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ORIGINAL PAPER

Villa del Casale (Piazza Armerina, Sicily): stone and glass tesserae in the baths floor mosaics

Marco Verità[1] · **Lorenzo Lazzarini**[1] · **Elena Tesser**[1] · **Fabrizio Antonelli**[1]

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Abstract The Villa del Casale at Piazza Armerina (Sicily) is world famous for its beautiful and extensive mosaic floors dating to the fourth-century A.D. Before a recent restoration of the Villa, it was possible to take a representative sampling of coloured glass and stone tesserae from the baths' floors that were examined by chemical and minero-petrographic analysis, respectively, with the goal of determining the production-centre/s of the glass, and the provenance of the stones. The laboratory techniques used were SEM-EDS on polished sections, powder XRD and polarised OM on thin sections, extended to reference stones found in a Villa's ancient storehouse. The obtained results had shown that the white and pinkish tesserae were exclusively made of local (from the Caltanissetta province) limestones, while the black ones were cut from an obsidian, likely of a Liparote origin; the turquoise and gold-leaf tesserae were exclusively made of glass manufactured according to the Roman and Byzantine tradition while both stone and glass were used for the dark and yellow hues (*giallo antico* was identified among stone yellow hues). As for the glass tesserae, the results of the chemical analysis of a few samples (mostly erratic) have allowed to date them to the fifth- to sixth-century and to the eighth- to ninth-century A.D. and thus to testify to a hitherto unknown ancient restoration of the Villa mosaics.

Keywords Piazza Armerina · Villa del Casale · Mosaics · Stone and glass tesserae · Archaeometry

Introduction

The Villa del Casale, near Piazza Armerina in south-central Sicily (Italy), is a fourth-century AD Roman villa included in the World Heritage list by UNESCO. The villa contains one of the most important known examples of Late Roman mosaic floors, in their complex a masterpiece made by North-African craftsmen (Di Vita, 1972–73). The archaeological excavations of the villa carried out in different periods starting in 1935–39 and afterwards in the 1950s (Gentili, 1959), 1980s (De Miro 1984), 1990s and more recently at the beginning of this century (Pensabene 2010). The most important discoveries were related to the beautiful floor mosaics which were found in an excellent state of preservation. Stretching over about 3500 square meters, these mosaics reflect themes common to the era (Fig. 1), with scenes of the hunt, capture and transport of animals, circus games, athletic competitions, Egyptian landscape scenes of rocks and forests, Labours of Hercules, etc. (Carandini et al. 1982; Pensabene 2010), now famous worldwide (Wilson 1993).

Other mosaics should have been present also on the walls, for example in the frigidarium of the baths, of which an apse decorated with waves has survived: they were probably replaced later by mural paintings and/or marble slabs.

The villa has undergone various interventions over the centuries. Since the beginning of the fifth century and throughout the following century, the mosaics were restored many times, namely in the "Grande Caccia" (big hunt) ambulacra, in the eastern wing of the peristile and in the baths. (Pensabene 2010). Life in the villa continued until the eighth century when a progressive abandonment began. Life was resumed in a small portion of the villa under the Arab occupation of Sicily in the tenth century followed by the Normand conquest

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✉ Fabrizio Antonelli
fabrizio.antonelli@iuav.it

[1] LAMA - Laboratorio di Analisi dei Materiali Antichi, University Iuav of Venice, San Polo 2468/B, 30125 Venice, Italy

Fig. 1 Details of the mosaic floors from which some of the analysed tesserae were sampled. **a** *Sala delle Unzioni* (unction room), white and pinkish tesserae. **b** *Sala delle Terme* (bathroom), green and blue tesserae perfectly similar to those sampled in the storeroom. **c**–**d** *Sala delle Terme*, yellow tessera (*giallo antico*) similar to that sampled in the storeroom

in the 80s of the eleventh century (Pensabene and Sfameni 2006). The original mosaics were partially damaged after the abandonment; the marbles were spoliated and much of the structures were covered by earth (Pensabene 2010). In the twelfth century, the Arab population of the island centre was much reduced after the violent revolt of the Lombard barons in 1160–1161; further destruction was produced by the earthquake of 1169. The Villa del Casale remained partly inhabited after that date and was definitely abandoned toward the end of the twelfth and beginning of the thirteenth centuries, when a new village, now corresponding to the town of Piazza Armerina, was founded at the top of a hill not far away.

The mosaics were restored soon after the Second World War (Bernabò Brea 1947), and then, several other times in the last 50 years, the most important intervention having been performed in the 1960s and in the first decade of this century. These interventions lead to the restoration of the mosaic floors; the plexiglass covering of the villa was replaced with a more traditional one imitating the coverings of ancient Roman houses and to a wide study campaign.

In spite of the high importance of the Villa, only the building techniques (Lugli 1963) and the marbles (Lazzarini 2009) have been studied in detail. The materials used for the mosaics (glass and stones), instead, have been the object of rare and limited laboratory analyses. A first macroscopic study by using the Mansell Colour Charts was made by the archaeologists that excavated the villa in the second half of the last century (Carandini et al. 1982). Analyses of glass mosaic tesserae were performed by Croveri et al. (2010) with the aim to investigate their chemical composition and state of preservation and to bring a contribution to the conservation of the Roman Villa. Later, other authors (Di Bella et al. 2014) extended the analyses to a large set of glass tesserae to investigate the chemical composition of glass, the nature of colourants and opacifying agents. In both studies, the chemical quantitative analysis identified mainly natron type glass compositions, except for a pale blue transparent glass tessera (probably support of a gold leaf tessera; see below) and one red opaque glass tessera made with a soda plant ash glass studied by Croveri et al. (2010). An analytical investigation including both stone and glass tesserae was made by the present authors upon commission of the “Centro Regionale per la Progettazione e il Restauro della Regione Sicilia” with the goal of characterising their constituent materials and determine their state of preservation. A preliminary report of the obtained results was published in (Lazzarini et al. 2004). Finally, few analyses of tesserae from other Roman mosaics in Sicily were also published (Sabatino 2007; Triscari et al. 2007).

The present study is aimed to implement the number of examined tesserae in order to reach a more representative selection of their various typologies (colour, macroscopic aspect, etc). Furthermore, it is aimed to verify whether the laboratory characterisation of the stone and glass tesserae can improve the poor information currently available about the mosaic decorations set up in the Villa from its foundation to its abandonment. The materials were characterised both minero-petrographically and chemically. As for the stone tesserae, the main aim was to determine the petrographic nature as well as the possible geographical-geological origin of the rocks used; the glass tesserae were analysed to determine their glass composition with the goal of throwing light on their production technology and, if possible, distinguishing original tesserae from those used in restoration interventions. These data may help in the reconstruction of the composite historical life of these important mosaics.

Material and methods

Direct sampling from the tesserae of the floor has been limited to a minimum; more samples were obtained from the storerooms of the villa: they were original tesserae erratically found during the excavation of the baths and nearby rooms and not reused during restoration. The general sampling criteria were oriented by the most complete possible representation of the various colours and textures of stones and glass tesserae. Five samples of excavated coloured rocks that according to a local restorer (Mr Germanà) were probably used for the preparation of tesserae (hereafter called "reference coloured stones") were also examined (Fig. 2) and compared with in situ tesserae.

All the stone tesserae and samples were labelled according to the their provenance as follows: PA-U, mosaic of the *Sala delle Unzioni* (Unctions room); PA-P, *palestra*; PA-R, loose tesserae stored in the storerooms; PA-RR, samples of reference coloured rocks (Table 1).

Fig. 2 Polished fragments of the excavated reference coloured stones. On the left PA-RR3–white stone, PA-RR2–pinkish stone, PA-RR1–red stone; on the right, from top to bottom: PA-RR5–black stone, PA-RR4–green stone

The samples of the stone tesserae and five samples of the reference coloured stones were prepared in thin sections to be studied under the polarising microscope (Leitz DM RXP). When necessary, a portion of the same samples was powdered and analysed by X-Ray Diffraction (Panalytical Empyrean diffractometer, radiation CuKα/Ni at 40 kV and 40 mA) to detect the main crystalline phases and the possible presence of clay minerals and dolomite not easily identifiable microscopically.

Sixteen glass tesserae were selected for analysis. They include 11 opaque or translucent coloured tesserae and five gold leaf tesserae. Gold leaf tesserae are made of a beaten thin gold leaf (0.3–0.8 μm thick) hot sandwiched between two glass layers. The glass embedded in the mortar (the support) is 5 to 10 mm thick, while a thin sheet of blown glass (the *cartellina*, usually 0.3 to 0.7 mm thick) protects the fragile metal leaf and adds to its brilliance. The analysed gold leaf tesserae came exclusively from the storerooms. They consist only of the support: the *cartellina* has been lost and only minute remains of the gold leaf were identified on their surface by SEM-EDS analysis.

After a preliminary observation by an optical microscope (Leika MZ12), glass fragments for analysis were dry cut from the tesserae and embedded in cross-section in acrylic resin in a teflon mould. The discs containing up to six fragments were ground and polished with diamond pastes down to 1-μm grain size. The polished sections were observed by optical microscopy in reflected light (Leika MZ12) and by scanning electron microscopy (Philips XL 30) in backscattered mode (where the grey levels indicated areas with different chemical compositions) after carbon coating.

Analyses were performed in two laboratories. The quantitative X-ray microanalysis of the glass tesserae was undertaken at the Stazione Sperimentale del Vetro (Murano, Venice), a microprobe (Cameca SX-50) equipped with three wavelength-dispersive X-ray spectrometers (PET, LiF and TAP crystals) was used. Twenty elements were quantified: X-ray Kα-lines were used except for Pb (Mα-line), Sb, As and Sn (Lα-lines). Operating conditions were accelerating potential 15 kV, beam current 20 nA (major and minor components) or 100 nA (trace elements). A 40 μm × 50 μm scanning electron beam and limited counting time (10 s for major and minor elements, 20 to 30 s for trace elements) were employed to minimise alkali drift during the irradiation. The net X-ray intensities were quantified by means of a PAP correction program supplied by Cameca. Reference glasses of certified composition (Corning B, C and D and NBS 620) were analysed under the same experimental conditions as the glass tesserae to verify the accuracy of

Table 1 Description of the 29 stone and glass tesserae and the five coloured reference rocks selected for analysis

Location of the sampled tesserae		Material type	Autoptic colour	Munsell colour
Anointings room	PA-U1	Stone	White	2.5Y 8/1 white
	PA-U4	Stone	Yellowish-reddish	10YR 6/4 light yellowish brown
	PA-U5	Stone	Yellowish-reddish	2.5YR 7/2 pale red
	PA-U6	Stone	Dark red	2.5YR 3/3 dusky red
	PA-U7	Stone	Pink	10R 8/2 pinkish white
	PA-U8	Stone	Black	Chart 1 for Gley 4/1 dark grey
	PA-U9	Stone	Green	Chart 1 for Gley 4/1 dark greenish grey
	PA-U10	Stone	Red	10R 3/6 dark red
Gymnasium	PA-P11	Stone	Red	10R 4/6 red
	PA-P13	Stone	Light green	Chart 1 for Gley 5/1 greenish grey
	PA-P14	Stone	Pink	10R 8/3 pink
	PA-P15	Stone	Dark yellow	10YR 7/6 yellow
	PA-P17	Glass	Blue	Not determined
	PA-P18	Glass	Blue	Not determined
	PA-P19	Glass/obsidian	Black	Not determined
Erratic tesserae from the storeroom	PA-R1	Glass	Turquoise	Not determined
	PA-R2	Glass	Blue	Not determined
	PA-R3	Glass	Red brown	Not determined
	PA-R4	Glass	Gold leaf	Not determined
	PA-R9	Glass	Blue dark	Not determined
	PA-R10	Glass	Turquoise dark	Not determined
	PA-R11	Glass	Blue-grey	Not determined
	PA-R12	Glass	Grey	Not determined
	PA-R14	Glass	Yellow green	Not determined
	PA-R15	Glass	Green yellow	Not determined
	PA-R16	Glass	Gold leaf	Not determined
	PA-R17	Glass	Gold leaf	Not determined
	PA-R18	Glass	Gold leaf	Not determined
	PA-R19	Glass	Gold leaf	Not determined
Coloured reference rocks	PA-RR1	Stone	Red	10R 4/4 weak red
	PA-RR2	Stone	Pink	10R 4/4 to 5/4 weak red
	PA-RR3	Stone	White	2.5Y 8/2 to 8/1 pale yellow to white
	PA-RR4	Stone	Green	Chart 1 for Gley 5/10Y greenish grey
	PA-RR5	Stone	Black	Chart 1 for Gley 2.5/N greenish black

the method. The accuracy for SiO_2, Na_2O and CaO is below 1% and for the minor oxides and traces is below 5%. Limits of detection in the range of 0.02–0.05% for most of the oxides were calculated. Further details of the analytical method are reported in (Verità et al. 1994). Three samples were also analysed by an X-ray fluorescence spectrometer (Tornado, Bruker) to determine trace elements. Under the selected experimental conditions (120 s measuring time), an accuracy of the order of 15–20% was estimated for strontium, zirconium and barium. The polished sections were investigated at the LAMA laboratory with a scanning electron microscope Philips XL30. Identification of the opacifiers, pigment particles and the composition of the gold leaves were performed by energy-dispersive X-ray microanalysis (EDAX Ametek).

Results and discussion

The description of the examined 29 stone and glass tesserae and five coloured reference rocks is reported in Table 1.

Stone tesserae and reference coloured stones

Stone tesserae

The results of the microscopic study of the thin sections of the stone tesserae distinguished on the basis of the groundmass colour are reported below and in Fig. 3.

White tesserae: the only one sample examined (PA-U1) resulted to be made of a biomicrite-biomicrosparite (Folk 1959), namely a wackestone according to the Dunham's classification (Dunham 1962), containing bioclasts of rudists, echinids and calcareous algae (Fig. 3).

Black tesserae (PA-U8, PA-P19): they differ both in macroscopic aspect and composition. The first one is formed by an azoic bituminous-carbonaceous microsparite (Folk 1959; Fig. 3), a packstone according to Dunham's classification (Dunham 1962), with a small (< 5%) detritic fraction made of quartz and limonitic iron oxides: its black colour is due to bituminous organic matter and fine carbonaceous particles both dispersed in the mass or concentrated in thin levels.

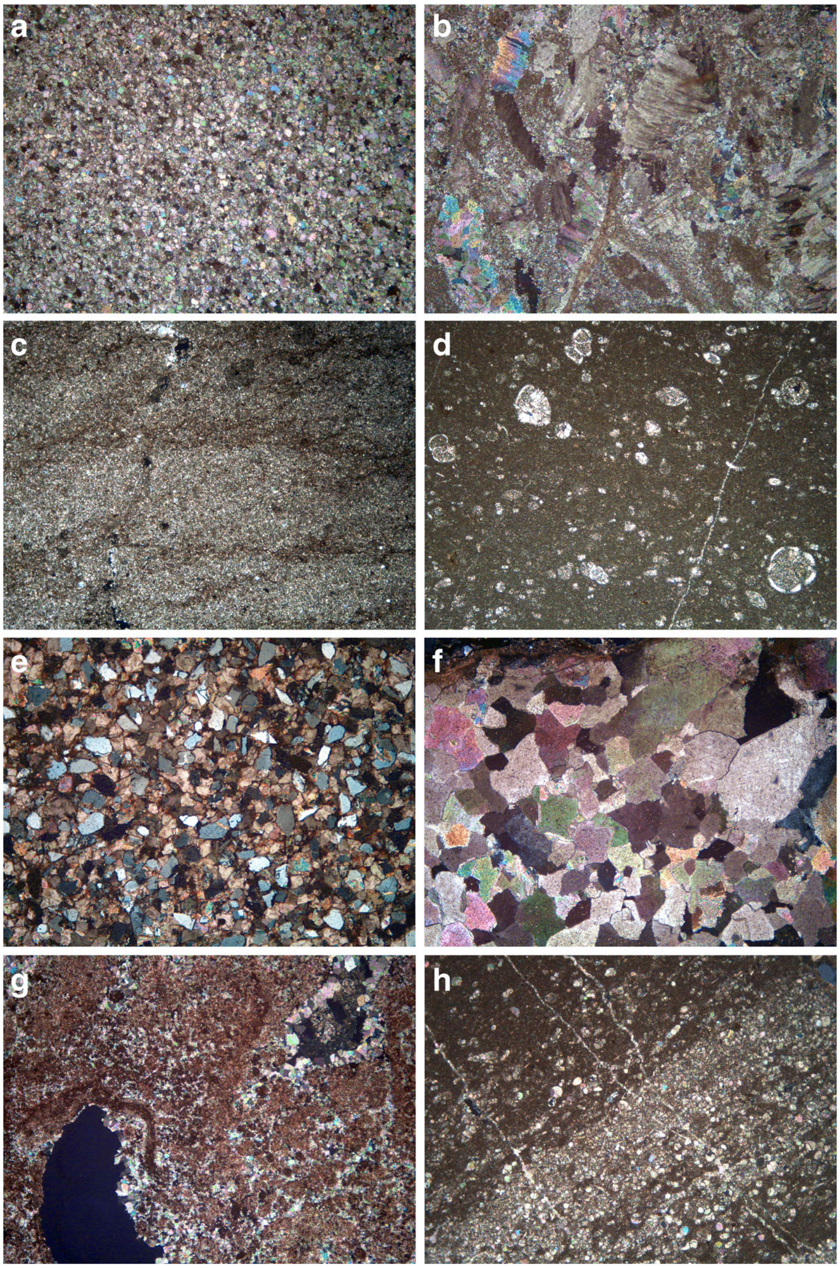

Fig. 3 Photomicrographs of the thin sections obtained from the different typologies of stone tesserae; crossed polarised light, long side of the pictures is 2.35 mm. **a**. Sample PAP15 (dark yellow tessera, giallo antico): microsparite with a mosaic fabric formed by equigranular idiotopic calcite crystals with dispersed small limonite particles. **b** Sample PA-U1 (white tesera): biomicrite-biosparite containing bioclasts of rudists, echinids and calcareous algae. **c** Sample PA-U8 (black tessera) azoic bituminouscarbonaceous microsparite. **d** Sample PAU4 (yellowish-reddish tessera): biomicrites containing very abundant planctonic microforaminifera, mainly Globigerinae Sp. **e** Sample PAU6 (pinkish tessera), subarcose/ lithic subarenite quartz-rich in a carbonatic-ochraceous (hematitic) cement. **f** Sample PA-U7 (pinkish tessera), dolosparite with an ipidiotopic-mosaic fabric formed by equigranular-euhedral/ subhedral dolomite crystals. **g** Sample PA-U10 (red tessera), travertine-type limestone with a deep red colour due to the presence of hematite in the mass or concentrated in small aggregates. **h** Sample PA-U9 (green tessera), ritmite composed of alternating poorly washed biosparite and biomicrite thin layers

The comparison with the petrographic data bank published by (Brilli et al. 2010) on “neri antichi” limestones from Tunisia (Djebel Oust, Djebel Azeiz, Ain al Ksir and Thala) and the Greek island of Chios allows both these origins to be excluded for PA-U8.

Tessera PA-P19 shows a vitreous aspect and a chemical composition with high alumina (Al_2O_3 13.6%) and relatively low concentrations of alkalis (Na_2O 4.2%; K_2O 5.4%; CaO 0.75%; MgO 0.04%), that is typical of natural glasses of volcanic genesis like obsidian.

The dark yellow tessera PA-P15 is a grainstone (Dunham 1962) with a mosaic fabric formed by equigranular idiotopic calcite crystals with dispersed small limonite particles and aggregates (Fig. 3). The macro- and microscopic aspects coincide very well with those of *giallo antico* (the *marmor numidicum* of the Romans), a highly diagenetised limestone exploited very close to the ancient town of *Simitthus*, the present Chemtou in Tunisia (Rakob 1993; Antonelli et al. 2010).

Yellowish-reddish tesserae PA-U4, PA-U5 are biomicrites containing very abundant planctonic microforaminifera (Fig. 3d), mainly *Globigerinae Sp*., abundant calcareous radiolars and some sponge spiculae (PA-U4). Globigerinae, associated with Globotruncane, Calpionellae and Ostracodi are present in the section of PA-U5: both tesserae were likely obtained from polychrome (whitish-yellowish-reddish) Cretaceous carbonatic formations of the “Scaglia” type .

Pinkish tesserae PA-U6 and PA-U7 are composed respectively of a sandstone and a dolosparite. PA-U6 may be classified as a subarcose/lithic subarenite (Folk 1974) and is mainly composed of quartz (angular, poly- and micro-crystalline/chert) and by traces of K-feldspar (microcline and perthite), muscovite and rock fragments (muscovite-quartzites), in a carbonatic-ochraceous cement coloured brown-red by hematite (Fig. 3e). The dolosparite (Folk 1959) or grainstone (Dunham 1962) shows a ipidiotopic-mosaic fabric formed by equigranular-euhedral/subhedral dolomite (0.2–1 mm) crystals (Fig. 3f); also the pinkish tessera PA-P14 belongs to the same lithotype.

The red tesserae PA-U10 and PA-P11 are made of a limestone with a porous texture, quite similar to that of travertines (Fig. 3g) with a deep red colour due to hematite finely dispersed in the mass, or concentrated in small aggregates. This rock may be classified as a grainstone (Dunham 1962) and is characterised by the presence of rare clasts of bivalves, small amounts of detritic, angular quartz and abundant *fenestrae,* often filled by sparite.

The green tesserae PA-U9 and PA-P13 are quite similar: the first one has the typical features of a ritmite (Fig. 3h) composed of thin layers of a poorly washed biosparite with allochems > 40% (Folk 1959) or packestone, (Dunham 1962) alternating with layers of a biomicrite (wackestone) with 10% allochems, in both cases formed by pelagic foraminifera (Globigerinae are the prevailing species), with rare detritic subangular quartz and iron oxide stains. The second tessera is a typical “wackestone”. The greenish colour is due to chlorite, detected by XRD analyses in both samples, even if not clearly detectable on a microscopic basis; a certain contribution may also be due to ferrous iron oxides.

Reference coloured stones

The results of the parallel minero-petrographic study of the stones excavated in the Villa (Fig. 2) may be summarised as follows:

PA-RR3 – *White stone*: this stone is a planctonic biomicrite (wackestone; Fig. 4a–b) with microforaminifera (Globigerinaes, Globigerinidae and Globotruncanae), sponge spiculae (mostly siliceous, but also with calcitic imprints) and rare carbonatic radiolars. Also present are small angular/subangular detritic quartz crystals. This stone may be strictly associated to the lithotype of the PA-U4 tessera, less well to PA-U5 since the Calpionellae are missing here.

PA-RR5 – *Black stone*: this stone is a fine-grained black lava showing an intergranular fabric, with microliths and phenocrysts of plagioclase, altered pyroxene, resorbed olivine (often in skeletal or swallow-tail crystals and with evident alteration into serpentine) and magnetite in a partially vitreous groundmass (Fig. 4c). On the basis of the microscopic study, it may be classified as a basalt/basaltic andesite. This lava has not been yet identified among the black tesserae analysed so far.

PA-RR1 – *Red stone*: this stone may be classified as a biomicrite wackestone (Fig. 4d); the reddish colour is due to hematite spread in the mass, or concentrated in styloliths; it contains abundant clasts of echinids and crinoids and small amounts of ostracods, corals, calcareous algae and siliceous radiolars. Detritic quartz and rare glauconite globules are present as accessory minerals. This lithotype has not been identified so far in the tesserae of the examined mosaics.

PA-RR2 – *Pinkish stone*: it is a red sandstone with an ochraceous-carbonatic cement that may be classified as a mature, well sorted subarkose (Fig. 4e) and is fully comparable to the lithotype of the PA-U6 tessera, although containing more hematite and bioclasts, and a few micro-perthitic feldspar, plagioclase, flint and fragments of granitoid and metamorphic rocks.

PA-RR4 – *Green stone*: also a biomicrite-biomicrosparite wackestone; (Fig. 4f); with about 15–20% allochems composed of abundant quartz, bioclasts of Globigerinidae, plates of echinoids, sponge spiculae and dispersed small masses of Fe-oxides/hydroxides and rare dolomite rhombohedra. These

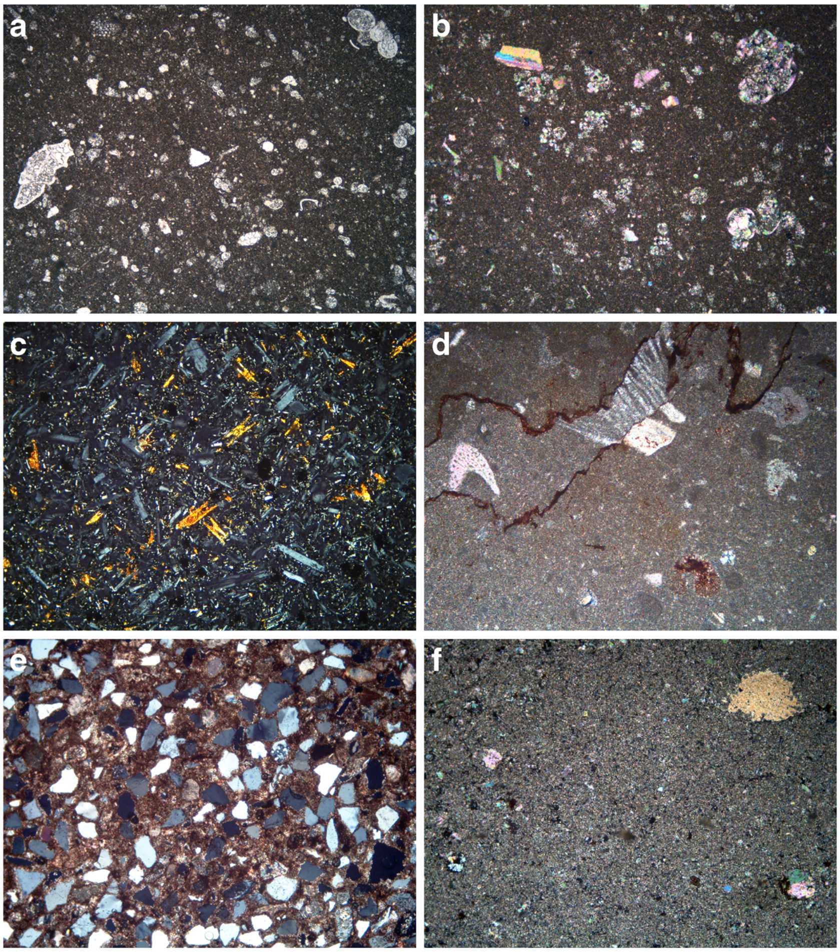

Fig. 4 Photomicrographs of the thin sections obtained from the reference coloured stones. **a**–**b** Sample PA-RR3 (white stone), planctonic biomicrite very rich in microforaminifera. **c** Sample PA-RR5 (black stone), fine-grained basalt/basaltic andesite with intergranular groundmass, microliths and phenocrysts of plagioclase, altered pyroxene and resorbed olivine. **d** Sample PA-RR1 (red stone), biomicrite with hematite spread in the mass or concentrated in styloliths. **e** Sample PARR2 (pinkish stone), mature and well sorted subarkose with an ochraceous-carbonatic cement; it is fully comparable to the lithotype of tessera PA-U6. **f** Sample PA-RR4 (green stone), biomicrite-biomicrosparite very similar to sample PA-P13. Crossed polarised light, except for **b**, which is plane polarised light, long side of the pictures is 2.35 mm

petrographic characteristics are very similar to those of PA-P13.

Glass tesserae

The quantitative chemical composition of the analysed glass tesserae is reported in wt% of the oxides in Table 2. The composition of coloured tesserae is an average one, including the analysis of the glassy phase and the opacifier-pigment particles. The composition of the base glass of the tesserae will be discussed first; colourants and opacifiers will follow.

Base glass

The base glass compositions were obtained by subtracting to the composition of the tesserae the content of colourants (cobalt, copper, manganese), opacifiers and pigments (antimony, tin, lead, etc.) and related elements, and then normalising the remaining oxides to 100%.

The base glass of all of the tesserae is of the soda-lime-silica type. Two compositional groups can be distinguished taking into account the potassium, magnesium (Fig. 5) and phosphorous contents. Potassium and magnesium are each below 1.5%, and phosphorus is below 0.2% for most of the tesserae, as would be expected for natron type glass produced in the Roman period (samples indicated with N in Table 2). This group (triangles in Fig. 5) includes the coloured tesserae (except the red tessera PA-R3) and three gold leaf tesserae (PA-R17, PA-R18 and PA-R19). Two coloured tesserae PA-R1 and PA-R9 show an unusually high content of phosphorous discussed hereafter. The natron type glasses were made by melting a batch of silica-lime sand and natron, a mineral soda. According to this technology used until the eighth to ninth century, the batch of raw materials was melted in large tank furnaces in few primary glassmaking sites. Up today, primary glassmaking sites have been identified in the Levant and Egypt (Freestone 2005; Freestone 2015; Phelps et al. 2016). The raw transparent glass produced; there was transported in the form of chunks to secondary centres to be remelted, coloured, opacified and shaped into slabs from which the tesserae were cut.

The percentages of major and minor oxides in the samples of this group are fairly variable (for example, Na_2O 14.5–19.0%; CaO 4.3–9.1%), and indicate the use of primary glasses prepared with different recipes with lower (Na_2O 15–16.5%) or higher (Na_2O 18.5–20%) amounts of natron.

Table 2 Chemical composition of the analysed glass tesserae, expressed in wt% of the oxides

Tessera	Colour	Glass type	Opacifier	SiO_2	Al_2O_3	Na_2O	K_2O	CaO	MgO	SO_3	P_2O_5	Cl	TiO_2	Fe_2O_3	MnO	Sb_2O_3	CuO	PbO	SnO_2	ZnO	CoO	SrO^*	ZrO_2^*	BaO^*
PA-R2	Blue	N	Ca-antim.	66.4	2.37	14.5	0.59	8.16	0.55	0.39	0.17	0.75	0.05	0.85	0.10	2.80	1.35	0.80	0.06	0.03	0.08			
PA-P17	Blue	N	Ca-antim.	67.3	1.82	18.8	0.53	5.44	0.67	0.42	0.03	1.10	0.07	0.71	0.03	2.80	0.07	0.12	0.03	0.05	0.04			
PA-P18	Blue	N	Ca-antim.	66.5	2.46	15.5	0.53	8.17	0.65	0.46	0.14	0.80	0.05	0.90	0.37	3.20	0.16	nd	0.03	nd	0.06			
PA-R11	Blue-grey	N	Ca-antim.	66.5	2.58	14.8	0.62	8.00	0.66	0.35	0.18	0.65	0.06	0.70	0.75	4.00	0.05	nd	0.03	nd	0.03			
PA-R12	Grey	N	Ca-antim.	68.3	2.28	18.2	0.63	6.30	0.95	0.33	0.08	1.10	0.07	0.47	0.38	0.85	nd	0.05	nd	nd	nd			
PA-R10	Turquoise dark	N	Ca-antim.	66.0	2.00	18.8	0.53	4.30	0.59	0.37	0.03	1.15	0.10	0.67	0.03	0.75	4.20	0.09	0.28	0.12	nd			
PA-R1	Turquoise	N	Ca-phosph.	68.7	2.60	14.7	0.82	9.10	0.67	0.15	0.28	0.90	0.07	0.45	0.03	nd	1.20	0.14	0,14	nd	nd			
PA-R9	Blue dark	N	Ca-phosph.	64.3	2.45	19.0	0.67	8.24	1.10	0.45	0.35	0.80	0.15	1.25	1.00	nd	0.07	0.06	nd	nd	0.06			
PA-R14	Yellow green	N	Pb-stannate	63.7	2.72	14.7	0.84	7.82	0.71	0.10	0.17	0.85	0.07	0.40	0.03	nd	0.40	6.50	0.95	nd	nd			
PA-R15	Green yellow	N	Pb-stannate	59.0	2.22	17.3	0.59	7.00	1.05	0.25	0.07	1.10	0.12	0.95	1.00	nd	0.10	8.00	1.20	nd	nd			
PA-R3	Red brown	SP	Cu	63.3	2.50	15.5	1.32	7.22	1.50	0.30	0.45	0.88	0.13	2.15	0.55	0.50	1.15	2.15	0.30	0.05	nd			
PA-R17(s)	Yellow hue (gold)	N	r	70.0	2.40	16.6	0.60	7.60	0.45	0.20	0.10	1.10	0.08	0.50	0.30	0.02	nd	nd	nd	nd	nd			
PA-R19(s)	Yellow hue (gold)	N	tr	70.0	2.58	16.0	0.47	7.65	0.24	0.13	0.10	1.10	0.05	0.35	1.26	0.02	nd	nd	nd	nd	nd	0.055	0.014	0.020
PA-R18(s)	Colourless (gold)	N	tr	70.4	2.68	15.4	0.47	7.69	0.22	0.16	0.08	1.09	0.05	0.35	1.26	0.06	nd	nd	nd	nd	nd	0.072	0.013	0.025
PA-R16(s)	Blue green hue (gold)	SP	Tr	66.7	1.93	17.4	1.54	7.10	2.05	0.23	0.35	1.15	0.13	0.97	0.20	0.15	nd	nd	nd	nd	nd	0.070	0.018	0.019
PA-R4(s)	Blue green hue (gold)	SP	Tr	65.3	1.95	18.7	1.60	6.90	2.20	0.28	0.35	1.05	0.17	1.10	0.18	0.18	nd	nd	nd	nd	nd			

Glass type: *N* natron glass, SP soda ash glass. (*s*) Support of gold leaf tessera. Opacifier: *Ca-antim*. calcium antimonate, *Ca-phosph*. calcium phosphate, *Pb-stannate* lead stannate

nd not detected

*Oxides analysed by XRF in the tesserae PA-R16, PA-R18 and PA-R19

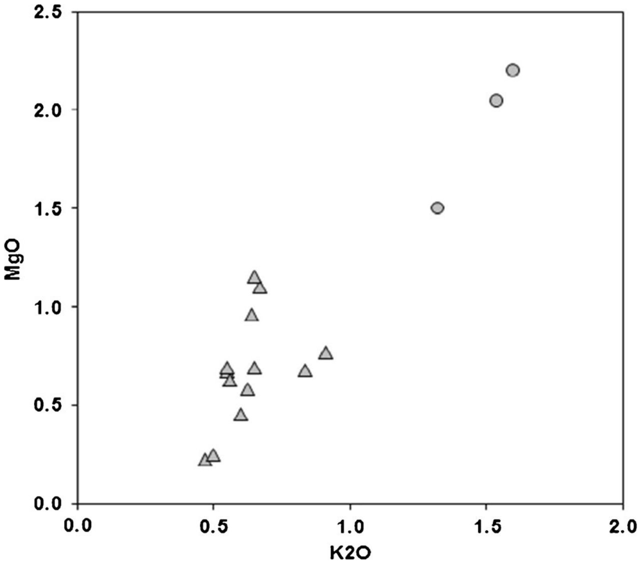

Fig. 5 Diagram of the magnesium and potassium contents of the natron type base glass samples (triangles) and of the intermediate between natron type and soda plant ash type base glass samples (circles)

The contents of aluminium (Al_2O_3 2–3%) and calcium (CaO 5–9%) indicate that the raw glass used for the production of the tesserae was of Levantine I type, produced in Palestine in the fourth to seventh centuries (Freestone 2005). It should be recalled that the type of primary glass used is not specific of individual workshops where mosaic tesserae were made. According to present knowledge, the colouring of the glass to prepare mosaic tesserae was made in secondary sites, which could get raw glass supplies from more than one primary site.

The second group (circles in Fig. 5) includes gold leaf tesserae (PA-R4 and PA-R16) and a red opaque one (PA-R3). The concentrations of potassium (K_2O 1.3–1.6%), magnesium (MgO 1.5–2.2%) and phosphorous (P_2O_5 0.35–0.45%) are higher than those of natron type glass and similar (but not equal) to those of glass obtained by melting a batch of silica sand and ashes of halophite plants growing in saline grounds (soda plant ash type glass). The barium content (BaO 0.02–0.03%) measured by XRF is similar to the barium content in the natron glass of sample PA-R19. The soda plant ash glass production is attested in the Mediterranean area since the eighth to ninth centuries. Natron type glass continued to be completely replaced by the new glass only in the twelfth to thirteenth centuries (Verità et al. 2002). The studies of Croveri et al. (2010) on tesserae from the Villa del Casale mosaics found two tesserae belonging to the soda plant ash type glass, while the analyses of Di Bella et al. (2014) refer exclusively to natron type glass tesserae.

In Table 2, the attribution of each tessera to one of these two groups is indicated as N (natron type) and SP (soda plant ash type), respectively. In group SP, the potassium content is lower than 2%, i.e the amount conventionally assumed as an indicator of the use of soda plant ash as a flux, being intermediate between natron type glass and soda plant ash type glass. Instead, the amount of magnesium and phosphorous fall within the limits of the soda plant ash glass type. Similar compositions were found in Byzantine mosaic tesserae in Italy (Torcello: Henderson and Roe 2006; Verità and Zecchin 2012; and Monreale: Verità and Rapisarda 2008) and Greece (Daphni and Hosios Loukas: Freestone et al. 1988, Brill 1999, Brill 2008, Arletti et al. 2010). This composition was found also in blown glass artefacts from Italy (Uboldi and Verità 2003) and Middle Eastern countries (Henderson et al. 2004). Some authors have interpreted this composition as a “mixed natron-soda plant ash composition” obtained by melting together natron glass and soda ash glass. This is possible, but other interpretations are also plausible (the use of a particular type of plant ash, for instance). It is important to recall that to the authors’ present knowledge, glass tesserae showing this intermediate composition were identified up today in Byzantine mosaics dated to the tenth to twelfth centuries. Nevertheless, recent analyses have found similar intermediate compositions in the late sixth- to seventh-century Byzantine glass weights (Schibille et al. 2016).

The presence of traces of antimony (Sb_2O_3 0.15–0.20%) in the gold leaf tesserae made with soda plant ash glass supports the hypothesis that this glass was melted by adding different kinds of cullet. In fact, it is generally accepted that the use of antimony as decolourant and opacifier of glass declined in the fourth- to fifth-century CE and it was utilised seldom after this date (Lahlil et al., 2008). Antimony was most likely introduced with natron type glass cullet opacified with Ca-antimonate, but the use of a antimony bearing raw material (e.g. roasted stibnite) cannot be completely excluded.

In any case, the gold leaf tesserae PA-R4 and PA-R16 do not belong to the original mosaic decoration but were inserted later in restoration interventions or in new mosaics not before the eighth to ninth centuries (see hereafter).

This is not the case of the red tessera PA-R3. Tesserae of this colour were already produced with plant ash or intermediate type glass in Roman time, when the other colours were exclusively made with natron type glass (Freestone and Stapleton, 2003). It is possible that the reducing conditions during melting that resulted from the use of vegetable ash ensured better success in obtaining this delicate colour, as discussed below (Verità and Santopadre 2015). Therefore, this tessera could belong to the original mosaic decoration.

Gold leaf tesserae

The production technology and chronology of gold tesserae can be evaluated by an analytical study of both the glass and the gold leaf of the tesserae (Neri and Verità 2013).

The glass of the tesserae PA-R17, R18 and R19 is natron type, while the glass of the tesserae PA-R4 and R16 is of soda plant ash type. It is known that in both the Eastern and Western areas, the long transition from a natron type to a soda ash type glass began in the eighth to ninth centuries and lasted until the end of the twelfth century. For this reason, it is possible to establish a period of production after the eighth to ninth centuries for the tesserae made with soda ash glass.

No complete gold tesserae were found, only the supports with a few fragments of gold leaf adhering. Because of the small size of the fragments, no accurate measure of the leaf composition was possible; anyway, in both samples, the analyses found that the metal leaf is made of a gold-silver alloy, with a significant silver content (Ag 5–6%; concentration expressed in wt% of the element). The use of circulating gold coins to obtain gold leaves for mosaic tesserae during Late Antique and Byzantine periods was recently attested (Neri and Verità 2013; Neri et al. 2016). It is known that the composition of circulating gold coins varied in time and sometimes also according to mints of coinage, and the match of gold coins and gold leaves compositions allows dating the tesserae according to the limits of the coins dating power. Gold coins with a silver content similar to the gold leaves of the tesserae PA-R17 and R18 circulated in the Roman empire mainly during the fifth to sixth centuries, and this dating can be suggested for the manufacture of the two tesserae.

Some transparent glass tesserae were analysed also in previous studies (Croveri et al. 2010; Di Bella et al. 2014) and considered and discussed as coloured tesserae, while they were probably the supports of gold leaf tesserae.

Opacifiers and colourants

The opaque or translucent aspect of coloured glass tesserae results from the presence of dispersed particles with a refractive index different from that of the glass. The type of opacifier used is reported in Table 2.

In most coloured tesserae, the average chemical composition identified significant amounts of antimony (Sb_2O_3 0.75–4.0%). SEM analyses revealed the presence of microcrystals below 2 μm in size, sometimes as aggregates of particles of some tenths of μm composed of calcium and antimony (Fig. 6a).

X-ray microanalyses revealed that these particles are composed of antimony (Sb_2O_3 65–75%) and calcium oxides (CaO 35–35%) i.e. the Ca-antimonate crystals used in Roman time and in some sites up to the early Middle Ages for the production of opaque mosaic tesserae (Neri and Verità 2012). The only two tesserae sampled from in situ mosaics (blue tesserae PA-P17 and P18 from the *palestra*) are of this type, i.e. natron glass opacified with calcium antimonate. Only Ca-antimonate was identified as an opacifier in coloured tesserae from Villa del Casale by other authors (Croveri et al. 2010; Di Bella et al. 2014).

A different technique was used for tesserae PA-R1 and PA-R9, whose opacity is due to a great amount of small bubbles and to white particles even large in size (Fig. 6b). The average chemical analysis of these tesserae identified a natron type base glass, yet with an unusually high phosphorous content (P_2O_5 0.27 and 0.35%, respectively); the phosphorous content of the natron type glass only exceptionally exceeds P_2O_5

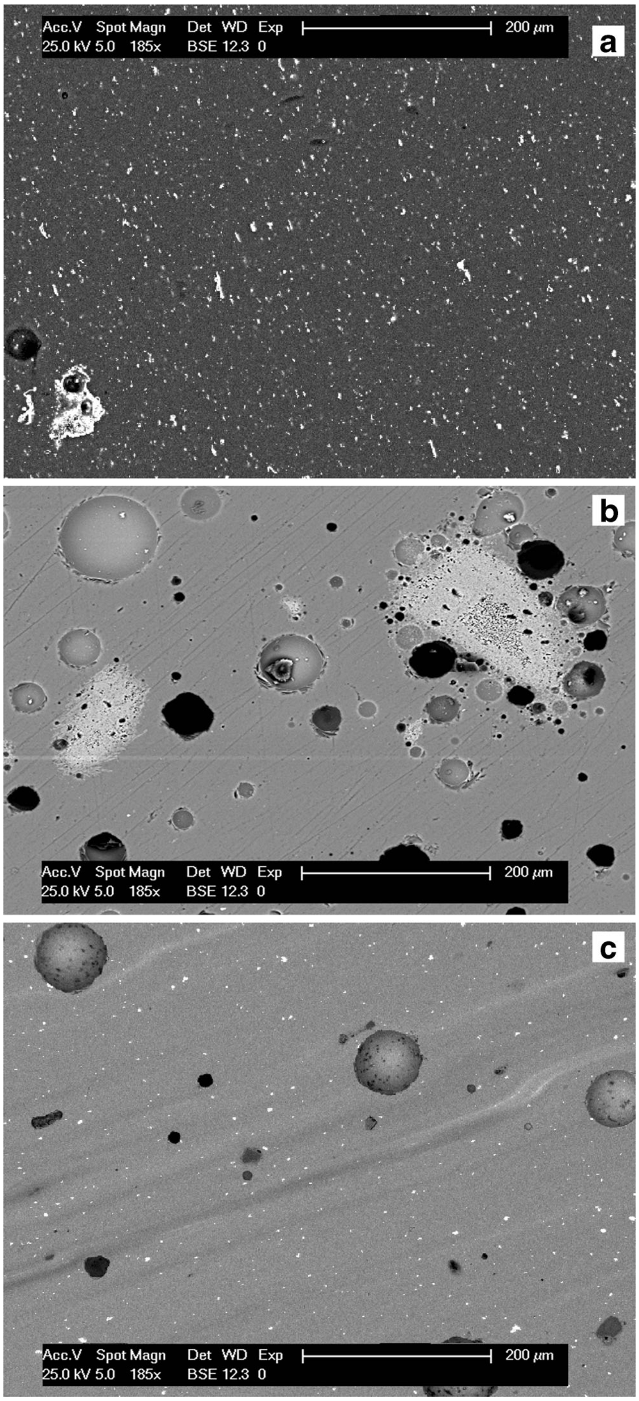

Fig. 6 SEM photomicrographs of the polished cross sections of **a** the blue tessera PA-R11 opacified with calcium antimonate particles (white dots) sometime aggregated in large particles (white in the left-low corner), **b** the blue tessera PA-R9 opacified by bubbles (circles) and bone ash particles (light grey), and **c** the yellow-green tessera PA-R14 opacified and coloured by small particles of lead stannate (white in the micrograph)

0.2%. The SEM-EDS investigation ascertained that the coarser white particles consist mainly of calcium phosphate (CaO 45–50%; P_2O_5 50–55%), whereas in smaller particles sodium (Na_2O up to 20%) is also present.

The chemical analysis and the porous structure of these particles confirm that they are bone ash fragments added to the melt just before shaping the glass slabs. The air trapped in the porosity and the reaction with the melt determine the characteristic small bubble release. The presence of sodium in the smaller particles results from the dissolution process of the particles in the glass melt involving formation of intermediate compounds (Verità 2010). A similar opacification technique (bubbles and particles of bone ash) was identified by the analyses of coloured tesserae, and mosaic cake remains excavated in a Byzantine basilica at Petra (Jordan), dated from the middle fifth to early eighth centuries (Mari and Rehren 2009) and in coloured tesserae from the fifth-century mosaics of the Neonian Baptistery in Ravenna, Italy (Verità 2010), in the sixth-century tesserae from Padua (Italy) (Silvestri et al. 2016) and in tesserae from early Islamic mosaics dated to the eighth century at Qusayr' Amra, Jordan (Verità et al. 2016). In all these cases, the composition of the glass is of natron type. These data allow to conclude that the opacification technique using bone ash was a Byzantine glassmaking technique in use between the fifth and the eighth centuries.

The colour and opacity of the red tessera PA-R3 result from micro-particles of metallic copper separated from the melt during cooling, probably following to the addition of iron (Fe_2O_3 2.15%) in reduced form (FeO) to facilitate the reduction of the dissolved copper and the precipitation of metallic microparticles. The base glass has an intermediate composition between natron type and plant ash type, with significant amounts of lead (PbO 2.15%) and tin (SnO_2 0.3%). These elements are probably related to copper (CuO 1.15%) added in the form of bronze scrap. The analyses detected also the presence of antimony (Sb_2O_3 0.5%), and SEM observation revealed the presence of rare particles of calcium antimonate, that may result from the introduction of white opaque glass in the melt (recycling of glass cullet). Similar observation were made by Di Bella et al. (2014), despite the natron type composition of the glass of the investigated red tesserae from the Villa del Casale mosaics.

In the yellow-green tessera PA-R14 and in the green-yellow PA-R15 colour derive from the dark green glass (copper and iron as colourants) and from yellow pigments. The different hues result from the different amounts of copper and iron dissolved in the glass phase. The pigments have essentially similar compositions in the two tesserae and are made of tin and lead oxides (SnO_2 40–45%; PbO 55–60%). Heterogeneity (alternate streaks of opaque and transparent glass and several bubbles) differs in the two tesserae: PA-R14 is rather homogeneous, yellow-green in colour, while R15 is green-yellow, very heterogeneous with intense yellow opaque streaks alternating with colourless transparent layers (Fig. 6c).

The heterogeneous aspect of these tesserae is characteristic of yellow glass obtained by adding to the molten glass the yellow pigments prepared from a calcined mixture of tin, lead and silica compounds. To avoid decomposition and dissolution of the particles and the loss of the yellow colour, the melt was roughly stirred and rapidly worked. The use of tin and the absence of antimony suggests a different, maybe later origin for these tesserae (Tite et al., 2008) as compared to other tesserae opacified with calcium antimonate. Nevertheless, other scholars studying yellow and yellow green tesserae from the same mosaics identified yellow pigments made also of lead and antimony (lead antimonate) and lead-tin antimonate. Recent studies showed that lead stannate, lead antimonate and lead-tin antimonate yellow pigments were in use at the same time (different yellow hues) also during the second-century AD (Verità et al. 2013).

The turquoise colour of tesserae PA-R1 (light, opacified with bone ash) and PA-R10 (dark, slightly opacified with Ca-antimonate) was obtained by adding copper in oxidised form (CuO 1.2 and 4.2%, respectively) to a manganese-free glass (MnO 0.03%). The presence of rare tin oxide particles (cassiterite crystals) in both samples suggests that copper was added as copper-rich alloys (copper-lead-tin alloy for tessera PA-R1, copper-lead-tin-zinc alloy for tessera PA-R10), and that the dissolved tin separated from the melt in the form of cassiterite crystals.

In the blue tesserae PA-R2, PA-R9, PA-R11 and PA-P17 and P18, the analyses detected the presence of cobalt oxide, which yields a deep colour even in small concentrations. In tessera PA-R9 a number of black particles of some tenths of μm in diameter were identified by optical microscopy and examined by X-ray microanalysis. They are composed of iron oxide (Fe_2O_3 75–80%), cobalt oxide (CoO 18–23%) and alumina (Al_2O_3 3–5%). These are undissolved residues of the cobalt ore; it is known that in Roman time, cobalt was added as a mineral in which other elements were also present (Gratuz et al. 1996). The addition of iron through the cobalt ore is confirmed by the higher concentration of iron of blue tesserae (Fe_2O_3 0.7–1.3%), as compared to the tesserae of other colours (Fe_2O_3 0.3–1.0%).

All the blue tesserae contain also copper, but only in the tessera PA-R2, its amount (CuO 1.35%) is significantly high to indicate a deliberate addition of copper to modify the blue hue of the tesserae. In tessera PA-R9 (the only blue one opacified with bone ash), no traces of tin were detected, which are present instead in the other tesserae opacified with antimony.

The grey colour of tesserae PA-R11 and R12 is given by calcium antimonate crystals dispersed in a well decolourised glass (also manganese was added).

The dove-grey colour hue of tessera PA-R12 was obtained by adding to a white glass opacified and coloured by Ca-antimonate microcrystals, reddish particles, having a silica-alumina composition containing minor amounts of iron, sodium, potassium and calcium (terracotta particles). In Roman time, terracotta particles were commonly used as pigments to modify the hue of grey, flash tone and green glass mosaic tesserae (Verità and Santopadre 2010).

Conclusions

The results obtained from the macroscopic and microscopic analyses carried out in this study on stone and glass tesserae from the mosaics of Villa del Casale (Piazza Armerina, Sicily) may be summarised as follows:

The white, pinkish and black tesserae have been obtained only from stones, including obsidian (which, for geographical reasons, is likely of a Liparote origin) for the black ones;

The blue, turquoise and gold leaf tesserae were exclusively made of glass;

Both stone and glass were used for dark red and yellow hues.

The study of the geological maps of central Sicily, including those related to the Piazza Armerina area (AA.VV. 1955) allows concluding that stones of local geological formations have been prevailingly used for the production of white, black, yellow, green and red tesserae. Although it is quite difficult to determine precisely the quarrying places of all the stone-tesserae, it is rather likely that the white, greenish and pinkish rocks classified as biomicrites belong to the so called “Scaglia varicolore” (polychrome marly limestone) that outcrop in various places of the Trapani province and is present, like the dolosparites, in the calcareous Triassic-Eocenic chaotic facies of the zone to the east and north of Valguarnera, to the north of Aidone, to the south of Barrafranca and in other areas of the Caltanissetta province, not very far from Piazza Armerina. In particular, travertines that may be associated to limestones coloured by hematite like those of PA-U10 and PA-P11 tesserae are present to the east of Barrafranca (for example on the southern slopes of Mount Torr); the red sandstone tesserae (for example PA-U6) may have been obtained from very oxidised horizons of the arenaceous formations dating to the Upper Pliocene outcropping around Piazza Armerina. The laboratory analyses have then confirmed the hypotheses made by the mosaics restorer Germanà about the identity of the stone-tesserae with the small stone blocks found in the excavations of the Villa, thus proving an in-situ production of the tesserae, some of which were also obtained from the leftovers of the stones used for the opera sectilia decorating several floors and walls of the Villa.

The analyses of the glass tesserae allowed tesserae of different origin to be identified, with characteristic glass compositions and/or type of opacifier used. Natron type glass tesserae opacified with calcium antimonate manufactured according to the Roman tradition have been used in the original mosaics, in particular for those colours that were not readily available among stones, such as the analysed blue tesserae from the mosaics of the *palestra.*

On the other hand, other glass tesserae sampled in the storehouses give evidence of at least two subsequent interventions in the Villa del Casale. A first intervention can be dated to between the fifth and seventh centuries, as demonstrated by the tesserae opacified with bone ash. The leaf of two tesserae refers to gold coins circulating between the fifth and sixth centuries, confirming that this period seems the most likely one for this intervention. A successive intervention using gold leaf tesserae can be dated to not before the eighth century (recent analyses of Byzantine glass weight seem to anticipate this date to the end of the sixth to beginning of the seventh centuries; Schibille et al. 2016), as attested by the glass composition of soda ash type. Because these are loose tesserae, it was not possible to ascertain whether it was a restoration intervention or the creation of new mosaics.

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