative error: 5.16% for CMOG A, 0.65% for CMOG B).

LA-ICP-MS analyses were conducted to determine major, minor, and trace elements in the glass, using a NexION 300× (Perkin-Elmer, Waltham, USA) ICP-MS equipped with an ESI NWR 213 laser ablation system (ESI New Wave Research Co., Cambridge, UK). Analytical settings and procedures are given elsewhere [1,23,24].

μ-XRD analyses were performed to identify crystalline phases in the glass matrix using a SMARTLAB XE – Rigaku diffractometer with a Cu X-ray source (λ = 1.5406

Å) and HyPix-3000 detector. Scans (2 mm spot size) covered 5°–70° 2θ, with a 0.2° step and 10°/min increment. Patterns were interpreted using DIFFRAC PLUS EVA (v.7.0.0.1) against JCPDS-ICCD, ICSD, and PCPDFWIN databases.

III. RESULTS AND DISCUSSION

A. Preliminary study of Olbia Pontica set

The samples from Olbia Pontica were initially examined under a stereo microscope mainly to assess the state of preservation and the original colours. Observations at different magnifications provided a general overview of the samples and helped to guide their subsequent preparation as cross sections: a proper orientation of the sample allow to avoid altered areas and allow all the glass colours to be accessible to the analytical beams (fig. 1). In addition, the internal morphology shall be visible, as it can also offer insights into manufacturing techniques [12,22].

The polished samples were again inspected under the optical microscope in order to guide the subsequent analytical step under the SEM, where colours are not visible. The vitreous matrix was then analysed using a the SEM-EDS. A flat and polished surface is ideal to minimise the dispersion of the X-ray emitted by the sample and to guarantee the accuracy of the obtained data. Non-polished samples may be in fact suffer from artefacts linked to surface-sensitive phenomena, such as leaching and contamination. The analysis of the pristine glass under the object's surface ensures that the compositional features are correctly represented, and can be detected and quantified if they are above the detection limit of the instrument (approximately 0.01 wt%) [16].

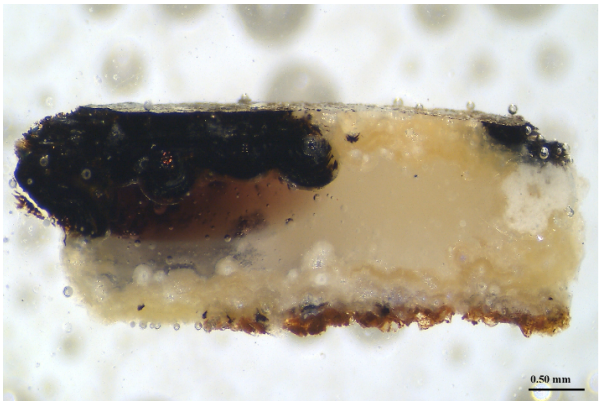

Fig. 1. Glass sample prepared as a cross section. The altered surface is visible along the entire edge of the fragment. Portions of different colours—opaque white and translucent violet—are clearly distinguishable.

As for the core-vessel samples, SEM observation generally confirmed their good state of preservation, with the exception of sample OB7A and OB2, where the level of the alteration was so advanced that the data could not be considered beyond the mere detection of the chemical elements.

The first millennium BCE was a period of experimentation in glass production in the Mediterranean. Some key elements allow us to distinguish between the different types of glass obtained with different technologies, with K_2O and MgO playing a major role. The analyses revealed that all the glass samples are low-magnesium glass (LMG), with MgO and K_2O values below 1.5% [25], suggesting that the fluxing agent used was natron, a natural mixture of minerals from evaporitic lake deposits, rich in sodium carbonates and bicarbonates, the most notable of which is trona ($Na_2CO_2 \cdot NaHCO_3 \cdot 2H_2O$). All samples showed similar K_2O and MgO contents, except for sample OB8A and OB3A, which exhibited slightly higher values. The composition of the first sample could be influenced by a diffuse network of fractures, which could have determined the leaching of the network modifiers [26]. The composition of the second sample could possibly indicate a recycling or re-melting processes [12].

As for the silica, the network former, it is generally assumed the sources may be highly pure quartz from pebbles or quartz veins, or quartz-rich sands [27]. Again, some key elements play a major role in identifying the source of silica. Specifically, the concentration of elements associated with feldspars or clays, such as Al_2O_3 and Fe_2O_3, and titanium-bearing minerals like ilmenite, rutile and titanite, help distinguish whether sand or another purer source of silica was employed. As for the samples considered here, the concentration of Al_2O_3 and Fe_2O_3 suggest the use of relatively pure sands [16]. Additionally, some samples contain in the glass the residues of incompletely dissolved quartz and feldspar grains.

In principle, SEM-EDS would allow to identify the elements associated with decorative features, including colorants and opacifiers. Nevertheless, in most cases their concentration are below the instrumental detection limit. In the considered sample set it was nevertheless possible to detect minimal amounts of CoO and CuO in blue glasses, and MnO in purple glasses [20].

Opaque white and yellow glasses were found to be rich in Sb, associated respectively with Ca and Pb. It is widely reported in the literature that these compositions correspond to calcium antimonates ($CaSb_2O_6$/$Ca_2Sb_2O_7$) and lead antimonate ($Pb_2Sb_2O_7$), respectively [16,21].

An exploratory comparison was also conducted using Al_2O_3 and Na_2O (fig.2) concentrations to attempt a preliminary classification, alongside the Mediterranean Group system proposed by archaeologists based on typology and chronology [9]. The same datasets used by Lü et al. were adopted for comparison; for further details, reference should be made to that study [7].

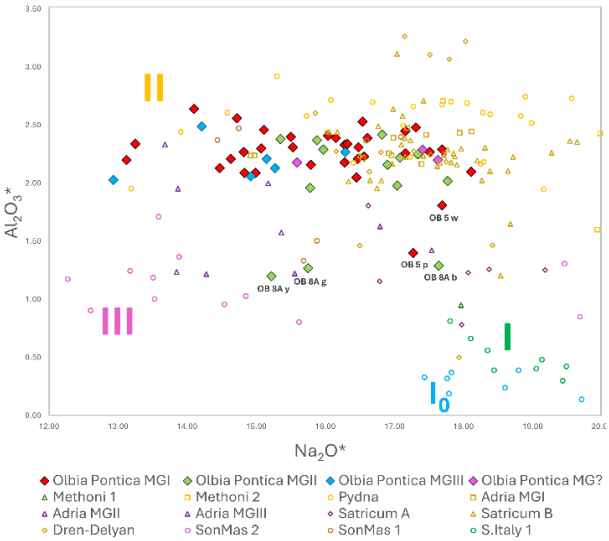

Fig. 2. Al_2O_3 vs Na_2O bivariate plot. The composition shows three major (I, II and III) and one minor (I_0) type for natron glass dated to the 8-2 c. BCE [7]. Data for samples from Olbia Pontica are also included.

Although related to the reduced set of elements that are detectable with SEM-EDS, it is nevertheless possible to obtain a preliminary picture of the relationship between archaeological and compositional classification for the samples from Olbia Pontica: all the samples assigned to Mediterranean Group (MG) III fall within the range of Lü's Type II glass, along with the samples with uncertain archaeological assignment. The samples attributed to MG I predominantly fall within Type II, with the possible exception of sample OB5, which may belong to Type III. Similarly, most of the samples assigned to MG II are positioned within Type II, except for sample OB8A, which plots within the Type III range. According to Lü's model [7], Type II glass is associated with Syro-Palestinian raw materials, while Type III glass is linked to Egyptian sources. However, further investigations focusing on trace elements are necessary to consolidate these preliminary results.

B. Archaeometric study of glass beads from Khortytsia island

The set of Khortytsia glass beads shows the integration of multiple analytical techniques to achieve a comprehensive, quantitative characterisation of their production, both in terms of bulk composition and decorative features. The investigation was mainly obtained through a non-invasive approach, as extensively reported in [21]. For major-to-trace elemental quantification as well as the identification of crystalline phases and coloring agents, the combination of LA-ICP-MS, p-XRF, FORS, and μ-XRD provided complementary insights. The measurement of major, minor, and trace elements in the glass matrix is obtained by LA-ICP-MS. Even within single artifacts, it was possible to distinguish between decorative layers and base glass composition because of its great sensitivity and spatial resolution. The accuracy of LA-ICP-MS determination was supported by internal standard calibration with Ca concentrations obtained *via* p-XRF. For two samples, polished cross-sections were obtained, allowing to demonstrate the quality of the results from the untreated surfaces of the archaeological objects. "Reduced" concentrations (normalized concentrations for SiO_2, Na_2O, K_2O, CaO, MgO, and Al_2O_3) enabled the robust classification of the base glass as natron-fluxed low-magnesia glass (LMG). Importantly, the high sensitivity and precision of LA-ICP-MS allowed for the detection of subtle compositional variations—such as slightly elevated K_2O levels in some samples. The large set of quantitative elemental data allows the samples to be grouped into two compositional clusters, primarily distinguished by their TiO_2 and CaO content. These elements, along with trace elements such as Sr, Zr, Th, enable more robust conclusions regarding the origin of the silica source through comparison with published reference groups [7]. Two compositional groups were identified: High Ca - Low Ti (associated with Type II glass and thus consistent with production in the Syro-Palestinian region) and Low Ca - High Ti (Type III glass, characterized by high concentration of components related to heavy minerals, which are indicative of Egyptian glass).

Internal heterogeneity was also highlighted by LA-ICP-MS, as beads of the same typology (such as cubic chevron beads) resulted to be made using chemically distinct glass, and multiple examples (such as KH3, KH8) comprise glass of both the compositional groups in the same item. This testifies that the bead-making workshop used glass of different origins interchangeably, showing that it was simultaneously supplied with glass from different trade routes. Despite the limited number of samples considered for the compositional analysis, the study outlines a clear direction for future investigations on these beads types, which are widely distributed in the regions north of the Black Sea.

Further analysis performed with μ-XRD and FORS are of a qualitative nature and are complementary to those performed with LA-ICP-MS, as they allow researchers to interpret and contextualize quantitative data more effectively. FORS proved effective in identifying characteristic electronic absorption bands associated with specific chromophores. In this study, blue glasses exhibited distinct absorption peaks at roughly 540, 590, and 645 nm, which are in line with the presence of Co^{2+} minerals. Wide bands in dark brown and black glasses suggested Fe^{3+} and Cu/Fe-related transitions, while green and turquoise regions displayed characteristics suggestive of Cu^{2+}. Although FORS does not provide concentration values, these spectral signatures offered qualitative confirmation of the colourants previously identified by

LA-ICP-MS, supporting the elemental assignments and enhancing the interpretation of colour technologies. µ-XRD, on the other hand, was used to investigate the presence of crystalline phases in the glass, especially, in the opaque colored decorations.

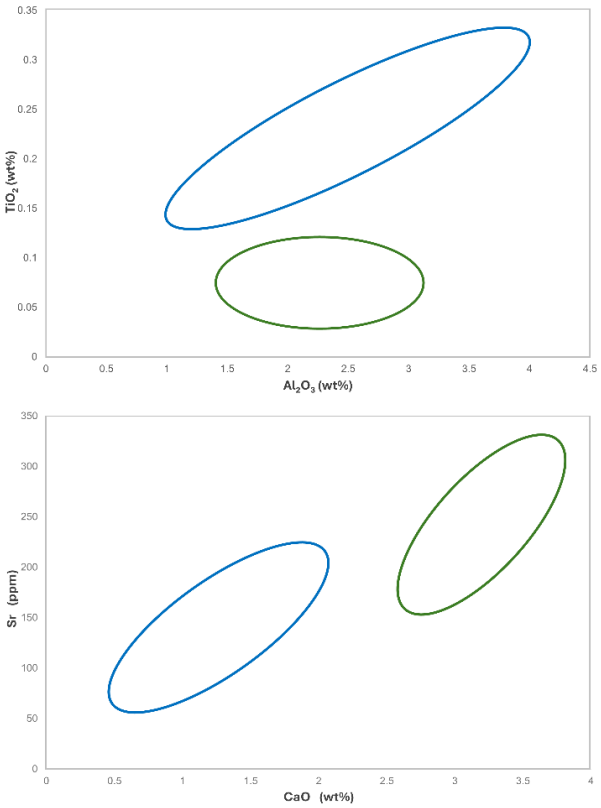

Fig. 3. (a) Al_2O_3 vs Ti_2O and (b) CaO vs Sr bivariate plots highlighting the two compositional groups for the beads from Khortytsia. Adapted from [21].

The yellow ones were confirmed to contain bindhemite ($Pb_2Sb_2O_7$), a widely used crystalline lead antimonate pigment for opaque yellow glass. This identification was consistent across several samples and aligned with the high PbO and Sb_2O_5 concentrations quantified by LA-ICP-MS. Analogously, contain calcium antimonate ($Ca_2Sb_2O_7$) was present in the white decorations, mostly in its orthorhombic form, suggesting prolonged heating at high temperatures during production—a technological choice likely aimed at achieving greater opacity and thermal stability. Even though results from µ-XRD were not quantitative, the technique can definitively identify phases, which is crucial information for reconstructing the chemical and thermal processes involved in glass production: the crystallization of orthorhombic calcium antimonate instead of the cubic phase indicates controlled heating and slower cooling [19].

IV. CONCLUSIONS

This work outlines the critical importance of a multi scale approach, from archaeology to archaeometry, for the study of archaeological glass. Quantitative and precise data are crucial for reconstructing technological traditions, raw material procurement, and ancient trade routes.

The combination of SEM-EDS, LA-ICP-MS, µ-XRD, and FORS allowed the acquisition of robust compositional data, minimising the effects of surface alteration and contamination. The preparation of polished cross-sections in nevertheless useful to obtaining reliable and representative bulk compositions. Quantitative analyses made it possible to detect even minor chemical variations, essential for differentiating between production technologies and raw material sources. At Olbia Pontica, SEM-EDS data enabled a preliminary classification of the samples into Types II and III, associated with Syro-Palestinian and Egyptian primary glass production. The Khortytsia beads, analyzed with higher-resolution techniques, revealed the coexistence of different compositional groups within the same archaeological contexts, suggesting complex supply networks and interactions between primary and secondary production centers.

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