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CHARACTERIZATION OF ROMAN GLASS FROM CASA BACCO DEPOSIT AT POMPEII BY WAVELENGTH-DISPERSIVE ELECTRON PROBE MICROANALYSIS (EPMA)

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ABSTRACT

A set of 15 of variously coloured and decorated Roman glass coming from Casa Bacco deposit at Pompeii were investigated by wavelength-dispersive electron probe microanalysis (EPMA). The analyzed objects are glassware and were selected on the basis of their different color (colorless, light blue green, light blue yellow, blue, yellow-amber and emerald green). The sample set includes two finely decorated glass objects, on which a morphological study by back-scattered electrons (BSE) technique was carried out. The aim of the paper was the chemical characterization of Pompeii glass also in relationship to the contemporary analyzed Roman glasses from Mediterranean area. Based on the results, the Pompeii glass fragments are soda lime silica glass, the typical composition of the Roman glass, usually found in many Western European sites, produced with selected raw materials, from the eastern Mediterranean, possibly in or near to Egypt. In particular, the chemical composition of green emerald glasses suggests the use of plant ash, related to the production of the emerald green color, possibly being made in the same eastern Mediterranean area.

KEYWORDS: Roman glass, Emerald green glasses, Pompeii, EPMA, Raw materials, Production technology, Mediterranean area.

1. INTRODUCTION

Numerous studies conducted in recent decades on the Roman glass, allowed to know raw materials and production technology in ancient time. The composition of the glass is already known: it is obtained by the fusion of three main components: silica as network-former, calcium oxide as a network-stabilizer and an alkaline component fraction, as flux to lower the melting point of the mixture. In Roman time natron was the main type of flux used even though limited local productions of glass produced from plant ash are known (Henderson, 1991; Towle and Henderson, 2007). The relative compositional homogeneity of the natron glass led to the accepted hypothesis that glass from raw materials was probably made in a limited number of primary workshops, and then distributed throughout the empire in the form of glass chunks to be worked in several secondary workshops (Rehren and Freestone, 2015; Jackson and Paynter, 2016).

Numerous studies of chemical and isotopic characterization have been carried out on ancient glass, allowing to go back not only to the production technology and to the diffusion of glass in antiquity but also to localize the raw materials and possible production centers (Freestone et al., 2003; Paynter, 2006; Wedepohl and Baumann, 2000; Freestone et al., 2000; Freestone, 2003; Jackson, 2005; Degryse and Schneider, 2008; Henderson et al., 2010; Ganio et al., 2012; Nenna, 2014; Brems and Degryse, 2014; Schibille et al., 2017).

Considering Pompeii town, the existing literature data on the production of glass (Verità et al., 2001; Vallotto and Verità, 2002; Verità, 2004; Arletti et al., 2006a; De Francesco et al., 2010; Boschetti et al., 2016; Caggiani et al., 2017) indicate that most of the Pompeii glass was produced with similar raw materials, probably from Egypt and Middle East regions. No archaeological evidence of raw material fusion centers was identified, although a considerable amount of raw glass was found in Pompeii. Pliny the Elder (23 - 79 AD) in the Naturalis Historia cites, as the main source of silica sand, the mouth of the River Belus, now called the Na'aman River, south of Acre in Israel and the Italian coastal sands, nearby Naples. For this reason, Silvestri et al., 2006 not excluded glass production in centers placed in Campania region (Italy).

In Pompeii, in addition to the very common blue-green glass, in which colouring and decolouring agents were not employed (De Francesco et al., 2010), there was a minor but high quality production of intensely colored and opaque glasses (green, blue, yellow, red) used both for blown artifacts and for mosaic tesserae (Verità, 2004).

Specialized centers could have been produced these glasses and then sent to secondary centers to be processed, effectively in Pompeii no colored and opaque raw glasses were found. The coloring/opacifier process could have taken place in secondary centers using of appropriate ingredients (Verità, 2004).

This new preliminary archaeometric study was performed on fifteen glass fragments characterized by different colors (colorless, light colored, blue, yellow-amber and emerald green). The choice of different colors is necessary to delineate the compositional and technological features of the single colored glass artifact also in relationship with literature data.

The purpose of this paper is to characterize the raw materials and to improve the knowledge of glass production technology of Pompeii in Roman times. The study was carried out by Electron Probe Micro Analyzer (EPMA) that has allowed the chemical characterization of the glasses. Back-scattered electrons (BSE) technique was employed to perform a stratigraphic analysis of the decorated glasses.

A great importance was given to emerald green glass, with the aim to extend and confirm the more recent analyses which defined particular, this glass production widespread by the 1st century A.D. (Jackson, S. Cottam, 2015).

Indeed, within the early Imperial glass vessel repertoire, emerald green vessels form an unusual compositional and typological group. Unlike most of the glass of the late Hellenistic and Roman period, emerald green glass was produced with the addition of plant ashes (Cottam and Jackson, 2018).

2. MATERIALS AND METHODS

The sample set consisted of 15 glass fragments dated 79 AD, coming from the Casa Bacco deposits of the archaeological site of Pompeii. The sampling was carried out with the assistance of archaeologists from the Archaeological Superintendence of Pompeii, in order to collect representative glass samples. Table 1 shows the ID number, description and color of the analyzed glasses. The sample set is mainly composed by different color glassware including cups, jars, plates, bottles, a raw glass chunk and a particularly interesting and very refined bird-shaped ampoule. Most of the studied glasses come from *Regio I, Insulae 11, 13 and 16*, the bird-shaped ampoule comes from *Regio II, Insula 3*, others from *Regio III, Insula 6* (Tab. 1).

Three glass samples and the raw glass are light blue green, two light yellowish green, one colorless, five emerald green, two fragments blue (one light and one deep blue) and only one is yellow-amber in color (Fig.1). Two samples are decorated: the light blue small jar VP13 is characterized by a white opaque decoration producing a "marbled effect" surface; the yellow-amber bird-shaped ampoule (VP2) shows traces of a discontinuous shiny metallic coating (Fig.1).

Table 1. Type, color and location of the analysed glasses. The term "Incertae" indicates the indefinite exact place of discovery (home, public building, shop, etc...).

Sample	*Id. number*	*Description*	*Colour*	*Provenance*
VP2	10235/A	Bird-shaped ampoule	Yellow-amber	*Regio II Insula 3*
VP3	11993/A	Unguentaria	Light Blue green	*Regio I Insula 16*
VP5	11424	Cup	Colourless	*Regio I Insula 13*
VP6	12066	Handled bottle	Light Blue green	*Incertae*
VP4	34901	Pitcher	Light Yellowish green	*Incertae*
VP9	5895	Jar	Light Blue green	*Regio III Insula 6*
VP12	12177	Plate	Light Yellowish green	*Regio I Insula 11*
VP16	13111	Raw glass	Blue-green	*Incertae*
VP11	11183	Cup	Blue	*Regio I Insula 13*
VP13	12073	Small jar	Marbled Blue	*Incertae*
VP7	18030	Plate	Emerald green	*Incertae*
VP8	18028	Cup	Emerald green	*Incertae*
VP10	11334	Plate	Emerald green	*Regio I Insula 13*
VP14	156/3	Plate	Emerald green	*Incertae*
VP15	18022	Plate	Emerald green	*Incertae*

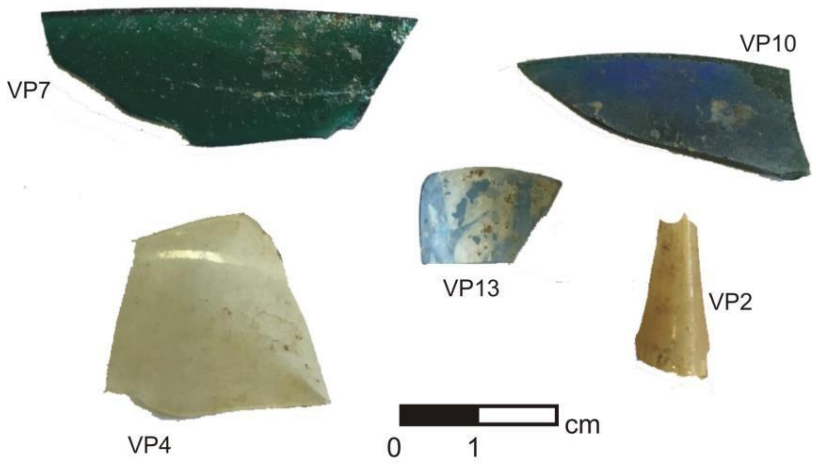

Figure 1. Representative analysed glass samples from Pompeii.

The samples show a quite good conservation status; these are partially deteriorated on the surface and show a discontinuous alteration layer producing localized iridescence effects. Marked corrosion phenomena are not evident in most glasses, except in the emerald green glasses which therefore have an opaque appearance.

Each glass fragment was preliminarily cleaned by an ultrasonic bath in order to eliminate any residual dust. Polished cross-sections were obtained, embedding the glass fragments in epoxy resin blocks, then transversely cut to enable unaltered glass surface analysis. The surface was finally polished with diamond pastes and then coated with a graphite layer to be studied by Electron Probe Micro Analyzer (EPMA), JEOL- JXA 8230 model, with a W/LaB6 source (Jeol, Tokyo, Japan), equipped with 5 Spectrometers WDS with LDE, TAP, PETJ and LiF crystals and a Spectrometer EDS – JEOL EX-94310FaL1Q - Silicon drift type (Res 129 eV).

The EDS system was used for the identification and quantization of the major elements, WDS system for minor and trace elements.

For each sample, the average of at least three EDS analyzes were performed on areas of 100 micron square (10x10) for a time of 30 seconds at 15 kV and probe current (electron beam) 15 nA. A defocused electron beam with a diameter of 50 μm was used so as to prevent volatilization of light elements such as sodium.

With EMPA, the composition was achieved by random point microanalyses, using spot analyses of about 1 μm, at least 6 for each samples.

Detailed EDS analyses on both decorated glass samples VP2 and VP13 was aimed to define the composition and technology of the decoration layer.

The BSE images of the cross section of the two glasses were used to determine the morphological parameters of the decorated glass layer, such as the thickness of the decoration and the corrosion layer.

The system was calibrated with a mixture of mineral and metal standards: Albite (Na, Al), Diopside (Ca, Mg, Si), while for the other elements oxides and metals were used.

Matrix corrections were calculated by ZAF method. The analytical error was ~1% rel. for the major elements, and it increases as their concentration decreases. The detection limits under the specified working conditions range between 0.001 and 0.2 wt% for WDS and EDS system, respectively.

3. RESULTS AND DISCUSSION

3.1. Glass characterization – raw materials

Chemical data by EDS and WDS analysis are listed in Tables 2 and in Tables 3. All the analyzed Pompeii glasses are soda lime silica glass with SiO_2, Na_2O and CaO in the ranges 64.61-72.35 wt%, 14.24-18.39 wt% and 5.79-8.64 wt% respectively, according to the literature data on glass of the period (Degryse and Schneider, 2008; Verità, 2004; Sayre and Smith, 1961; Aerts et al., 1999; Arletti et al., 2006b; Silvestri et al., 2005; Fermo et al., 2016; Foster et al., 2009).

Table 2. Major elements. Results of EMPA-EDS analysis performed on 15 Pompeii glass samples. Elements are expressed in wt %. (-) below the detection limit.

Sample	Na_2O	MgO	Al_2O_3	SiO_2	P_2O_5	SO_3	ClO	K_2O	CaO	TiO_2	MnO	FeO
VP2	16.45	0.57	2.45	68.89	0.14	0.30	1.87	0.58	8.38	-	0.02	0.37
VP3	15.13	0.57	2.36	71.59	0.18	0.20	1.56	0.58	6.63	0.02	0.93	0.24
VP5	15.73	0.47	1.95	71.91	0.17	0.31	1.66	0.45	5.79	0.05	1.16	0.36
VP6	14.85	0.57	2.40	71.41	0.15	0.20	1.68	0.55	7.30	-	0.66	0.22
VP4	16.19	0.60	2.26	70.60	0.19	0.33	1.63	0.45	7.13	-	0.40	0.23
VP9	16.18	0.73	2.64	71.33	0.18	0.28	1.35	0.54	6.29	-	0.27	0.21
VP12	14.51	0.51	2.46	72.35	0.16	0.25	1.62	0.45	7.27	0.10	0.03	0.29
VP16	17.40	0.58	2.44	67.88	0.13	0.31	1.19	0.72	8.12	-	0.47	0.36
VP11	15.26	0.55	2.27	69.32	0.23	0.18	1.34	0.80	8.64	0.05	0.47	0.90
VP13	17.60	0.66	2.43	66.94	-	0.28	1.73	0.47	7.80	0.11	1.00	0.98
VP7	18.39	1.46	1.66	66.32	0.72	0.67	1.62	1.36	5.95	0.16	0.76	1.05
VP8	16.83	1.53	2.34	65.83	0.47	0.55	1.46	1.20	7.10	0.29	1.19	1.23
VP10	14.23	3.25	2.07	64.41	1.48	0.32	1.27	1.95	8.23	0.20	0.87	1.34
VP14	15.00	2.27	2.34	65.21	1.09	0.47	1.39	2.17	7.61	0.18	1.26	1.40
VP15	17.28	2.35	1.76	64.69	0.90	0.43	1.79	1.40	7.44	0.20	0.38	1.15

Table 3. Trace elements. Results of EMPA-WDS analysis performed on 15 Pompeii glass samples. Elements are expressed in ppm. (-) below the detection limit.

Sample	Sn	Mo	Cu	Co	Cr	Pb	Sb
VP2	103	53	350	40	33	-	-
VP3	103	10	20	-	30	160	-
VP5	53	43	233	-	10	-	-
VP6	7	3	26	3	3	73	-
VP4	53	16	103	30	3	140	-
VP9	123	56	63	13	10	-	-
VP12	80	3	53	10	16	-	-
VP16	120	-	23	-	10	56	-
VP11	73	23	1930	1453	6	60	-
VP13	50	6	876	166	10	13	-
VP7	5176	-	24676	-	10	373	-
VP8	5140	30	21996	-	33	663	-
VP10	3343	50	19056	-	36	270	-
VP14	3733	63	20460	-	6	740	-
VP15	3116	30	18283	-	50	123	-

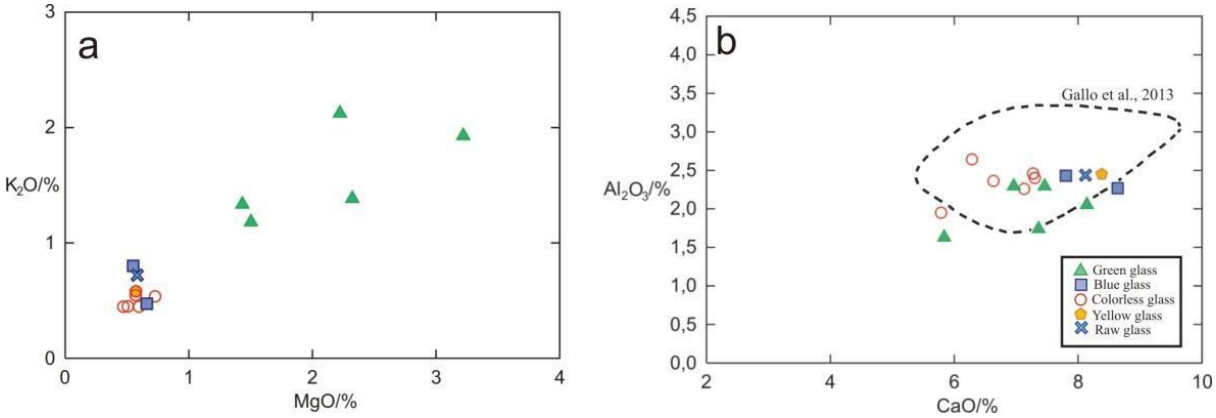

Figure 2. MgO vs. K_2O (a) and CaO vs. Al_2O_3 (b) for the Pompeii glasses. Dotted area contains the compositional field of 'typical' Roman glass after Gallo et al. (2013).

In Fig.2a, MgO vs. K_2O plot, the low concentrations of potash and magnesia, (less than 1.5 wt %) for most of the analyzed samples (VP2, VP3, VP4, VP5, VP6, VP9, VP11, VP12, VP13, VP16), indicate the use of natron as a flux (Paynter, 2006; Freestone, 2003).

The colorless and the light colored glasses (VP3, VP4, VP5, VP6, VP9, VP12 and VP16 raw glass) have the highest and the most homogeneous values of silica, between 70.60 wt% and 72.35 wt% (Tab.2), that could indicate more careful selection of raw materials.

The emerald green glasses (VP7, VP8, VP10, VP14 and VP15) have the highest concentrations of MgO (1.46 - 3.25 wt%), K_2O (1.20 - 2.17 wt%) and P_2O_5 (0.47 – 1.48 wt%) respect to all the other glass fragments (Tab.2).

Lilyquist and Brill (1993) suggest concentrations of magnesia and potash above 1.5 wt% indicate manufacture using plant ash rather than solely natron.

For Jackson and Cottam (2015) phosphorus pentoxide and magnesia, and potash and magnesia, are strongly correlated in the early to mid1st –century emerald green glasses from Frejus and Barzan in France, Colchester in England and Ribnica and Trojane in Slovenia, which suggests that the green samples as well as those from other datasets (Gallo et al., 2013; Henderson, 1996; Thirion-Merle et al., 2005) form a single compositional group using the same alkali type or potentially that natron glass was mixed with a plant ash which was high in phosphorus.

As shown in Table 2, all the emerald green glasses analyzed have the lowest SiO_2 values, if compared to the typical Roman natron glass; this may be related to the need to introduce enough alkali, trough greater quantities of plant ash, in order to flux the silica (Jackson and Cottam, 2015). Cottam and Jackson, (2018) suggest that emerald green glasses represent a specialized production, using a plant-ash component, during the 1st century AD.

Calcium and aluminum concentrations, together with iron and titanium, are in general particularly diagnostic of the sand source employed for silica-soda-lime glass production, as they reflect the impurities (calcite, feldspars, clay minerals and heavy minerals) present in the original sand (Freestone et al., 2000; Freestone, 2003; Gallo et al., 2013; Freestone, 1994; Freestone et al., 2002).

The Fig. 2b, CaO vs. Al_2O_3 plot, shows the comparison of the analyzed Pompeii glasses with the main 1st - 3rd century A.D. compositional groups ('typical' Roman glass) found in the Western provinces (Arletti et al., 2006b; Foy et al., 2003; Silvestri, 2008; Silvestri et al., 2008) as in Gallo et al., 2013. Most of Pompeii glasses fall in the area of the European Roman glass production. This compositional range, already found in many Western European sites and in the Mediterranean area, is ascribable to the use of similar raw materials for the glass productions of the entire Empire, probably from the Middle-East area (Sayre and Smith, 1961; Silvestri et al., 2005; Foy et al., 2003; Silvestri, 2008; Nenna et al., 1997; Picon and Vichy, 2003; Al-Bashaireh et al., 2016, Liritzis et al., 2018). Roman glass is thought to have been made from coastal sands of the Syro-Palestinian region, probably near the mouth of the river Belus (Foy et al., 2003).

In Fig.2b it should be noted that most emerald green glasses (VP15, VP10 and VP7) show the lowest aluminum content but calcium concentration similar to the mineral soda glasses. Only the VP7 glass displays the lowest calcium values (Tab.2 and Fig.2). Calcium content of the emerald green Pompeii glass is very similar to some of the few emerald green glass of Archaeological Museum of Adria (North eastern Italy) of Gallo et al., (2013). Jackson and Cottam, 2015) suggest that different (low-lime) sand source, crushed quartz or plant ash particularly low in calcium was used for emerald green glass production. However, the addition of plant ash, not neces-

sarily indicate that plant ashes fulfilled the role of the flux but the higher content of magnesium, lime and potassium than natron, along with the presence of small amounts of charcoal in the ash would favour the formation of the green colour (Cottam and Jackson, 2018).

It is therefore possible that the emerald green color of Pompeii glasses was obtain by the addition of plant ash to the natron main component in the production areas of natron glass, most likely in the Eastern Mediterranean as suggested by Jackson and Cottam, (2015).

3.2. Colouring/decolouring agents

Roman glasses generally, show a high level of technology, particularly in terms of color control, as testified by the presence of coloring and decoloring agents in the glass production. As already mentioned above, the analyzed Pompeii glasses show different colors: light blue green, light yellowish green, colorless, blue, yellow-amber and emerald green.

In the FeO vs. MnO plot (Fig.3) the Pompeii glass samples are divided in two groups, on the basis of the FeO content. The light colored (light blue green and light yellowish green glasses, VP3, VP4, VP6, VP9, VP12) the yellow-amber (VP2), the colorless (VP5), and the raw glass (VP16), show a low iron concentration (FeO 0.21-0.37 wt%), that would confirm, together with the high SiO_2 value (Tab.2), the use of a selected sand for the production of this group of glasses.

The blue and the emerald green glasses instead have the highest FeO contents from 0.90 to 1.40 wt% and so, are well separated from all the other glasses as shown in Fig.3. The manganese concentrations vary considerably in both groups, distinguished by iron content, as evidenced in Fig.3. In particular, in the lower FeO group, two light blue-green glasses (VP3, VP6) and the colorless glass VP5, have MnO>0.5% in weight, which indicates the intentional addition of manganese as decoloring agent (Jackson, 2005), probably as pyrolusite (MnO_2). The latter one was particularly widespread in the Roman period to neutralize the color due to the iron oxides naturally present in the raw materials (Jackson, 2005; Silvestri et al., 2005; Silvestri et al., 2008). This group comprises mainly glass with manganese above background levels (MnO >0.025 %) and Sb_2O_5 below the detection limits of the EPMA instruments used for the analyses (ca. 0.03 %) in Schibille et al., (2017), similar to the equipment used in this research to analyze the Pompeii glasses.

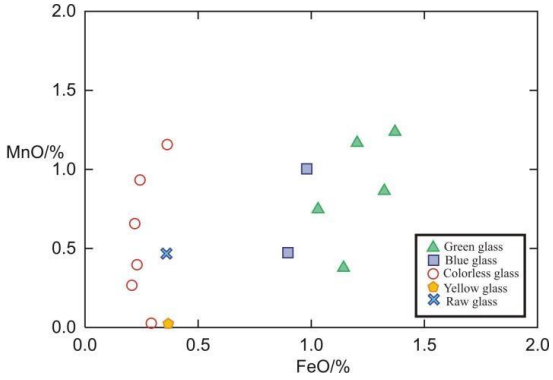

Figure 3. FeO vs. MnO plot of all analysed Pompeii glasses

It is important to point out that in all the analyzed Pompeii glass samples, antimony is not present or it is below the detection limit, except in the white decoration of the blue marbled glass (VP13) where it was added as opacifier agent, as will be discussed later.

Since in all the analyzed Pompeii glasses, antimony is below the detection limit, as evidenced in Tab.3, the use of antimony as decoloring agents, could be excluded. As a result, manganese-based compounds were the only decoloring agents used for the few decolored Pompeii glasses analyzed here, and so they are included in the Mn-decoloured group, of the four main groups of Gliozzo, (2017).

The other light colored glasses (VP4, VP9, VP12, the natural blue green raw glass VP16 and the yellow amber VP2 samples) are characterized by MnO content below 0,47 wt% (Tab.2), too low to be considered as intentionally added. This group is consistent with the Low-manganese (low-Mn) glass of Jackson and Paynter (2016) with low concentrations of manganese of up to 0.8 wt% and no antimony, and with the NE-I/ unintent-Coloured group of Silvestri et al., (2018). This naturally coloured glasses is the dominant, raw, blue–green glass by the first century, and is thought to be manufactured in the Syria-Palestine region (Foy et al. 2003).

In two samples, the yellow-amber (VP2) and the light yellowish green (VP12) glasses, the MnO content is very low (0.02 and 0.03 wt%, respectively, see Tab.2), that is, close to the detection limit, but they are very similar in composition to the other low FeO glasses. For those two glasses, but particularly for the yellow amber glass (VP2) may be hypothesized that the low manganese contents are likely to be reflected in their colour technology, for these the yellowish or amber colour, probably may be due to the ferrisulphide chromophore (Schibille et al., 2017). The formation of this colour required reducing conditions and would have been favoured in a glass which had not been oxidized for the purpose of decolouration by the addition of Mn or Sb (Freestone and Stapleton, 2015).

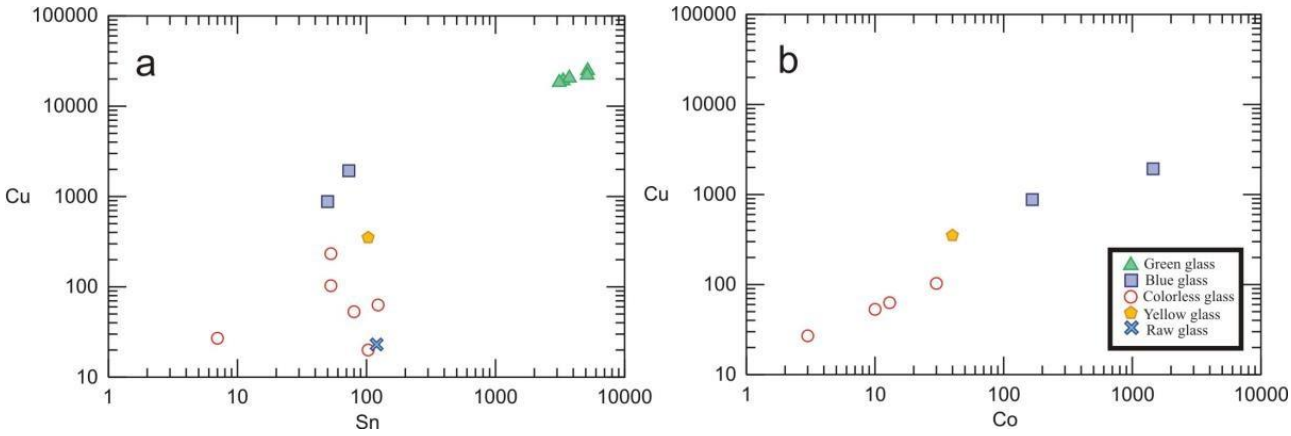

Figure 4. Log-log Sn vs. Cu plot (a) and log-log Co vs. Cu plot (b). In (b) the cobalt free glasses were excluded.

Quite all, blue and emerald green glasses, show MnO > 0.5 wt% values (except the deep blue glass VP11 and the emerald green VP15) and FeO values ranging from 0.90 to 1.40wt%.

As already highlighted in Fig. 2a, the K_2O, MgO and P_2O_5 contents of the emerald green glasses suggest the use of different raw materials, such as the addition of plant ash. Indeed, they form a single compositional group using the same alkali type, and possibly the same source (Jackson and Cottam, 2015), as well as those from other green glass datasets (Gallo et al., 2013; Henderson, 1996; Thirion-Merle et al., 2005).

The CaO values of emerald green glasses are similar to those natron-based, but the low SiO_2 concentration, high FeO and TiO_2 contents (Table 2) indicate the use of distinct raw materials, possibly richer in heavy/mafic minerals in the case of soda ash glass (Gallo et al., 2013).

As clearly shown in the Fig.2a, the blue glasses are natron-based Roman glass, their color is essentially due to cobalt, as will be discussed later in the following paragraph.

3.3. Coloured glasses

3.3.1. Emerald green glasses

Emerald green is a Roman glass colour appearing in the first decades of the first century CE and going out of general use by the last quarter of that century (Cottam and Jackson, 2018). As already shown in Fig.2a and in Fig.3, the chemical composition of green emerald glasses suggests the use of plant ash and so different raw materials for their production. Verità (2004) indicate for the few Pompeii green glasses analyzed, the use of sands of different origins compared to the most of the other Pompeii analyzed glasses.

The Fig. 4a, the log-log Sn vs. Cu plot, shows the emerald green glasses with the highest Cu and Sn contents, clearly separated from all the other glasses.

All the emerald green glasses have high copper contents, above 10.000 ppm (18283-24676 ppm) well correlated with tin concentrations (3116-5176 ppm). According to Jackson and Cottam (2015), the presence of copper in glass, as Cu_{2+}, can produce either blue or green hues, but the colour probably depends upon interactions of copper and iron and to some extent manganese, tin, lead (Tab.2) and antimony. As mentioned before, in all the Pompeii glasses, antimony is always below the detection limit of the used EMPA. The high tin content may be linked to bronze addition, as lead at low concentrations, as suggested by Jackson and Cottam (2015).

The chemistry of the emerald green glass suggests that it is part of a natron-glass production system, possibly being made in the same general region. The introduction of plant ashes seems to be related to color production (Cottam and Jackson, 2018). The exact provenance of the glasses is more difficult to ascertain, but the trace element data indicate the sands used to produce these glasses have characteristics common to those from the eastern Mediterranean, possibly in or near to Egypt (Jackson and Cottam, 2015).

The differences but also the similarities of the raw materials used to produce plant-ash and colorless mineral-soda Roman glasses, suggest that the emerald green glass production was not an entirely distinct, separately located industry, but was very close to the other primary centers (Jackson and Cottam, 2015).

3.3.2. Light colored, amber and blue glasses

As shown in Fig. 4 and in Table 3, the colorless and most of light blue and yellowish green glasses have low values of Cu, Sn, Co and Pb, indicating the use of 'fresh' glass and so, as already highlighted, the use of selected raw materials. Only some glasses show Cu and/or Pb contents higher than 100 ppm (Table 3), suggesting possible recycling of coloured cullet and/or scraps added during melting (Gallo et

al., 2013). Indeed, trace elements such as Co, Zn, Sn, Cu and Pb, can give information about the glass recycling process in ancient times (Jackson, 1996; Freestone et al., 2002). Values of such elements comprised between 100 and 1000 ppm have been considered as contamination, while values exceeding 1000 ppm have been referred to as an intentional addition (Gliozzo, 2017).

The recycling of glass in ancient times was a very common practice not only in Roman times but also in the following periods, as evidenced by the chemical composition of numerous glasses found at Pompeii and in the surrounding areas (Arletti et al., 2006a; De Francesco et al., 2010; De Francesco et al., 2014; Arletti et al., 2006c).

The unintentionally blue-green color of raw glass VP16, is due to the impurities naturally present in the sand (Tab. 3).

The log-log Co vs Cu plot (Fig. 4b) displays only the blue glasses, the amber yellow one and four colorless glasses (VP4, VP6, VP9, VP12). The other glasses were excluded because they are cobalt free.

Apart from iron and manganese, copper and cobalt were important colouring agents in the ancient glass industry (Mirti et al., 2002). The deep blue glass VP11 has significant amounts of cobalt and copper (1453 ppm and 1930 ppm, respectively) which suggest the use of trianite ($2Co_2O \times CuO \times H_2O$) as coloring agent, commonly added for the production of Roman blue glass (Arletti et al., 2008). The VP13 sample is light blue in color, probably due to lower Co and Cu content than in VP11 and the high Fe and Mn content (about 1wt %), as shown in Fig. 3 and Tab.2.

As already said the yellow-amber color of the bird shaped ampoule was probably obtained in strongly reducing conditions (Schibille et al., 2017), since the low contents of chromophore elements may be considered as contamination (Gliozzo, 2017).

3.4 Decorated glass

The two decorated Pompeii glasses include the yellow-amber bird shaped ampoule (VP2) and a light blue small jar (VP13). The yellow-amber VP2 glass shows on the external surface a discontinuous residue of a metallic coating, while the light blue small jar VP13 is characterized by a white opaque decorations producing a "marbled effect" surface (Fig.1).

Table 4. EMPA results on VP2 and VP13 decorated glasses. Data are expressed in wt%. VP2 - EL: External Layer (metallic); VP13 - DL: decoration layer; WCRY: chemical composition of calcium antimonate crystals.

	VP2	VP13	
	EL	DL	WCRY
Na_2O	1.7	10.19	2.24
MgO	0.95	0.42	-
Al_2O_3	5.97	1.56	0.75
SiO_2	31.8	42.98	20.77
P_2O_5	0.62	-	-
SO_3	0.79	0.44	-
ClO	0.71	0.51	-
K_2O	-	0.37	-
CaO	2.42	4.64	14.34
TiO_2	-	-	-
MnO	-	-	-
FeO	1.39	0.1	-
CuO	0.9	-	-
SnO_2	52.73	-	-
Sb_2O_3	-	38.78	61.91

The micro-structural analysis performed on the two decorated glasses, has highlighted the technologies used for the external decorations and different and very interesting morphologies of glass alteration. Since the glass alteration study was not the purposes of this work, it will be the subject of future detailed studies. Table 4 shows the results of the EDS analyses obtained on the decoration layers of these two samples.

3.4.1 VP2 glass

The EMPA analysis performed on the external polished cross section of the yellow bird-shaped ampoule glass VP2, has highlighted the presence of three distinct portions as shown through the BSE images in Fig.5a. The external layer (EL) has a strong shining metallic effect, it is very thin, only a few microns, has an irregular thickness and is missing in some portions, probably its original thickness is not preserved. The intermediate layer (IL) shows a darker grey hue in comparison to pristine glass (PG), probably owing to the alteration of buried glass in a humid environment (Silvestri et al., 2005; Cox and Ford, 1993; Salviulo et al., 2004; Huisman et al., 2008). It has a constant thickness of 20 microns with cracks and detached portions in some areas (Fig 5a).

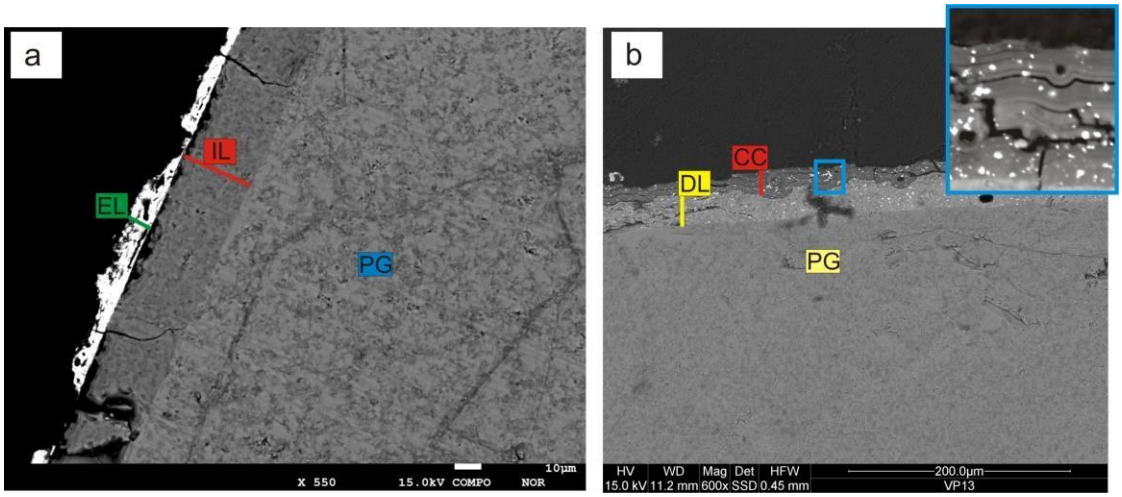

Figure 5. BSE images of the decorated glasses: a) VP2 decorated glass - PG: Pristine Glass; IL: Intermediate Layer (dark grey hue); EL External Layer (metallic layer). b) VP13 decorated glass - PG: Pristine Glass; DL: Decoration Layer; CC: Corrosion Crust.

Table 4 shows the results of EDS analyses performed on the metallic external layers. The intermediate layer has very similar features of the gel layer according to Muller et al., 1995. The external layer (EL) shows high concentrations of tin, up to an average value of 52.73 wt%, but other metals such as iron (1.39 wt%) and copper (0.90 wt%) are present. The external metal layer also contains SiO_2 (31.80 wt%), Al_2O_3 (5.97 wt%) and Na_2O (1.70 wt%) as shown in the Table 4.

Unfortunately, the bad state of conservation of the very thin decoration layer prevents the complete understanding of its original chemical composition. However, we could suppose the use of a tin-based metallic compound on the surface of the glass object to give a shiny decorative effect. Further analysis will be necessary to better know the technological aspects of the metallic layer.

3.4.2 VP13 glass

The BSE images obtained by the electronic microprobe on the external polished cross section of the light blue small jar VP13, is shown in Fig.5b. The decoration layer (DL) is clearly distinguished from the pristine glass (PG), due to the lighter grey hue and the presence of numerous scattered crystals. Its thickness is quite regular, about 40 microns, but in some portions, it is fractured and thinned. The outermost part of the decoration layer is dark gray in color, displays a thin finely laminated corrosion crust (CC), composed of iridescent lamellae parallel to the unaltered glass surface, of variable thickness ranging from a few microns to about 20 microns (Fig. 5b).

Similar morphologies have been reported in the literature for altered glasses of several archaeological sites (Silvestri et al., 2005; Cox and Ford, 1993; Salviulo et al., 2004; Morgenstein et al., 1999; Dal Bianco et al., 2004; Dal Bianco et al., 2005). The EMPA analyses were performed on the well-preserved portion of the decoration layer (DL), and on the crystals (Tab.4).

The decoration layer (DL) is characterized by very high Sb_2O_3 content (up to 38.78 wt%), which was certainly intentionally added to achieve the desired white opaque decorative effect. All the other oxides are lower than in the pristine glass: SiO_2 and Na_2O are respectively of 42.98 wt% and 10.19 wt%, Al_2O_3 shows a content of 1.56 wt% and CaO of 4.64 wt%, as reported in Tab.4. This composition suggests the use of an antimony rich glass to obtain the white opaque decorative effect, as the marbled effect of the VP13 small jar. This type of decoration, highly widespread in Roman period, was obtained by pressing thin strands of molten glass on the surface (Stern, 2004).

The EDS analyses on the white crystals (CRY) inside the decoration layer (Fig.5b), showed significant average concentrations of antimony oxide (61.91 wt%) and calcium oxide (14.34 wt%) as main chemical elements. On the basis of the results we can assume that the white crystals were formed in situ, following the reaction between an antimony-based agent, probably in the form of antimony oxide (Sb_2O_3, Sb_2O_5), and the calcium present in the glassy matrix (Boschetti et al., 2016; Lahlil et al., 2008; Lahlil et al., 2010). The calcium antimonite could be in form of an orthorhombic ($Ca_2Sb_2O_7$) or hexagonal ($CaSb_2O_6$) phase on the basis of the temperatures used for decoration process.

4. CONCLUSIONS

The analysis of fifteen glass fragments of I century AD, including some emerald green glasses and two decorated glasses, from Casa Bacco deposits in

Pompeii allowed us to characterize the raw materials and the technology employed for their production.

The chemical characterization evidenced that most of the analyzed Pompeii glasses, the colorless, the light blue green and light yellowish green glasses, the yellow-amber, the blue and the raw glass, are soda lime silica glass, the classical composition of Roman glass productions, usually found in many Western European sites.

The compositional homogeneity, particularly for the colorless glass and light colored glass is ascribable to the use of similar and well selected raw materials, probably imported coastal sands from the Middle East and natron from Egypt. The intentional addition of manganese, as decoloring agent, was detected only in the colorless glass and two light blue green glasses. The other light colored glasses and the naturally colored raw glass are Mn-poor, below the intentionally addition value confirming the raw materials careful selection.

The yellow-amber color of the bird shaped ampoule is probably due to the ferrisulphide chromophore. The formation of this color required reducing conditions, but because of the low manganese content, no addition of decolouring agents and oxidation conditions were necessary.

The chromophore agents in the production of the deep blue glass are cobalt and copper which suggest the use of trianite ($2CO_2O \times CuO \times H_2O$) commonly added in Roman blue glass. The decorated light blue glass, owes its color not only to cobalt (lower than in the deep blue) but also to iron and manganese contents.

For the Pompeii emerald green glass production, different raw materials were used. The highest values of potash, magnesia and phosphorus pentoxide in these glass fragments, and their strong correlation suggests the addition of plant ash for their production. The Pompeii emerald green glasses form a single compositional group, similar to the emerald green glasses from other European sites confirming the hypothesis of an active specialized glass production close to the other primary centers in the Eastern Mediterranean, possibly in or near to Egypt.

The micro-structural analysis performed on the two decorated glasses has highlighted the use of specialized technology of the Roman glassmakers and different morphologies of glass alteration. In the yellow-amber bird-shaped ampoule glass, the refined outermost shining metallic layer represents a decorative effect, obtained using a tin-based metallic compound, but the alteration state prevents to quite understand the original composition and so the technological aspects.

The marbled decoration of the blue glass, is formed by a superficial glass layer obtained by the use of an antimony-based opacifier agent (calcium antimonite), to obtain the white opaque decorative effect.

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