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Analytical study of Roman glasses from Southeastern Spain

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Recent archaeological excavations carried out in the Iberian-Roman city of La Alcudia (Illicia, Hispania) have provided some important assemblages of Roman glass. The present paper summarizes the results of archaeological and archaeometric studies carried out on two assemblages from different sectors and chronology. The first set of glasses was unearthed in a sector corresponding to a section of the city's west wall. The level in which the glasses were found is dated from the mid 1st to the mid 2nd century AD. The second set of glasses comes from an area known as Casitas Ibéricas (4th–7th centuries AD). These glasses were found in ditches and pits, which had disturbed the more ancient archaeological levels. Most of the fragments in both sets represent blown glass. The archaeological study concentrated on determining the chemical composition of a representative selection of glass fragments from the two chronological periods in order to observe possible differences between them. Chromophores responsible for glass colour were identified. Moreover, the state of conservation of the glasses was evaluated in order to determine the nature of degradarion processes. The samples were studied using conventional optical microscopy (OM), X-ray fluorescence spectrometry (XRF), field emission scanning electron microscopy (FESEM), energy dispersive X-ray microanalysis (EDX), and visible spectrophotometry (VIS).

KEY WORDS: glass, Roman Period, archaeometry, chemical composition, colour, conservation state

INTRODUCTION: ARCHAEOLOGICAL BACKGROUND OF LA ALCUDIA

The archaeological site of La Alcudia (Illicia), located in the modern city of Elche (southeastern Spain) (Fig. 1), has been the object of irregular excavation for more than a century by now (Abad and Hernández 2004). Recent archaeological explorations in 2003–2007, undertaken in coordination with the La Alcudia Foundation for Archaeological Research and the Archaeological Team from the University of Alicante (Spain), have established a historical sequence, encompassing several levels corresponding to

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different cultures. Among these, the most important are those from the Iberian Period, the Roman Empire and Late Antiquity. The present work focuses on sectors 6B and 4C (Fig. 2), which provided some very interesting glass assemblages. The glasses did not come from original contexts, but rather from rubbish dumps and street surfaces, hence they are residual in layers testifying to the antiquity and populousness of the city. It is also why most of the glasses were quite fragmented.

Sector 6B

Fieldwork began in 2003 going on 2004 and was continued in 2006, resulting in the discovery of a 70-m long section of the city's west fortifications. Furtive digging in the past has left this wall in a varying degree of preservation.

During the first campaign in 2003, different glass objects, whole and fragmentary, were found and identified (some dishes and unguentaria) in a rubble level; all were dated between the late first and early second century AD, showing the general time frame of the levels documented in this area.
It is supposed that first furtive or disturbance actions (mainly of stones) took place from the end of the first century up to sixth-seventh centuries AD. Pottery from corresponding stratigraphic units, dated on the second part of the first century AD, was found together with some glass fragments, e.g., jug Isings forms 32–55, dark blue pillar-moulded shallow bowl Isings form 34, and other vessels, such as beaker Isings form 34.

Abundant pottery from the second part of the first century and the beginning of the second century AD was unearthed in the rubble levels. These units provided a small assemblage of glass vessels including the most common shapes of this period, such as mould-made pieces of colourless and coloured glass (the oldest, predominantly emerald green Isings forms 1–18 and 22 and colourless dishes Isings form 2, made around the end of the first century AD); blown glass (conventional and decorated vessels with wheel-cut lines); luxury pieces made of colourless glass (some of them with honeycomb decoration Isings form 21, one of the finest cups known); and bluish-green prismatic bottles Isings form 50, and unguentaria Isings form 28.

The last stages of wall plundering have been dated to the sixth-seventh centuries AD. From this unit comes a small decorated bottle of greenish glass with a coil applied around the neck.

Some other stratigraphic units (corresponding to test 5) yielded glass and pottery from the Islamic period. The recovered glasses are considered as residual. Another unit, containing decorated vessels of colourless glass, was dated between the fourth and fifth centuries AD based on the pottery. Of greatest significance in this set was a wine beaker with coil base and a second concentric coil inside (Isings form 85b) (Sánchez de Prado 2004a).

Sector 4C

Since the summer of 2005 the La Alcudia Foundation for Archaeological Research and the Archaeological Team from the University of Alicante have been conducting practical archaeological training in sector 4C, which is commonly referred to as Casitas Ibéricas. The dump, identified in the course of excavations, provided several glass fragments together with pottery dated to the end of the sixth and beginning of the seventh century AD, e.g., several glass dish fragments (Sánchez de Prado 2004a).

A silo with abundant ceramic and glass remains was also identified in another unit. The pottery reached into the seventh century AD. As for the glass, all the fragments can be assigned to common shapes in use from the second half of the fifth to at least the seventh century AD. The most common type is a shallow bowl with fire-rounded rim (42.8% of the whole ensemble), occurring together with jugs decorated with a spiral coil under their rim. Most of these vessels demonstrate relatively low quality and residual greenish or yelowish colours, which suggests that they were made probably from recycled glass. Therefore, either ceramic or glass materials from this sector are documented in levels related to the Late Antiquity, which definitely shed new light on several aspects of the archaeology of Ilici in the sixth-seventh centuries AD (Sánchez de Prado 2004b, forthcoming).

ARCHAEOOMETRIC STUDY OBJECTIVES

The present work focuses mainly on determining the chemical composition of a representative set of glasses found in sectors 6B and 4C of La Alcudia site. The objective is to trace possible differences between them. The evaluation of the conservation state of both sets of samples was also undertaken with the aim to provide a diagnosis of the pathologies connected with their degradation and to assign the chemical physical mechanisms related to such degradation. Furthermore, the research focused on characterizing the chromophores responsible for the different colours observed in the glasses.

EXPERIMENTAL PART

Sample selection

The recovered glass pieces were cleaned conventionally and in some cases restored, especially if the objects were to be exhibited. Thus, layers of degradation and products of corrosion were removed before carrying out the archaeometric study. Figure 3 gives a macroscopic view of a glass piece before and after restoration.

Among the whole ensemble of glass pieces from sectors 6B and 4C, 26 representative samples were selected for analysis. These were all small, shapeless fragments with no value for drawing, exhibition or conservation purposes (Fig. 4). The degree of degradation and transparency were varied, except for one piece, leaving no doubt that selected samples were of glass. The one exception was an opaque white piece (Fig. 4B), which did not show glassy brightness but a microcrystalline appearance, which suggested a particular study for this sample. As far as the colour is concerned, all the samples from sector 4C were olive green, while those from sector 6B were either colourless or coloured: blue, bluish-green, yellow and violet.

Characterisation techniques

The conservation state of the samples was observed by conventional optical microscopy (OM) using an Olympus DP-II microscope. The surface of glasses was observed without any preparation, that is, OM micrographs correspond to the state of the glasses as-received by the laboratory.
The chemical composition of the samples was determined by X-ray fluorescence spectrometry (XRF). A Philips PW-1410 wavelength dispersed X-ray spectrometer equipped with a tube of rhodium was used. Analytical determinations were obtained through standardless analytical software Uniquant 4.22 based on fundamental parameters. After being cleaned and the crust of corrosion completely removed, the samples submitted for chemical analyses were ground in an agate mortar. Then, pressed boric acid pellets were made, using a mixture of n-butylmethacrylate and acetone (10:90 wt%) as binding medium. Thus, the XRF semiquantitative results correspond to the average chemical composition of the bulk of glasses.

Field emission scanning electron microscopy (FESEM) was used with the aim of identifying degradation pathologies in microscale. A JEOL JMS 6500F microscope was employed, attached to an energy dispersive X-ray spectrometer (EDX) from Pentafet Link, with a windowless silicon (lithium) detector and using acceleration voltages of 15 kV. Semiquantitative EDX microanalyses were obtained by using the ZAF method of correction and allowed the study of particular surface features, as well as other local deteriorations in a cross-section of the samples. For FESEM observations and EDX microanalyses, the samples were coated with a thin layer of carbon in order to make them conductive.

The glassy nature of the white opaque sample was studied by X-ray diffraction (XRD). This analysis was undertaken with a Siemens D-5000 unit, using Kα of copper radiation (1.54056 Å), under set conditions of 40 kV and 30 mA between 2θ = 2°–80°. A small amount of a very fine powdered sample was used for XRD analysis.

The glass chromophores were characterised by visible absorption spectrophotometry (VIS) carried out with a Shimadzu 3100 instrument equipped with an integration sphere. Spectra were recorded in the 325–800 nm range on clean and plane-parallel samples of approximately 1 mm in thickness.

RESULTS AND DISCUSSION

Chemical composition (XRF)

Figure 5A shows the XRF results for chemical composition of the samples plotted in a ternary diagram. In diagrams of this kind, the main network glass former (SiO₂) and the two main network glass modifiers (Na₂O and K₂O) have been placed on two axes, respectively, while the other components (MgO, CaO, Al₂O₃, MnO and Fe₂O₃) have been accounted for on the third axis. All the chemical compositions of the glasses analysed lie inside the loop drawn in the ternary diagram (Fig. 5A). Moreover, the chemical composition of other Roman glasses from different sites and differently dated also lies inside that loop (e.g., Rincón 1984; Domínguez-Bella and Jurado-
Fig. 3. Fragment of a blue glass bottle from La Alcudia-Ilici (sector 6B): A) before restoration; B) after restoration.

Fig. 4. Main pathologies observed by OM on the surface of samples. A) Isolated and partially interconnected craters filled with corrosion products on colourless glass. B) Interconnected craters showing mass loss on colourless glass. C) Corrosion crusts causing interference colours in olive green glass.

Fig. 5. A) Compositional ternary diagram (wt %) for oxide components of the samples studied. Other glass compositions have been included for comparison purposes. B) Detail of the ternary diagram for Roman glass compositions. Results for Roman glasses from another site on the Iberian Peninsula have been included for comparison purposes.

Fresnedillo 2004; Gómez-Tubío et al. 2006; Carmona et al. forthcoming). This area corresponds to soda lime silicate glasses with a similar chemical composition typical of Roman glasses. As seen in Figure 5A, this kind of compositional data plotting allows different and similar compositions to be compared easily. For instance, average compositions of Islamic and Medieval glasses are pretty far from the loop of Roman glass compositions. On the other hand, similarities with average compositions of modern conventional glass can also be checked; even though modern glass has a higher SiO$_2$ percentage, that is, its composition is just within the border of the loop for Roman compositions. Figure 5B shows a detail of the ternary diagram in which only selected analytical results have been drawn in the interest of clarity. Comparison with analytical results formerly obtained in glasses from another Spanish Roman site (La Dehesa de la Oliva in Patones, Madrid, first to fifth centuries AD) indicate that both sets of glasses have a very close chemical composition. This suggests that batch mixtures and melting conditions were accurately controlled. Nevertheless, some small differences related to the SiO$_2$ content can be observed in Figure 5B, which varies between 63 and 73 wt%, approximately. This could be attributed to the period in which the glasses were produced, since the following tendency has been observed: most of the samples from the first to third centuries AD contain lower
SiO₂ percentages. Therefore, the careful evaluation of chemical composition of Roman glasses could reveal information indicating the production date, since changes in silica sources occurred between the Roman and post-Roman glasses (Aerts et al. 2003; Silvestri et al. 2006).

Moreover, a comparison of the contents of some oxides has brought some interesting results. Table I summarises average chemical composition of the samples from the two sectors of La Alcudia, as well as the average composition of glass samples from Patones. The low percentages of K₂O and MgO determined in all cases can indicate that the glasses were melted from natron as the only precursor of Na₂O (Sayre and Smith 1961). As is known, when soda-rich plant ashes are used as a source of Na₂O, K₂O and MgO percentages increase to 2-3 wt% and 4-6 wt%, respectively (Tite et al. 2006). Similarly, the low percentages of Al₂O₃ in all cases could testify to quartz sand being used as a SiO₂ precursor. When feldspars or ground chert are the SiO₂ source, the Al₂O₃ content can reach up to 5 wt%. MnO, Fe₂O₃ and TiO₂ percentages are very close in glasses from Patones and those from sector 4C of La Alcudia, while for glasses from sector 6B of La Alcudia the contents of these oxides are almost one order of magnitude lower. These results obviously suggest that most modern glasses from La Alcudia (sector 4C) and glasses from Patones are very similar, even with regard to percentages of oxides considered as impurities. In addition, the low content of MnO, Fe₂O₃ and TiO₂ and the higher SiO₂ percentage of glasses of the La Alcudia sector 6B demonstrate the better quality of these glasses in comparison with the two former sets. This fact agrees with the chronology of the sites: glasses from the fourth to seventh centuries AD correspond to the post-Roman period in which glass production declined into a lower quality, generally greenish colouring and repetition of decorative motifs. In addition, it also suggests that more ancient glasses could have been imported into Southeastern Spain from other parts of the Empire, while in post-Roman times most of the glasses could have been produced on a small scale in local workshops in the Iberian Peninsula (Sánchez de Prado 2004b).

State of conservation (OM)

OM examination detected three principal kinds of pathologies on the surface of all of the samples studied: i) isolated craters combined with interconnected craters in which corrosion products appear deposited (Fig. 6A); ii) very abundant craters, completely interconnected and in which loss of mass is evident (Fig. 6B); iii) corrosion crusted formed by stratified layers which produce interference colours (Fig. 6C). The layers forming corrosion crusted are easily detached and, in some cases, such corrosion crusted on the two sides of the sample are thicker than the unaltered glass remaining inside.

Degradation mechanisms (FESEM and EDX)

Under FESEM, isolated and partially interconnected craters filled with corrosion products were observed. Several concentric circles starting from a central point can sometimes be observed in the craters (Fig. 7A). This feature corresponds to the chemical attack produced by hydrolytic media under stationary conditions. The first consequence is a pit that increases in diameter, while insoluble products of corrosion are deposited on the corroded area. Hydrolytic attack continues inside the glass bulk and causes interconnection of neighbour pits (Fig. 7B). As seen in the table in Figure 7, the Na₂O content in areas 1 (surface) and 2 (crater) (Fig. 7A) are very low in comparison with the Na₂O percentage in the unaltered glass bulk (area 3 in Fig. 7B). This is due to a dealkalisation process enhanced by hydrolytic attack. When ground humidity interacts with the glass surface, ion-exchange of H⁺-ions from water by Na⁺-ions from glass takes place. The result is the leaching of the Na⁺-ions and the formation of Si-OH silanol groups in the glass surface. This explains the relative increase of SiO₂ in area 1 (Fig. 7A), i.e., the formation of a silica gel layer. In the crater (area 2 in Fig. 7A), corrosion products are deposited and a higher amount of insoluble compounds can be detected (e.g., Al₂O₃, MnO, Fe₂O₃, PbO).

The second stage of degradation is characterised by completely interconnected craters in which loss of mass has occurred. After cleaning and, in some cases, restoration, the elimination of the very brittle crust revealed a deeply degraded glass surface.

![Fig. 7. FESEM micrographs of partially connected craters filled with corrosion products on the surface of olive-green glass from sector 4C. A) View of the surface. B) View of cross-section. Attached table shows EDX results (wt %) for several areas.](image-url)
Although FESEM images show convex forms, these are in fact concave and correspond to completely interconnected craters. In Figure 8 (table), the corresponding EDX results from the glass surface are compared with the analysis obtained from unaltered bulk glass. The decrease in Na₂O percentage on the surface indicates a dealkalinisation process, which causes the SiO₂ to increase on the surface.

The third stage of degradation is the formation of corrosion crust due to accumulation and deposition of insoluble products. The microstructure of a crust formed by several layers can be observed in Figure 9. Mechanical resistance of overlapped layers is very brittle and causes easy detachment of the whole crust. Chemical composition of the corrosion crust (area 1 in Fig. 9) indicates an intense dealkalinisation process. EDX results shown in Figure 9 (table) confirm the loss of Na₂O and the corresponding relative increase of unleached SiO₂ and Al₂O₃ (glass-former oxides). For comparison purposes, the composition of area 2 in Figure 9 has been included. The increase in SiO₂ is connected with the formation of a characteristic silica-gel layer of glass exposed to humid environments for long periods of time. In addition, other oxides (Fe₂O₃ and MnO) increase their percentages as a consequence of accumulation on the surface from the glass bulk or from the ground environment.

Sample number 3 from sector 6B corresponds to the only white opaque material found (Fig. 4F). As mentioned above, this sample did not show a glassy brightness, but a microcrystalline appearance. The surface is slightly rough and somehow similar to a polycrystalline material. These macroscopic properties suggested that this sample was probably not glass. In order to clarify the glassy or crystalline nature of this sample, XRD patterns were recorded. The results obtained demonstrate the presence of CaCO₃ (calcite) with a low degree of crystallinity and a wide diffraction band which can be assigned to a glassy matrix. Therefore, since no definitive conclusion could be reached on the basis of XRD analysis, the sample was observed by FESEM and additionally microanalysed by EDX. Figure 10 shows FESEM micrographs of this sample and the table attached summarises EDX results obtained in the different areas observed. EDX results for area 2 (Fig. 10B) represent the average chemical composition of a material composed mainly of CaO, SiO₂, and MgO in a lower percentage. In addition, SO₃ has been detected, which may indicate the formation or deposit of calcium sulphate. On the other hand, EDX results for area 3 (Fig. 10C) indicate that the background of the surface of this sample could be strongly dealkalinised glass. This is explained by the high percentage of SiO₂, which could correspond to a silica gel layer formed on the surface as a consequence of the dealkalinisation process. This is also consistent with the small contents of alkaline (Na₂O and K₂O) and alkaline earth (MgO and CaO) oxides. For area 1 (Fig. 10A), EDX results showed a mixture of features found in areas 2 and 3.

Taking into account these analytical data, the white opaque sample from sector 6B could be a strongly dealkalinised glass with insoluble salts deposited on its surface.
The provenance of these salts is uncertain and more research is still needed to clarify whether the Ca²⁺-ions were deposited from the ground or from the glass bulk after a leaching process.

**Characterisation of chromophores (VIS spectrophotometry)**

All the samples with bluish, light-greenish and white residual hues showed VIS spectra with very weak absorption bands, mainly near the ultraviolet range. As Fig. 1A shows, diffuse features corresponding to the absorption of Fe²⁺-ions (440 and 1100 nm, blue colour) and Fe³⁺-ions (380, 420, and 440 nm, yellow colour) could be assigned. An additive chromatic combination of blue and yellow colour yields the characteristic olive-green residual hue of all the glasses from sector 4C. In bluish samples, the absorption bands corresponding to Fe²⁺-ions dominated over those of Fe³⁺-ions.

VIS spectra of deep blue samples (Fig. 1B) showed the three characteristic absorption bands of Co²⁺-ions at 538, 600, and 650 nm, respectively (Banford 1977). Moreover, these spectra also showed a less intense absorption band at about 435 nm, which can be attributed to Fe³⁺ and Fe²⁺-ions, responsible for a residual green colour masked by the intense blue hue of Co²⁺-ions.

The samples with violet colour showed visible absorption spectra dominated by a unique band at 499 nm. This band is undoubtedly assigned to Mn²⁺-ions (Fig. 1C). Likewise, the visible spectra of yellow samples showed a unique band at about 410 nm (Fig. 1D). Such a band could be assigned to Fe³⁺-ions (nominal absorption = 420 nm) and Ag⁺ atoms forming disperse nanocolloids (nominal absorption in the 400–420 nm range, depending on the size, shape and orientation of the colloidal particles) (Carmona et al. 2005). The symmetric Gaussian shape of the band strongly
suggests that $\text{Ag}^0$ colloids could be the chromophores responsible for yellow samples. Furthermore, silver-containing minerals or silver ores were well-known and easy to be obtained by Roman glassmakers.

CONCLUSIONS

Chemical and physical archaeometric characterization of two ensembles of glasses from the Iberian Roman city of La Alcudia-Heli (Conventus Cartaginensis, Hispania) has been carried out. The chemical composition determined for representative samples indicated very controlled and close recipes. However, some small but evident differences were observed, possibly to be explained in chronological terms, since changes in silica sources occurred between Roman and post-Roman glasses. Thus, the more ancient glasses from sector 6B could have been imported into Southeastern Spain from other parts of the Roman Empire, whereas the post-Roman glasses from sector 6C could have been produced in local workshops of the Iberian Peninsula. The relative proportion of oxide percentages determined suggests that natron was the only raw material used as a source of $\text{Na}_2\text{O}$. In addition, it also suggests that quartz sand was probably the only raw material used as a $\text{SiO}_2$ precursor.

As far as the conservation state is concerned, the glasses were unearthed in an advanced state of degradation. They showed deep pits, craters (partially or completely interconnected, depending on the fragment), corrosion crusters and surface layers causing interference colours. As a consequence of cleaning and, in some cases, restoration works carried out before the archaeometric study was accomplished, most of the samples lost the alteration layers and corrosion crusters.

The degradation mechanisms identified correspond to the characteristic chemical attack already described for other Roman glasses. That is: i) intense dealuminisation due to ground humidity interaction, ii) formation of silica gel layers on the glass surface, iii) mass loss in craters near the glass surface due to both burial and cleaning/restoration effects.

Finally, the chromophores responsible for the different colours of glasses were identified: $\text{Fe}^{2+}/\text{Fe}^{3+}$ (residual greenish, bluish, light blue and olive green); $\text{Co}^{2+}$ (deep blue); $\text{Mn}^{2+}$ (violet) and $\text{Fe}^{3+}$ and/or $\text{Ag}^0$ (yellow).

Acknowledgements

The authors wish to thank the financial support provided by the PIE-CSIC 20061 01 031 and the bilateral PAN-CSIC 2006 PL 0011 projects. N.C. acknowledges a CSIC-ESF postdoctoral contract. The professional support of the Historical and Cultural Heritage Thematic Network from the CSIC is also gratefully acknowledged.

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