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The three polychrome mosaics of S. Aloe quarter in Vibo Valentia (Calabria, Southern Italy): chemical characterization of glass tesserae

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***Abstract* – In the present study, the glass tesserae from three Roman mosaics located in S. Aloe quarter -Vibo Valentia (Calabria Italy) were analysed, with the aim of investigating the colouring and opacification techniques.**
The micro-chemical characterization of the tesserae was conducted by means of EMPA-WDS and LA-ICP-MS.
Results show that all the tesserae are natron-based and chemically comparable with major Roman compositional groups. In addition, the colouring/opacification techniques identified are consistent with the presumed Roman dating of the mosaics.

I. INTRODUCTION

In the Roman time, the mosaics were considered luxury goods, especially if the tesserae were made of rare and expensive materials such as marble and/or coloured glass [1].

Generally, opaque and coloured glasses were used to produce the desired effects, indeed the tesserae were applied near each other to create beautiful images [1].

Glass was mainly produced using a mixture of three main components: silica sand, lime and alkaline compounds or "fluxants" able to facilitate its production [2]. The ratios of the three components and the amount and type of fluxants allow the separation of glasses on a compositional basis. In addition, the chemical characterization of glasses provides important information, not only on the composition of raw materials, but also on the period and method of glass production [3].

The research work conducted in [2] traced the different use of alkaline fluxes in the Mediterranean and European world from the 2nd millennium BC and the first half to the 2nd millennium AD.

In detail, the glass was produced using the natron (a soda rich mineral salt) as flux and a sand rich in calcium, as stabilizer [4,5] from the first millennium BC until the VIII century AD. Subsequently, natron was gradually substituted by ashes of coastal plants containing, in addition to sodium, high percentages of potassium and magnesium.

This paper reports the results of an archaeometrical investigation on the glass tesserae of the mosaics, dated between the II and III centuries AD, discovered in the archaeological area of S. Aloe in Vibo Valentia (Calabria, Southern Italy).

The colour was determined by the means of a spectrophotometer.

The chemical analyses were conducted by EPMA-WDS (Electron Probe Micro Analyser with Wavelength-Dispersive Spectroscopy) and LA-ICP-MS (Laser Ablation with Inductively Coupled Plasma Mass Spectrometry).

The glass tesserae were analysed in order to determine their composition and consequently, the technological process of production. In this regard, similarities and/or differences among tesserae from the three mosaics were examined to allow the individuation of the glassmaking techniques applied. In addition, the comparison between our results with the major Roman compositional groups provided the possible provenance of raw materials employed for glassmaking production.

II. THE S. ALOE ARCHAEOLOGICAL SITE

The S. Aloe quarter in the city of Vibo Valentia is an archaeological area dating back to the period between the first century BC to the fifth century AD [6-8].

Originally, Vibo Valentia was a Greek colony, founded by the Locri people at the end of the VII century BC. It became then a Roman colony in 192 BC [9].

The city saw its golden age during the Roman Empire, especially in the III century AD.

This archaeological quarter expands for about thirty thousand square meters, at about 470 meters above sea level.

In the S. Aloe quarter, three polychrome mosaic floors were found in a good state of preservation. They are named Nereid, Geometric and Four Seasons. They differ in their iconography, the period in which they were realized and their executive techniques [10,11].

Fig. 1. The three mosaics studied: a) Nereid; b) Geometric; c) Four Seasons.

The Mosaic with the emblem of the Nereid shows a naked Nereid riding a hippocampus swimming in the sea surrounded by three dolphins (Figure 1a). It covers a square area of 4.00m x 4.26 m. The dolphins, waves, the Nereid mantle and the hippocampus body were depicted using grey and blue glass tesserae. The different shades of colour produce beautiful light and shadow effects.

The Geometric mosaic is arranged in two areas (Figure 1b). The first one includes a rectangular horizontal space composed of white tesserae. It covers an area of 4.50m x 5.95 m. The second one, with vertical development, includes a carpet of rectangular shape in which, on parallel vertical rows, a motif with white crosses develops. It covers an area of 6.70m x 3.0m. A band of white tesserae, adorned with swastikas and black squares, indicates the passage from one field to another of the mosaic.

The third mosaic was discovered in the thermal baths area of S. Aloe quarter. It is enriched with several plant-related elements and animal-related details, such as fruit tree, rose shoots, aquatic plants, peacocks, partridges, ducks and a pheasant. At the four floor angles there are female figures symbolizing the four seasons [8] (Figure 1c). It was realized with both vitreous and stone tesserae. The vitreous tesserae are variously coloured: grey, blue, yellow, green and red (see Table1). The stone tesserae were used for the opaque areas of the mosaic. The macroscopic observation indicates the use of different kinds of stones such as marble, volcanic, and sedimentary rocks. It covers a square area of 4.80m x 5.10m.

All the three mosaics were dated between the II and III centuries AD.

III. MATERIALS AND METHODS

The sampling of 32 glass tesserae from the three mosaics was conducted with the aim to take all the various colours present.

The tesserae were collected from detached parts of the flooring, in 2018 during the restoration phase.

Table 1 lists all studied samples giving information about their mosaic of provenance (Nereid, Geometric, Four Seasons) and colour.

This latter one was determined precisely thanks to Colorimetric analysis. In fact, the colorimetric coordinates of all tesserae was measured by a Konica Minolta CM-2600D spectrophotometer (Chiyoda, Tokyo, Japan), through the determination of L*, a*, and b* colorimetric coordinates of the CIELAB (Commission Internationale de l'éclairage L = L*, A = a*, and B = b* colorimetric coordinates) space.

The measurements of Nereid and Geometric were reported in Table 2, whereas Four Seasons data were collected in [12].

The chemical characterization was conducted using two complementary micro-analytical techniques EPMA-WDS (Electron Probe Micro Analyser with Wavelength-Dispersive Spectroscopy) to determine major element concentrations and LA-ICP-MS (Laser Ablation with Inductively Coupled Plasma Mass Spectrometry), to detect trace element contents.

Before the analyses, glass tesserae were polished with diamond clothes and observed using a stereomicroscope.

EPMA analyses were carried out using a JEOL-JXA 8230, equipped with 5 Spectrometers WDS. Moreover WDS distribution maps are edited in RGB color model.

The EPMA-WDS was used under the following operating conditions for chemical analyses: acceleration voltage 15 kV, probe current 10 nA and a defocused beam of 10 μm to avoid the loss of alkali. Furthermore, to prevent the loss of Na and ensure the correct acquisition of other element concentrations, an acquisition time of 15 s was selected.

LA-ICP-MS. analyses were carried out using an Elan DRCe instrument (Perkin Elmer/SCIEX), connected to a New Wave UP213 Nd-YAG laser probe (213 nm). The procedures for data acquisition were those normally used in the EMPA and LA-ICP-MS laboratories of the Department of Biology, Ecology and Earth Sciences, University of Calabria (Italy) [13-15]. In particular, ablation was performed with spots of 50 – 80 microns, with a constant laser repetition rate of 10 Hz and fluence of about 20 Jcm^{-2}. For all analyses a transient signal of intensity versus time was obtained for each element using a 60 s background level (acquisition of gas blanks), followed by 40 s of ablation and then 60 s of post-ablation at background levels. Data were transmitted to a PC and processed by the Glitter program. Three point analyses were carried out on each glass tessera selecting the portions without alterations.

Table 1. List of mosaic's tesserae collected in the Nereid, Geometric and Four Seasons mosaics inside the archaeological area of S. Aloe in Vibo Valentia.

Label	Mosaic	Color
QsGr1, QsGr2	Four Season	grey
NA1, NA2, NA3, NA4	Nereid	light blue
QsA1, QsA2, QsA3	Four Season	
GBl	Geometric	blue
NBl	Nereid	
QsBl1, QsBl2, QsBl3	Four Season	
GV	Geometric	green
NV1, NV2, NV3, NV4	Nereid	
QsV1, QsV2, QsV3, QsV4, QsV5, QsV6, QsV7, QsV8, QsV9, QsV10	Four Season	
NG1	Nereid	yellow
QsG1	Four Season	
QsR1	Four Season	red

IV. RESULTS AND DISCUSSION

The samples are characterised by SiO_2 content varying from 64 wt.% to 71 wt.% (except for the yellow and red tesserae of the Four Seasons mosaic containing respectively 60.18 wt.% and 58.3 wt.%), Na_2O content from 13 wt.% to 20 wt.%, (only the yellow tessera of the Four Seasons and the blue one of the Geometric show respectively 10 wt.% and 11.5 wt.%), CaO ≈ 5 - 7.5 wt.%, and low Al_2O_3 (≈2 - 3 wt.%).

Table 2. Colorimetric measurements carried out on tesserae of Nereid and Geometric mosaics. Notes: L for the lightness from black (0) to white (100), a* from green (−) to red (+), and b* from blue (−) to yellow (+).*

Tessera	Mosaic	Color	Colorimetric Coordinates		
			L*	a*	b*
NA1	Nereid	light blue	45.39	-0.96	-9.86
NA2	Nereid		48.07	-2.11	-11.62
NA3	Nereid		52.67	-2.21	-10.32
NA4	Nereid		60.91	-3.84	-5.51
GBl	Geometric	blue	37.70	5.87	-25.62
NBl	Nereid		33.35	5.20	-22.29
GV	Geometric	green	41.58	-13.77	8.64
NV1	Nereid		48.99	-14.98	13.57
NV2	Nereid		50.75	-2.43	17.31
NV3	Nereid		34.57	-7.58	-3.23
NV4	Nereid		45.88	-3.07	-2.93
NG1	Nereid	yellow	63.95	6.85	36.53

The K_2O and MgO contents are below 1.50 wt.% which are coherent with the glassmaking tradition of natron-fluxed glass (Figure 2). Only the green tessera NV4, collected from the Nereid mosaic contains K_2O= 1.77 wt%.

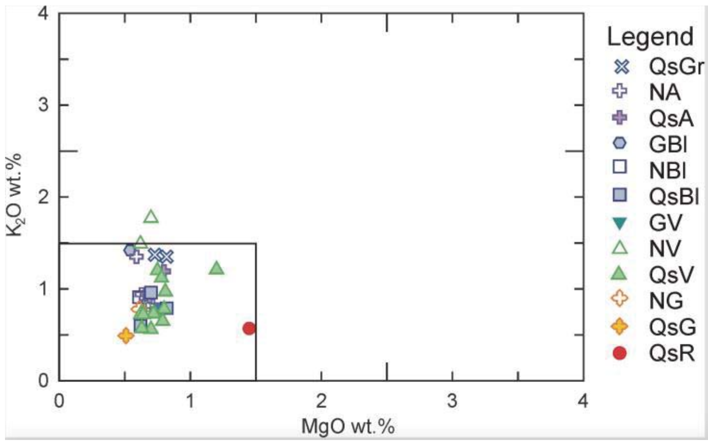

Fig. 2. Binary diagram MgO vs K_2O. The square area encloses the composition of natron glass.

Previous studies demonstrated that in the Roman age, the primary glasses were produced in a limited number of glass making centres and successively the slab of glass were distributed to the secondary fabrication workshops to be coloured, opacified and re-worked [16-18].

As concerns the chemical composition, the glass of the period of interest (I –III century AD) were divided in: Roman-Sb, Roman-Mn, Roman mixed Sb-Mn, according to the different contents of Sb and Mn used as decolourants [17, 19].

Comparing our results with the bibliographic data, the majority of the samples studied here, despite the great geochemical variability, shows similarity with the Sb/Mn Roman glass. Only few samples match the Mn-glass. Instead, the yellow and red tesserae display different compositions which is not possible to establish whether due to the base glass or to the colouring and opacifying techniques.

As for the variable contents of lead in the tesserae, samples can be divided into two subgroups: high lead (HPb) showing PbO concentrations higher than 1 wt.% and low lead (LPb) with a PbO content lower than 1 wt.%. The HPb group includes the red (QsR1) yellow (QsG1 and NG1) and some green tesserae. Among them, the red and yellow samples contain higher concentrations of PbO (QsR1 - PbO = 9.22 wt.%; QsG1 –

PbO = 17.49 wt.%; NG1 – PbO=7.68 wt.%).

A great number of samples show high Sb contents. The high concentrations in Pb and Sb are related to the presence of lead antimonate $Pb_2Sb_2O_7$ micro crystals that are homogenously distributed in the glass. They are clearly distinguishable in the Si-Pb-Sb maps of the NG1 tessera (Fig. 3).

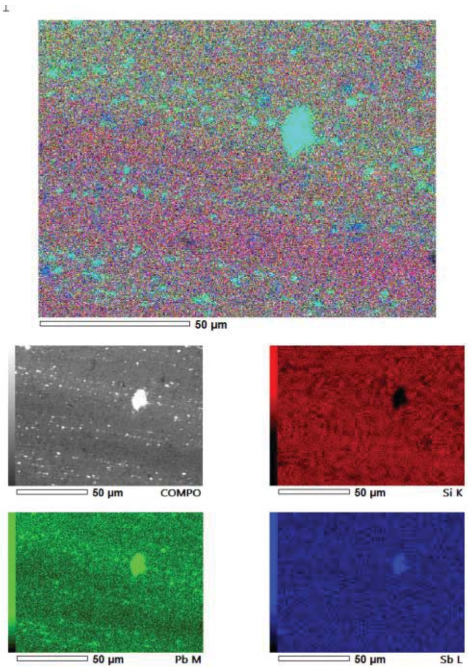

Fig. 3 WDS distribution mapping in RGB model of Si, Pb, Sb in the N-G1 yellow tessera from the Nereid mosaic. The distribution of these elements highlights the presence of lead antimonate $Pb_2Sb_2O_7$ micro crystals.

V. CONCLUSIONS

The archaeological area of S. Aloe quarter in Vibo Valentia includes three precious polychrome mosaics. The chemical study of 32 glass tesserae collected from all the three floors suggests the use of natron as a flux according to the Roman tradition. All glass tesserae are coloured and opacified.

In particular, the colouring agents identified during the analysis are cobalt and copper, introduced in the blue, light blue and green samples as bronze scrap.

The colour of the red (QsR1) is due to the use of cuprite. Opacifiers are all antimony-based with different natures according to the chemical composition and colours.

The microcrystals of Pb-Sn-antimonates have been detected in the yellow tesserae testifying that they are responsible for QsG1's typical colour.

These new data increase the information about the S. Aloe quarter and more generally on the Calabrian archaeological context.

REFERENCES

[1] F.Licenziati, T.Calligaro, "Study of mosaic glass tesserae from Delos, Greece using a combination of portable µ-Raman and X-ray fluorescence spectrometry", J. Archaeol. Sci. Rep., vol.7, 2016, pp. 640–648.

[2] L.Angelini, B.Gratuze, G.Artioli, "Glasses through history", EMU Notes in Mineralogy, vol.20(3), 2019, pp. 87-150.

[3] B.Wagner, A.Novak, E.Bulska, J.Kunicki-Goldfinger, O.Schalm, K.Janssens, "Complementary analysis of historical glass by scanning electron microscopy with energy dispersive X-ray spectroscopy and laser ablation inductively coupled plasma mass spectrometry", Microchim. Acta, vol. 162, 2008, pp. 415-424.

[4] J.Henderson, "Tradition and experiment in 1st millennium AD glass production: the emergence of Early Islamic glass technology in late antiquity", Acc. Chem. Res., vol.35, 2002, pp. 594–602.

[5] C.Boschetti, J.Henderson, J.Evans, C.Leonelli, "Mosaic tesserae from Italy and the production of Mediterranean coloured glass (4rd century BCE–4th century CE). Part I: Chemical composition and technology", J. Archaeol. Sci. Rep., vol.7, 2016, pp. 303–311.

[6] E.Arslan "Vibo Valentia" in: "Enciclopedia dell'Arte Antica II Supplemento", Treccani, Roma, Italia, 1997, vol. VII, pp.1162.

[7] F.Sogliani, "Repertorio delle fonti scritte per la ricostruzione della vicenda insediativa di Vibo Valentia tra Tarda Antichità e medioevo, Sulle tracce della Storia. Studi in onore di Vincenzo Nusdeo nel decennale della scomparsa", M.D'Andrea Ed., ADHOC: Vibo Valentia, Italy, 2012, pp. 271–335.

[8] L.Faedo, "Aspetti della cultura figurativa in età romana", in: "Storia della Calabria antica. 2. Età

italica e romana", S.Settis Ed., Gangemi, Roma, Italia, 1994, pp. 601-605.

[9] D.Palomba, "Vibo Valentia. B. Storia della ricerca archeologica", in: "BTCGI, XXI, Pisa-Roma-Napoli", 2012, pp. 889-900.

[10] M.T.Iannelli, P.G.Guzzo, G.Gaglianese, B.Minniti, A.M.Rotella, P.Vivacqua, "Hipponion tra la seconda metà del IV e la fine del III sec. a.C.: ricostruzione topografica alla luce di nuove scoperte", in: "Enotri e Brettii in Magna Grecia Modi e forme di interazione culturale", G.De Sensi Sestito, S.Mancuso Eds., Rubettino, Soveria Mannelli (CZ), Italy, 2017, vol. II (2), pp. 397-510.

[11] A.M.Rotella, "L'indagine archeologica nel quartiere romano di S. Aloe", in: "Hipponion Vibo Valentia Monsleonis. I volti della città", M.T.Iannelli Ed., Laruffa, Reggio Calabria, Italy, 2014, pp. 117-134.

[12] E.Fiorenza, N.Rovella, M.D'Andrea, M.Musella, F.Sudano, A.Taliano Grasso, D.Barca, "Vitreous Tesserae from the Four Seasons Mosaic of the S. Aloe Quarter in Vibo Valentia–Calabria, Italy: A Chemical Characterization", Minerals, vol. 10, 2020, pp.658-676.

[13] D.Barca, E.Basso, D.Bersani, G.Galli, C.Invernizzi, M.F.La Russa, P.P.Lottici, M.Malagodi, S.A.Ruffolo, "Vitreous tesserae from the calidarium mosaics of the Villa dei Quintili, Rome. Chemical composition and production technology", Microchem. Jour., vol.124, 2016, pp. 726–735.

[14] D.Barca, G.Lucarini, F.Fedele, "Provenance of obsidian artefacts from the WāDī ATH-THAYYILAH 3 Neolithic site (eastern Yemen plateau) by LA-ICP-MS", Archaeometry, vol.54 (4), 2012, pp. 603–622.

[15] D.Barca, E.Fiorenza, M.D'Andrea, E.Le Pera, M.Musella, F.Sudano, A.Taliano Grasso, "Chemical and Petrographic Characterization of Stone and Glass Tesserae in the Nereid and Geometric Mosaics from the S. Aloe Quarter in Vibo Valentia–Calabria, Italy", Minerals, vol.9(12), 2019, pp.729-748.

[16] I.C.Freestone, "The Provenance of Ancient Glass through Compositional Analysis", Mater. Res. Soc. Symp. Proc., 2005, 852, OO8.1.1-OO8.1.14.

[17] S.Maltoni, A.Silvestri, "A mosaic of colors: investigating production technologies of Roman Glass Tesserae from north eastern Italy", Minerals, vol.8, 2018, pp. 255-286.

[18] D.Barca, F.C.Papparella, "Chemical characterization of vitreous finds from Cosenza cathedral (Calabria – Italy) by the combined use of analytical techniques", Open Archaeol., vol.6, 2020, pp. 63-85.

[19] N.Schibille, A.Sterrett-Krause, I.C.Freestone, "Glass groups, glass supply and recycling in late Roman Carthage", Archaeol. Anthrop. Sci., vol.9(6), 2017, pp.1223–1241.