Nota Técnica:
Patologías y estudio analítico de materiales procedentes de mosaicos de Carmona e Itálica

Technical Note:
Pathologies and analytical study of mosaic materials from Carmona and Italica

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RESUMEN

Este trabajo presenta los resultados del estudio realizado en muestras de mosaicos romanos procedentes de Carmona e Itálica (Sevilla). El objetivo principal del estudio consistió en la caracterización químico-física de teselas de vidrio y fragmentos de mortero para determinar su composición, evaluar su estado de conservación y estudiar su tecnología de producción. Las muestras se caracterizaron mediante lupa binocular, MEB-EDS, espectrofotometría visible, DRX y análisis petrográfico. Los resultados indicaron que las teselas se realizaron con vidrio de silicato sódico cálcico, aunque una de ellas mostró elevados contenidos de óxidos de plomo y de cobre. Las muestras procedentes de Carmona presentaron patologías de deterioro asociadas a la humedad retenida por el mosaico durante su enterramiento, mientras que las muestras de Itálica presentaron huellas de biodeterioro. Asimismo, los resultados indicaron que en las teselas se utilizó un vidrio de inferior calidad técnica que el empleado en piezas de vajilla de cronología semejante.

Palabras clave: teselas, vidrio, mortero, romano, deterioro.

SUMMARY

This work reports on the characterization of Roman mosaic samples from Carmona and Italica (Spain). A physico-chemical study of both glass tesserae and mortar fragments to determine their composition, to evaluate their state of conservation and to study their production technology was carried out. Samples were characterized by means of a binocular microscope, SEM-EDS, visible spectrophotometry, XRD and petrographic analysis. The tesserae were made from soda lime silicate glass, even though one of them showed high contents of lead and copper oxides. The samples from Carmona showed deterioration pathologies associated to the humidity affecting the mosaic during burial, while those from Italica presented traces of biodeterioration. The data also indicated that the glass used in the tesserae had a lower technical quality than that used in tableware pieces of similar chronology.

***Keywords:** tesserae, glass, mortar, roman, deterioration.*

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1. INTRODUCTION

The mosaic has been used throughout the History as a decorative item, even though it also plays a structural function since it is part of a floor or wall. They are composed of different layers named *statumen, rudus, nucleus*, mortar and the *tesserae* final layer (Figure 1a). The preparation of a Roman mosaic was documented by Vitrubio in *De Architectura* and by Plinio in *Naturalis Historia libri.* The first step of the process consisted on setting the *statumen* (formed by coarse stones), followed by the *rudus* (a lime and sand mortar mixed with stone fragments). Then the *nucleus* was added, which was composed of a lime mortar and finer elements such as grounded bricks or marble powder. On the *nucleus* the *sinopia* (a coloured design) was accomplished by means of ceramic or lead patterns, rules, strings, sectors, and other tools. Finally the *tesserae* were inserted (1, 2). These pieces, in general, were cubic in shape and could be produced on stone, marble, ceramic or glass. The different colouring of the *tesserae* allowed the elaboration from simple geometric motifs to complex figurative representations.

The most ancient mosaics were used as wall decoration. In the ancient city of Uruk (Fourth millennium BC), very simple geometrical motifs made of fired clay with black, red and white colours were found. During the seventh to the third centuries BC, the mosaics made with river or sea pebble stones were expanded throughout the Mediterranean basin. They were mainly used for decorating temples and sanctuary floors. This kind of mosaics was usually produced for private houses up to the first century BC. Nevertheless, the higher

development of mosaics was attained in the third century BC when the regular use of *tesserae* made with different materials definitively increased the variety of colours and shapes.

In the Iberian Peninsula the most ancient mosaics are dated between the seventh and the fourth centuries BC. They were produced with small stones which formed simple geometrical motifs. After the Roman conquest (third to second century BC), mosaic craftsmen coming from the Lazio were settled in the main cities of the Iberian Peninsula. They founded local workshops in which they made mosaics following Italic techniques and motifs. Polychrome *tesserae* started to be used in the second century AD, contemporary to the white and black pavement that was still used up to the fourth century AD (2-4).

The ancient city of Italica (Santiponce, Seville) is an example of the wide variety of mosaics. In this site more than one hundred mosaic pavements have been documented. Among them there are big rich polychrome decorated mosaics, as well as other simpler and less luxury. Unfortunately, a great part of these mosaics has been lost as a consequence of spoliation or poor conservation. In addition, those conserved are subjected to weathering conditions such as sun, rain and frost, as well as to local fauna and flora which attacked and disintegrated them slowly (5). In previous works carried out on several mosaics from Italica, the advanced biodeterioration experienced by both *tesserae* (6, 7) and mortars (8, 9) has been shown.

The main objective of the present research was the characterization of two sets of building materials formed by glass *tesserae* and mortar fragments from a mosaic of Carmona and three mosaics of Italica. The characterization was undertaken with the purpose to determine their chemical composition and to evaluate their state of conservation in order to diagnose deterioration mechanisms developed on such materials. Finally, this work was also focussed on determining some technological aspects concerning production of these materials.
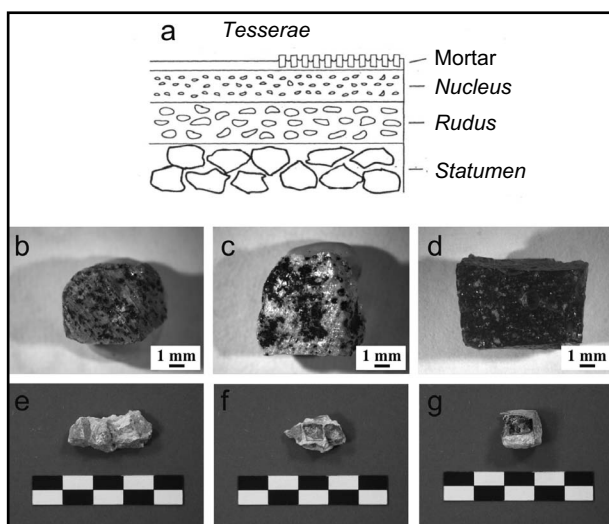

Figure 1. a) Cross-section scheme of a classic mosaic pavement according to reference (2), b) Image from sample C-7, c) Image from sample C-12, d) Image from sample I-3, e) Image from sample C-3. f) Image from sample C-5, g) Image from sample I-2 (scales in cm).

2. EXPERIMENTAL PART

2.1. Description of samples

Two sample sets from Carmona and Italica (Seville) have been studied. Samples from Carmona belonged to a mosaic fragment recently discovered during public works. The fragment corresponds to the right bottom part of a big mosaic that represent the four seasons. The first set is composed of one complete *tessera*, seven *tessera* fragments and four mortar fragments (Table 1). The *tessera* fragments were directly taken from the inner side of the mosaic (Figures 1b and 1c).

The mortar fragments showed remains of silica gel layers from the *tesserae* to which they were adhered (Figures 1e and 1f). The colour indicated in Table 1 for mortar samples refers to the colour of this silica gel layer.

The second set of samples belonged to three different mosaics dated on the second century AD from the city of Itálica (Santiponce, Seville): the Neptune mosaic from the Neptune House, the Planetary mosaic from the Planetary House and the Tellus mosaic from the Birds House. The set is composed of 5 samples: one complete *tessera*, three *tessera* fragments and one grip mortar to which a glassy *tessera* and a calcareous *tessera* were adhered (Table 1). None of the samples is higher than 2 cm in length (Figures 1d and 1g). The Tellus samples presented, when they were taken before 1991, some parts of the original mortar and evidences of the biodeterioration caused by lichens (7).

2.2. Characterization techniques

The characterization techniques used to study the set of samples have been the following: binocular microscope, scanning electron microscopy and energy dispersive X-ray spectrometry (SEM-EDS), reflection visible spectrophotometry, X-ray diffraction (XRD) and petrographic analysis.

Binocular microscope observations were undertaken with a Leica model MZ16, provided with a digital camera Leica model DC300. SEM observations were carried out with a Hitachi microscope model S-3400-N (CCHS). The samples were observed on their surface and on a polished cross-section. Prior to observations the samples were coated with carbon as a conductive medium, by using a Sputter Coater Polaron SC7620. EDS microanalysis were accomplished with a spectrometer Bruker AXS (133 eV) attached to the electronic microscope.

The study of chromophores was undertaken by reflection visible spectrophotometry with an equipment Ocean Optics model HR 4000 CG. Spectra were recorded in the 250-1100 nm wavelength range.

Crystalline phases were determined with a Phillips X'Pert MPD diffractometer, using the copper $K\alpha$ radiation (1.54056 Å), under working conditions of 45 kV and 40 mA. X-ray diffractograms were recorded between $2\theta = $ 5-60º on powdered samples obtained in an agate mortar to a grain size lower than 30 µm.

Petrographic analysis was carried out with a light transmitted optical microscope Kyowa model Bio-Pol 2, equipped with a polarization device and a rotating platen. Petrographic micrographs were obtained with a digital camera Leica model DFC 480.

3. RESULTS AND DISCUSSION

3.1. Surface characterization of glasses
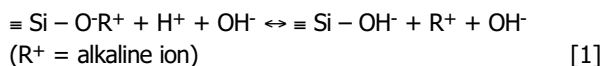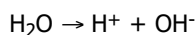
Samples from Carmona

Samples from the mosaic of Carmona showed breaks, cracks and wearing. Moreover, all the *tesserae* presented

Table 1
Description of the samples studied.

Name[a]	Mosaic	Sample	Colour
C -1	Four season	*Tessera* fragment	Turquoise
C-2	Four season	Complete *tessera*	Turquoise
C-3	Four season	Mortar	Green
C-4	Four season	Mortar	Green
C-5	Four season	Mortar	-
C-6	Four season	Mortar	White
C-7	Four season	*Tessera* fragment	Greyish blue
C-8	Four season	*Tessera* fragment	Dark blue
C-9	Four season	*Tessera* fragment	Grey
C-10	Four season	*Tessera* fragment	Dark blue
C-11	Four season	*Tessera* fragment	Grey
C-12	Four season	*Tessera* fragment	Grey
I-1	Neptune	*Tessera* fragment	Turquoise
I-2	Neptune	Mortar with *tesserae*	Dark green
I-3	Planetary	*Tessera* fragment	Dark blue
I-4	Tellus	*Tessera* fragment	Greyish blue
I-5	Tellus	Complete *tessera*	Dark green

[a] Abbreviations: C (Carmona), I (Italica).

iridescent silica gel layers (Figure 2a). Such iridescent layers are due to surface dealkalinization. The soil humidity directly in contact with the mosaic pieces causes the ion-exchange of water protons H+ with the alkaline ions from the alkaline and earth-alkaline oxides of the glass (Na_2O, K_2O, MgO) following the reaction [1].

$$H_2O \rightarrow H^+ + OH^-$$

$$\equiv Si - O^-R^+ + H^+ + OH^- \leftrightarrow \equiv Si - OH^- + R^+ + OH^-$$
$$(R^+ = \text{alkaline ion}) \hspace{3cm} [1]$$

Humidity is propagated through the cracks and breaks of the mosaic pieces. That is why iridescent layers are also formed in the inner part of the *tesserae*, which could affect the mechanical integrity of the piece (Figures 2a and 2d). Silica gel layers showed white or yellowish colouring.

In between iridescent layers of the glass samples, dark dendritic deposits were detected (Figures 2b and 2e). Sometimes such deposits coated the entire sample surface, as can be observed in sample C-10. EDS microanalyses carried out on the dark deposits of the most attacked samples (samples C-2, C-10 and C-11) showed a high content of lead oxide (PbO), iron oxide (Fe_2O_3) and manganese oxide (MnO) (Table 2).
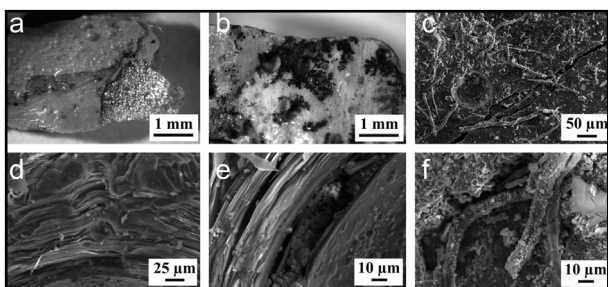

Figure 2. a) Binocular microscope image of the iridescent layer from sample C-1, b) Binocular microscope image of dark deposits from sample C-11, c) SEM micrograph of calcareous deposits from sample C-10, d) SEM micrograph of iridescent layers from sample C-10, e) SEM micrograph of dark deposits from sample C-10, f) SEM micrograph of calcareous deposits from sample C-12.

On the surface of samples C-10, C-11 and C-12 some filamentous structures were observed containing a high calcium percentage (Figures 2c and 2f), which could be the result of biological activity in the past.

Samples from Italica

The *tessera* fragments from Italica showed the usual pathologies of a chemical attack developed after biological interaction (Figure 3a), as previous works conducted with similar pieces demonstrated (7), even though some *tessera* surfaces did not show evidences of the biological attack (Figure 3b).
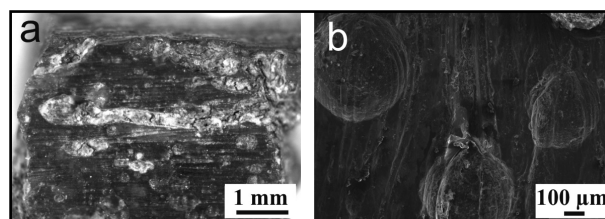

Figure 3. a) Binocular microscope image of evidences of biological attack in sample I-1, b) SEM micrograph from the surface of sample I-1.

3.2. Characterization of the glass bulk

The glass bulk characterization of *tesserae* gathers valuable information on the original glass since, in contrast to the surface, it has not experienced deterioration processes. From a total of 17 samples, only 4 (C-2, C-10, C-11 and I-2) presented a sufficient size to undertake a confident study of the glass bulk of the piece.

Samples from Carmona

The chemical composition of samples C-2, C-10 and C-11 corresponds to a soda lime silicate glass (Table 2). This is the common composition of most Roman glasses of this chronology. The macroscopic observation from the bulk of pieces revealed a glass with many bubbles and not melted inclusions with brown and black colours (Figure 4). This indicated a low quality and poor refined glass.

The sample C-2 is an opal glass, turquoise blue coloured. EDS microanalyses carried out on its polished surface, and consequently on the bulk glass, showed a high content of antimony oxide (Sb_2O_3 = 3.9 wt. %) (Table 2) that could be attributed to the presence of calcium antimonite. This compound was used in the Iberian Peninsula up to the fifth century AD as an opacifying agent (10-12). On the contrary, samples C-10 and C-11 are translucent glasses with a Sb_2O_3 content lower than 2.5 wt. % (Table 2).
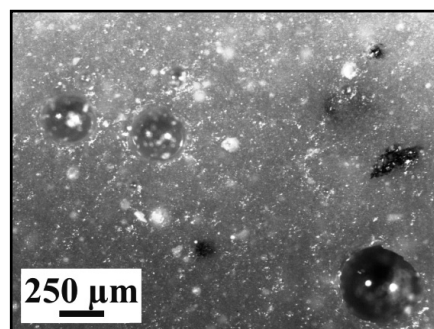

Figure 4. Binocular microscope image of bubbles and not melted particles from sample C-2.

Table 2
Average results of EDS microanalyses of the samples studied.

| Comp. (wt. %) | Samples [a] | | | | | | | | | | | |
| | C-2 | | C-4 | | | | C-10 | | C-11 | | I-2 | |
	Int.	Dep.	Iris.	Dep.	C. roja	Mort.	Int.	Dep.	Int.	Dep.	Int.	Mort.
Na_2O	16.9	0.3	1.4	0.9	0.1	0.4	20.6	0.4	17.0	0.7	6.8	0.4
MgO	0.7	1.2	0.9	2.0	0.2	0.2	0.7	0.8	0.3	1.2	0.6	2.3
Al_2O_3	2.5	9.6	10.6	10.7	2.1	1.6	3.0	5.7	2.2	5.8	3.0	8.3
SiO_2	56.1	31.2	61.7	20.3	8.5	6.9	61.9	28.0	66.7	32.5	41.5	21.0
P_2O_5	0.2	1.8	0.2	1.0	0.1	0.4	0.1	1.6	n.d.	1.2	0.1	2.8
SO_2	0.1	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.
Cl^-	0.7	0.4	n.d.	n.d.	n.d.	0.2	1.1	0.4	1.0	0.3	0.8	0.2
K_2O	1.2	2.6	4.8	2.3	1.0	0.7	0.7	2.4	0.8	1.7	0.5	1.5
CaO	8.5	25.8	9.9	42.9	66.4	87.8	7.4	14.8	7.2	16.0	7.5	56.9
TiO_2	0.4	1.3	0.8	1.4	0.6	n.d.	0.4	3.6	0.2	3.6	n.d.	0.7
MnO	0.8	7.8	0.2	5.0	0.6	n.d.	0.9	19.5	0.5	13.6	0.2	n.d.
Fe_2O_3	1.1	10.5	3.2	10.2	19.2	1.6	1.0	16.4	1.0	12.9	4.9	3.7
CoO	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	1.4	n.d.	1.2	n.d.	n.d.
CuO	5.9	2.0	0.9	1.5	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	9.8	n.d.
Sn_2O	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	1.9	n.d.
Sb_2O_3	3.9	n.d.	5.0	n.d.	n.d.	n.d.	1.5	n.d.	2.2	0.9	n.d.	n.d.
PbO	1.0	5.5	0.3	1.9	1.1	0.3	0.6	5.0	0.8	8.4	22.3	2.2

[a] Abbreviations: Int. (sample bulk), Dep. (dark deposits), Iris. (iridescent layer), C. roja (red layer), Mort. (mortar), n.d. (not detected).

Samples from Italica

Sample I-2 presented a very different chemical composition in comparison with the other pieces analyzed, either from Italica or Carmona. It is a soda lime silicate glass containing high percentages of lead oxide (PbO = 22.3 wt. %) and copper oxide (CuO = 9.8 wt. %) (Table 2). This composition is similar to that used in frits and tile enamels of later chronology (fourteenth century AD) (13), and suggests a possible reusing of wastes from other glasses. However, the restoration and consolidation processes undertaken in this mosaic during the twentieth century, in which some polyester *tesserae* and glass from contemporary bottles were even used, could explain the different composition of this *tessera*.

3.3. Characterisation of the glass colours

The samples studied did not present a sufficient size to prepare a slab (1 mm in thickness) to be analyzed by absorption spectrophotometry in the visible range. For this reason the study of chromophores was undertaken by visible reflection spectrophotometry, even though the signal obtained by this method is weaker and less resolved than that obtained by the absorption method. In most cases the habit of the spectra did not allow the complete assignment of chromophores.

Samples from Carmona

The study of samples C-2, C-10 and C-11 was carried out on a clean and polished surface of the pieces. The reflection spectrum of sample C-2 showed the characteristic band of Fe^{2+}-ions (1007 nm). The turquoise colouring and the EDS analysis of this piece (Table 2) indicate the overlapping of the Fe^{2+}-ion band with that of the Cu^{2+}-ion (not resolved in the spectrum). The spectrum of the dark blue sample C-10 presented the characteristic bands of Co^{2+}-ions (535, 591 and 648 nm, respectively) and Fe^{2+}-ions (1011 nm). In the spectrum of sample C-11, the reflection bands of Fe^{3+}-ions (377, 420 and 460 nm, respectively) were assigned (Figure 5).

Samples from Italica

Chromophores characterization was carried out on the surface of the glass samples I-1, I-3, I-4 and I-5, even though the surface irregularity diminished notably the spectra resolution. The habit of the spectra from samples I-1 and I-5 did not allow a conclusive assignment of chromophores. The spectrum of sample I-3 (dark blue) showed the absorption bands of Co^{2+}-ions at 536, 592 and 649 nm, respectively. In the reflection spectrum of sample I-4, the bands of such ions were assigned at 540, 596 and 651 nm, respectively, as well as the bands of Fe^{3+}-ions at 380, 425 and 457 nm (Figure 5). The blue greyish colour of the sample is attributed to

the chromatic combination of both chromophores: cobalt and iron.

3.4. Characterization of the mortar

The mortar is the layer, together with the *nucleus*, the *rudus* and the *statumen*, on which the *tesserae* are placed (Figure 1a). Mortar samples C-3, C-4 and C-6 presented remains of iridescent layers from the corresponding extracted *tesserae*, whereas sample I-2 exhibited two *tesserae* adhered, one of them glassy and the other calcareous.

Samples from Carmona

Crystalline phases detected by XRD (calcite and quartz) (Figure 6a), petrographic results (Figure 6b) and the

mortar chemical composition (87.8 CaO, 6.9 SiO_2 wt. %) indicated the use of a lime mortar with a small proportion of silica sand. This kind of mortar is commonly used in mosaics of this chronology. Sample C-4 is the only one with an additional red layer between the mortar and the iridescent layer, showing variable thickness and porous appearance (Figure 7a and 7b). Its EDS analysis (Table 2) indicated a high content of Fe_2O_3 (19.2 wt. %). This layer could be related to the *sinopia* used by craftsmen to elaborate the design prior to the mosaic production (1, 2). Between the red layer and the iridescent layer another fine stratum with high contents of iron oxide (Fe_2O_3), manganese oxide (MnO) and lead oxide (PbO) (Table 2) (Figures 7b, 7c and 7d) was located. Such fine stratum is connected with dark deposits found in other pieces (section 3.1, *Samples from Carmona*).
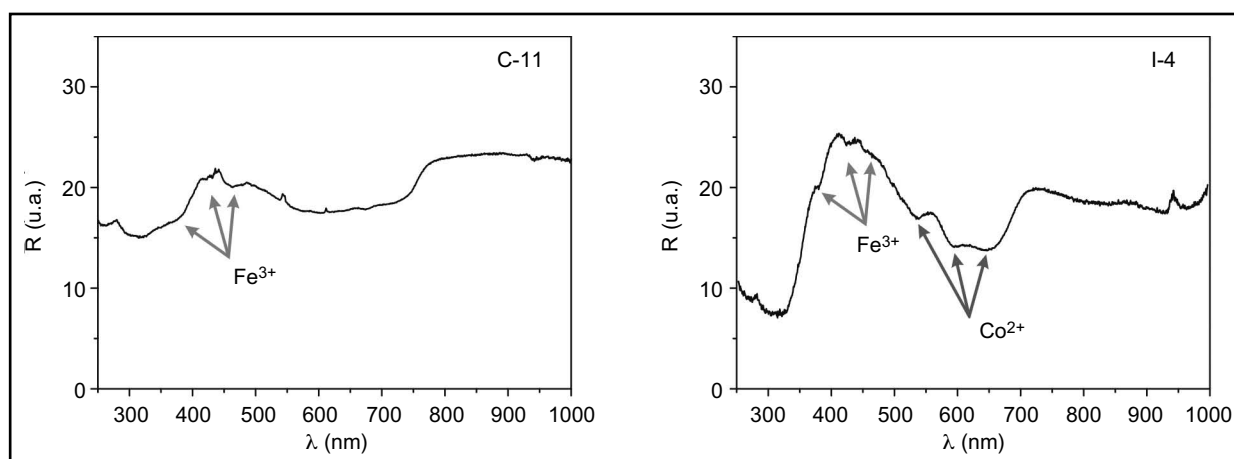

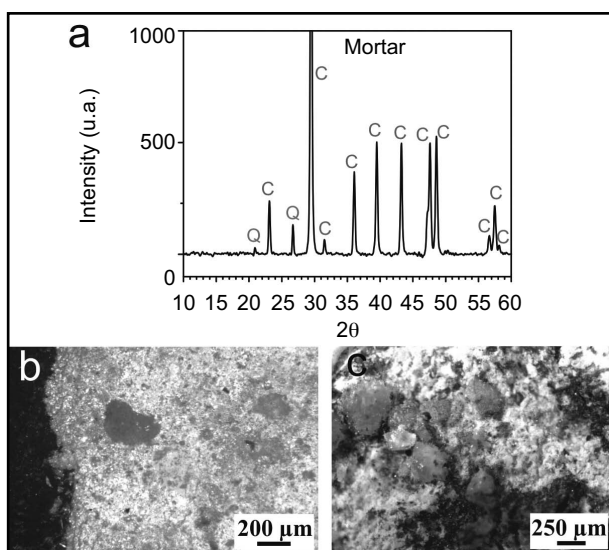Figure 5. Optical reflection spectra from samples C-11 (grey) and I-4 (greyish blue).

Figure 6. a) X-ray diffractogram of the mortar from Carmona, b) Thin section image of the mortar from Carmona, c) Binocular microscope image of the mortar from Italica.
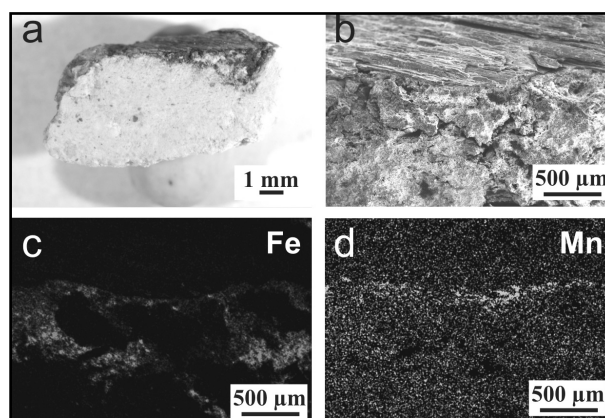

Figure 7. a) Binocular microscope image of the different layers from sample C-4, b) SEM micrograph of the different layers from sample C-4, c y d) Mapping of the different layers from sample C-4.

Samples from Italica

Sample I-2 is a mortar fragment adhered to a glassy *tessera* and a calcareous *tessera*. On its surface big sand grains (250 μm) have been detected, as well as lichen remains described in previous works (9) (Figure 6c). EDS surface microanalysis of the mortar showed a lower concentration of CaO and higher content of SiO_2 and MgO (Table 2). This result could indicate that this lime mortar contains more silica sand than that used in the mosaic from Carmona. The presence of other crystalline phases, as dolomite, albite or aragonite, identified in mortars of the same chronology (14) must not be discarded. This mosaic was consolidated and restored with reposition materials and filling mortars in the twentieth century. Therefore, it is not possible to assign a Roman origin for this mortar.

4. CONCLUSIONS

The characterization of a set of mosaic samples from Carmona and Italica determined the chemical composition of the glass *tesserae* and their state of conservation. The majority of *tesserae* were produced with soda lime silicate glass, which is the common composition of Roman glasses used for tableware pieces and windows. Nevertheless, one of the samples from Italica showed a different chemical composition characterized by high contents of lead and copper oxides. Such composition could suggest the reusing or recycling of contemporary glass wastes. Moreover, the presence of bubbles, strings and not melted particles in the *tesserae* indicates the use of a low quality glass in comparison with that used in tableware glass.

Samples from the mosaic of Carmona showed a severe surface deterioration. The deep dealkalinization process experienced by the pieces caused the formation of thick iridescent layers which, together with dark deposits, have completely transformed the surface. In addition, deterioration has also damaged the integrity of the pieces since they were irreversibly fissured, fractured and fragmented. On the contrary, samples from Italica showed evidences of a chemical attack from biological origin. Such samples have been subjected to the attack of lichens, due to their outdoor exposition for many decades in which no cleaning or maintenance treatments were accomplished.

ACKNOWLEDGEMENTS

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