Original Study

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Chemical Characterization of Vitreous Finds From Cosenza Cathedral (Calabria – Italy) by the Combined Use of Analytical Techniques

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Abstract: This article presents an archaeometrical research carried out on twenty-six vitreous finds collected in the Cosenza Cathedral (Calabria, Italy). The glasses have been subdivided in two typo-chronological groups. The first group is composed of 14 vitreous samples dating to the 4th–6th century AD. The second group includes twelve samples; seven are stems of funnel-shaped hanging lamps which date between the 12th and the 13th century AD, two are bottlenecks of *balsamaria* and three are concave bases. The aims of this study were the determination of the chemical composition of vitreous finds and the individuation of the primary glass sources. The samples were characterized through Electron Probe Micro Analyser with Wavelength Dispersive Spectrometer (EPMA-WDS) and Laser Ablation with Inductively Coupled Plasma Mass Spectrometry (LA-ICP-MS). The data confirm that all the finds of the first group are “silica-soda-lime” type glasses characterized by a high content of Na₂O and a low content of K₂O and MgO. On the contrary, the samples of the second group, showing higher contents of K₂O and MgO, are vegetable silica-soda-lime glasses. Their composition confirms the typological attribution to the medieval period.

Keywords: glasses, Roman, Medieval, LA-ICP-MS, EPMA-WDS

1 Introduction

The raw materials employed by glassmakers for glass production consist of a mixture of three main components: silica sand, lime and alkaline compounds or “fluxants” able to facilitate the production of glass (Fiori, Vandini & Mazzotti, 2004; Silvestri, Molin, & Salviulo, 2005a, 2008; Angelini, Gratuze, & Artoli, 2019).

The different ratios of the three components allows grouping of the glasses according to their compositional basis. Importantly, the variations in percentage and type of fluxants present allows insights into the temporal and geographical origins of glass manufacturing. Previous studies (Angelini et al., 2019 and references therein) 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. Examining the timeline proposed by Wederphol (1997), from the 2nd millennium BC to 800 BC, two different alkaline fluxes were used for glassmaking; plant ash soda was preferred in Mesopotamia Egypt, mixed soda-potash in Northern Italy. From the 1st millennium BC until the 7th century AD the flux was mostly constituted by compounds of sodium introduced using natron

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i.e. a soda rich mineral salt. From the 8th century AD the use of mineral soda was gradually phased out, in favour of ashes of coastal plants containing, in addition to sodium, high percentages of potassium and magnesium (De Juan Ares, Fernández Calderón, Muiz López, García Alvarez-Busto, & Schibille, 2018; Fiori et al., 2004; Phelps, Freestone, Gorin-Rosen, & Gratuze, 2016; Schibille, Gratuze, Ollivier, & Blondeau, 2019). In particular, potassium-rich plant ashes were preferred in central Europe and soda rich plant ashes in the eastern Mediterranean (De Juan Ares et al., 2018 and reference therein). At the same time, lead-rich glasses appeared in Europe and in the Islamic east (De Juan Ares et al., 2018).

Therefore, the chemical characterization of vitreous specimens from archaeological sites provides important information, not only on the composition of raw materials, but also on the period and method of glass production (Wagner et al., 2008).

The present investigation focused on some glass finds collected during an excavation campaign conducted in 2008 in the Cosenza Cathedral (Calabria, Italy) (Figure 1). The research is aimed at investigating the glass compositions by comparing them to the known primary glass groups produced in Syria, Palestine and Egypt and at correlating the typological and chemical analyses with a chronological attribution.

2 Materials

During the restoration of Cosenza Cathedral in 2008, a significant quantity of vitreous objects was recovered from the excavations of the church floor (Figure 1). Cosenza is a city that has played an important role since Roman times, thanks also to the favourable geographical condition that places it along the road axis of the Annia Popilia. At first Byzantine, Cosenza then became the headquarters of a Lombard gastaldato. In this historical-political context, the cultural building, as learned from written sources, became a diocese as early as the 4th century AD (Otranto, 1995). Today, as in the past, the Cathedral is the fulcrum of the historical city center.

Figure 1: a) Cosenza city in Calabria (Italy); b) the Cosenza Cathedral; c, d) photos of the excavations conducted in 2008; e) map of the Cathedral.

The stratigraphic investigation has interested the area actually occupied by the apse and presbytery of the church (Figure 1). Despite the numerous renovations and restorations to which the cathedral has been subjected over the centuries, the preliminary and macroscopic examination of all the recovered finds allowed us to classify them chronologically to between the Hellenistic and post-medieval ages. The chronology of the attendance phases has been reconstructed thanks to the presence of wall structures and the recovery of different types of finds: black-painted ceramics, lamps of *Firmalampem* type, glazed ceramics and so on.

The burial environment (wet soil) has no doubt played a part in the poor preservation and extreme fragmentation of these vitreous finds.

After a preliminary examination, the samples have been catalogued and subjected to a type-morphological analysis in order to identify their likely form and thus provide a possible chronology. Considering the two different attendance phases of the archaeological context the samples were divided into two groups.

The first group includes a total of fourteen finds. Among them, nine glasses were recovered from a large trench (approx. 0.30 m breadth x 0.40 m length and 0.49 m depth), corresponding to the stratigraphic unit (US) 122 (Figure 1). This trench cut an ancient baptismal font, characterised by an *opus tessellatum* (i.e. a white and black mosaic floor) (Figure 1) diagnostic of the end of the 4th and the beginning of the 6th century AD (for the archaeological context see Roma & Papparella, 2018, p. 161). These vitreous objects most likely represent the furnishings used for lighting the baptismal area, *super fontem*, and for executing the sacrament's practices. In the same trench ten metallic wicks and coal residues were also found.

The nine samples recovered from the US122 include height lamps (Table 1) and one bottle (US122-bott) (Figure 2). The lamps are Isings 134 type, precisely Ubaldi I.1 (Ubaldi, 1995) and similar (Isings, 1957). These types were common in different Calabrian coeval archaeological sites like Botricello and Cropani (Aisa & Corrado, 2003; Aisa & Papparella, 2003).

1A: HIMT1

1B: HIMT2

Figure 2: Vitreous finds of the first group dated to the 4th–6th century AD.

The bottle (US122-bott) corresponds to the Isings 126 type (Isings, 1957), and it is characterised by a high cylindrical neck. This specimen shows the rim and the neck decorated by spiral-wound filaments, green colored like the whole bottle. These decorations are very similar to those of some bottles of Copia-Thurii (Luppino & De Presbiteris, 2003). The characteristics of the US122-bott sample indicate an age from the 4th to the 6th century, although similar decorations were found mainly in the vitreous finds of the 6th century AD (D. Stiaffini, personal communication).

The other samples of the first group were collected from the US124 and US129 stratigraphic units. Two samples are lamps (US124-50 and US129-4), one is a concave base (US124-48) and the other one is a beaker (US124-49), (see Figure 2).

The second group includes twelve samples found in the area around the apse of the Cathedral. Although these finds cannot be attributed to a reliable stratigraphy, they can be related to the use-phase of the original apse of the church, datable to the Federician era. Seven samples are stems of funnel-shaped hanging lamps (Table 1). They are similar to numerous samples recovered in other Calabrian places of worship such as S. Niceto di Motta San Giovanni (Reggio Calabria) dating between the 12th and the 13th century AD (Coscarella, 2003). Among the other samples, two are bottlenecks of *balsamarium* (Spo-61; Spo-z) and three are concave bases (Spo/65-66; Spo-con) (Figure 3).

With regard to the colour, the artifacts can be divided into colourless and coloured, with the colours ranging from light to dark green and light brown (see Figures 2, 3, and Table1).

Figure 3: Vitreous finds of the second group dated from 12th–13th century AD.

Table 1: List of the examined vitreous finds collected in the Cosenza Cathedral.

First group (4th–6th AD)	Sample	Shape	Type	Color
	US122-7	Lamp	Isings 134/Uboldi I.1 (and similar)	green
	US122-9	Lamp	Isings 134/Uboldi I.1 (and similar)	green
	US122-10	Lamp	Isings 134/Uboldi I.1 (and similar)	green
	US122-75	Lamp	Isings 134/Uboldi I.1 (and similar)	colourless
	US122-76	Lamp	Isings 134/Uboldi I.1 (and similar)	green
	US122-77	Lamp	Isings 134/Uboldi I.1 (and similar)	green
	US122-78	Lamp	Isings 134/Uboldi I.1 (and similar)	green
	US122-y	Lamp	Isings 134/Uboldi I.1 (and similar)	green
	US122-bott	Bottle	Isings 126	light green
	US124-47	Lamp	Isings 134/Uboldi I.1 (and similar)	light green
	US124-48	Concave base		light green
	US124-49	Beaker/goblet?	Isings 111	colourless
	US124-50	Lamp	Isings 134/Uboldi I.1 (and similar)	green
	129-4	Lamp	Isings 134/Uboldi I.1 (and similar)	green
Second group (12th–13th AD)	US18-3	funnel-shaped hanging Lamp		green
	US18-5	funnel-shaped hanging Lamp		colourless
	Spo-61	Neck of <i>balsamarium</i>		colourless
	Spo-65	Concave base		light brown
	Spo-66	Concave base		colourless
	Spo-69	funnel-shaped hanging Lamp		light brown
	Spo-70	funnel-shaped hanging Lamp		light brown
	Spo-71	funnel-shaped hanging Lamp		colourless
	Spo-72	funnel-shaped hanging Lamp		light brown
	Spo-73	funnel-shaped hanging Lamp		colourless
	Spo-con	Concave base		green
	Spo-z	Neck of <i>balsamarium</i>		colourless

3 Methods

Chemical analyses of vitreous finds were carried out using EPMA-WDS (Electron Probe Micro Analyzer with Wavelength-Dispersive Spectrometer) and LA-ICP-MS (Laser Ablation with Inductively Coupled Plasma Mass Spectrometry). Before proceeding to the analytical phase, a small fragment of about 3 mm x 3 mm was collected from each sample and cleaned in an ultrasound bath with Millipore water, to remove all traces of soil (Barca, Abate, Crisci, & De Presbiteris, 2009). Samples were then fixed on slides with the fresh side facing upward. Due to their small size, three/four samples were positioned on each slide.

To determine major element concentrations, EPMA-WDS analyses were carried out using a JEOL-JXA 8230, equipped with 5 Spectrometers WDS: LDE (including both LDE1 and LDE2), TAP, PET(J), LiF, diffraction crystals. This last crystal has a diffraction plane of 2.0.0. (e.g. $2d=0.40267$ nm). 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 μ m 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 seconds was selected. Before the analyses, the sample surface was covered by a carbon sputter coating. Three point analyses were carried out on each sample. The detection limits for most elements were about 0.2 wt%.

To determine the trace element concentrations, LA-ICP-MS analyses were conducted using an Elan DRCe (Perkin Elmer), connected to a New Wave UP213 solid-state Nd-YAG laser probe (213 nm). Samples were ablated by laser beam in a cell, and the vaporised material was then flushed (Gunther & Heinrich,

1999) to the ICP, where it was quantified. Each ablation crater was generally 50 μm in diameter and about 70 μm in height. Three-point analyses were carried out on each sample.

The procedures for data acquisition were those normally used in the Mass Spectroscopy Laboratory of the Department of Biology, Ecology and Earth Sciences, University of Calabria (Barca, De Francesco, Crisci, & Tozzi, 2008; Barca, Lucarini, Fedele, 2012; Barca et al., 2019).

Calibration was performed on glass reference material SRM612-50 ppm by NIST (National Institute of Standards and Technology) in conjunction with internal standardisation, applying SiO_2 concentrations (Fryer, Jackson, & Longerich, 1995) from EPMA-WDS analyses. In order to evaluate possible errors within each analytical sequence, determinations were also made on BCR 2G by USGS (Basalt Columbia River Glass by United States Geological Survey) glass reference material as an unknown sample, and element concentrations were compared with reference values from the literature (Pearce et al., 1997; Gao et al., 2002). Accuracy, as the relative difference from reference values, was always better than 10%, and most elements plotted in the range $\pm 5\%$ (Table 2).

Table 2: Analyses by LA-ICP-MS of BCR-2G (United States Geological Survey) standard glass (in ppm). Comparison between literature data (Gao et al., 2002) and results from present study.

	BCR-2G	Std ⁽²⁾	BCR-2G-Standard	Std ⁽²⁾	Accuracies ⁽³⁾
	This study ⁽¹⁾		Gao et al. (2002)		
V	448	10.4	425	7	-5.46
Cr	16.14	0.8	17	2	5.06
Co	40.2	2.1	38	1	-5.79
Cu	19.6	1.2	18	1	-8.83
Ni	12.20	0.7	12.7	0.9	3.94
Rb	48.4	3.6	51	3	5.08
Sr	336	9.0	321	6	-4.76
Y	33.3	1.7	31	2	-7.32
Zr	158	6.4	167	8	5.56
Nb	11.8	0.8	10.9	0.6	-8.26
Sn	2.5	0.2	2.4	0.4	-4.17
Sb	0.53	0.09	0.51	0.87	-3.92
Ba	656	13	641	14	-2.41
Pb	11.6	1.13	10.9	0.5	-6.42
La	24.4	2.0	25	1	2.56
Ce	56	2.0	52	2	-7.52
Pr	6.8	0.5	6.3	0.4	-7.94
Nd	28.3	1.2	27	1	-4.81
Sm	6.7	0.3	6.3	0.5	-6.35
Eu	2.07	0.1	1.91	0.09	-8.38
Gd	5.95	0.5	6.5	0.6	8.46
Tb	0.87	0.08	0.95	0.07	8.00
Dy	5.48	0.6	6	0.4	8.67
Ho	1.09	0.04	1.2	0.07	9.17
Er	3.5	0.3	3.3	0.2	-5.76
Tm	0.49	0.05	0.46	0.04	-6.52
Yb	2.95	0.4	3.2	0.3	7.81
Lu	0.44	0.05	0.47	0.04	7.02

⁽¹⁾ Values determined by LA-ICP-MS. Mean values of 10 measures.

⁽²⁾ Std=Standard deviation

⁽³⁾ Accuracies were calculated on data from Gao et al. (2002).

4 Results and Discussion

4.1 Major Element Concentrations

Table 3 lists the composition of major element concentrations in wt% of oxides determined by EPMA-WDS. Each datum represents the mean value of three analyses recalculated to a total of 100%.

With the aim of identifying the alkali source used in the glass production, all data were plotted on the triangular diagram $\text{CaO} - \text{Na}_2\text{O} - (\text{K}_2\text{O} + \text{MgO})$ introduced by Gratuze (2004) (Figure 4a). In the diagram four areas corresponding to the four compositional groups distinguished by Hellemans, Cagno, Bogana, Janssens, & Mendera (2019) have been drawn.

All samples of the first group plot in the area N. 1 showing high content of Na_2O and low contents of K_2O and MgO , therefore they can be classified as “silica-soda-lime” glasses. Only two samples of the first group (US122-76 and US122-Y), showing lower Na_2O contents, plot near the area N. 1.

The finds of the second group plot in the area N. 2 of the diagram (Hellemans et al., 2019), therefore they can be classified as “vegetable silica-soda-lime” glasses of the Medieval period. Only the two samples SPO-61 and SPO-con plot on the border line of the N.3, therefore they can be classified as mixed alkali glass.

Table 3: Major and minor element concentrations in wt% of oxides determined by EPMA/WDS. *std*=standard deviation.

First group 4th–6th AD	SubGroup/Type	Color	SiO_2	TiO ₂	Al_2O_3	FeO	MnO	MgO	CaO	Na_2O	K_2O
US122-7	1A HIMT1	green	68.62	0.39	3.15	2.81	1.34	0.96	5.61	16.61	0.51
<i>std</i>			0.46	0.03	0.02	0.10	0.02	0.02	0.03	0.55	0.03
US122-9	1A HIMT1	green	68.65	0.57	3.47	3.16	1.58	1.37	5.07	15.57	0.55
<i>std</i>			0.84	0.06	0.07	0.15	0.06	0.03	0.01	0.46	0.01
US122-10	1A HIMT1	green	69.72	0.56	3.27	1.73	2.14	1.08	5.99	14.96	0.54
<i>std</i>			0.45	0.01	0.02	0.16	0.13	0.02	0.41	0.48	0.04
US122-77	1A HIMT1	green	67.02	0.54	2.72	2.10	0.86	1.20	5.55	19.61	0.41
<i>std</i>			0.21	0.07	0.11	0.12	0.01	0.05	0.14	0.29	0.04
US122-78	1A HIMT1	green	68.54	0.38	3.07	2.92	1.45	0.97	5.92	16.19	0.55
<i>std</i>			0.77	0.02	0.04	0.05	0.01	0.04	0.16	0.27	0.01
US124-50	1A HIMT1	green	67.11	0.64	3.21	2.17	0.97	1.53	4.85	19.12	0.40
<i>std</i>			0.59	0.06	0.18	0.07	0.03	0.02	0.04	0.21	0.02
US129-4	1A HIMT1	green	67.71	0.57	3.10	1.56	2.40	1.35	5.85	17.02	0.45
<i>std</i>			1.13	0.04	0.03	0.12	0.03	0.05	0.02	0.46	0.01
US122-76	1A HIMT1 altered?	green	74.17	0.60	3.73	3.41	1.61	1.40	5.16	8.52	1.39
<i>std</i>			1.63	0.05	0.07	0.16	0.06	0.04	0.12	0.43	0.05
US122-Y	1A HIMT1 altered?	green	74.21	0.48	3.29	3.20	1.59	1.05	6.11	8.86	1.20
<i>std</i>			0.30	0.04	0.05	0.12	0.04	0.01	0.09	0.39	0.04
US124-47	1B HIMT2	light green	66.58	0.23	2.73	0.66	0.76	1.35	7.57	19.55	0.56
<i>std</i>			0.16	0.03	0.02	0.03	0.06	0.03	0.11	0.24	0.00
US124-48	1B HIMT2	light green	73.99	0.22	2.55	0.69	0.75	0.88	7.72	12.35	0.84
<i>std</i>			0.68	0.03	0.33	0.06	0.05	0.01	0.33	1.60	0.26

US124-49	1B HIMT2	colourless	65.50	0.24	2.91	0.61	1.37	2.43	7.22	19.07	0.65
<i>std</i>			0.57	0.02	0.32	0.46	0.67	0.97	2.34	3.40	0.03
US122-75	Roman-Mn/ Levatine I ?	colourless	69.21	0.07	3.09	0.21	0.29	0.93	8.31	17.19	0.70
<i>std</i>			0.66	0.01	0.04	0.02	0.03	0.06	0.50	1.30	0.05
US122-bott	?	light green	66.89	0.22	2.38	0.70	1.09	2.05	4.02	22.57	0.29
<i>std</i>			0.70	0.04	0.05	0.02	0.06	0.05	0.04	0.03	0.04
Second group 12th–13th AD	SubGroup/Type	Color	SiO ₂	TiO ₂	Al ₂ O ₃	FeO	MnO	MgO	CaO	Na ₂ O	K ₂ O
US18-3	2A	green	67.90	0.21	4.61	1.43	0.65	2.63	6.34	13.23	3.00
<i>std</i>			1.26	0.01	0.20	0.03	0.04	0.19	0.08	0.93	0.08
SPO65	2A	light brown	69.49	0.19	3.20	1.49	0.86	3.07	7.63	11.54	2.54
<i>std</i>			1.58	0.02	0.41	0.21	0.10	0.15	0.68	1.90	0.04
SPO-66	2A	colourless	65.63	0.14	3.38	1.17	0.98	3.37	9.52	13.27	2.56
<i>std</i>			0.24	0.02	0.10	0.06	0.06	0.01	0.16	0.36	0.08
SPO-70	2A	light brown	66.57	0.24	4.83	1.71	0.84	2.62	6.46	13.77	2.95
<i>std</i>			0.14	0.03	0.15	0.05	0.03	0.08	0.10	0.10	0.02
SPO-71	2A	colourless	66.74	0.20	4.29	1.84	0.75	2.57	7.13	13.30	3.17
<i>std</i>			0.33	0.03	0.06	0.01	0.06	0.07	0.07	0.33	0.08
SPO-72	2A	light brown	65.32	0.12	3.27	1.27	0.83	5.20	9.54	12.63	1.83
<i>std</i>			0.25	0.01	0.01	0.10	0.02	0.02	0.03	0.06	0.05
SPO-Z	2A	colourless	69.83	0.14	3.39	0.96	0.67	2.24	6.49	13.26	3.01
<i>std</i>			0.31	0.04	0.09	0.12	0.02	0.05	0.11	0.38	0.06
US18-5	2B	colourless	66.62	0.17	2.22	0.54	0.56	4.98	7.20	16.09	1.62
<i>std</i>			0.70	0.02	0.18	0.04	0.03	0.35	0.72	1.40	0.10
SPO-73	2B	colourless	66.15	0.14	1.96	0.59	0.51	4.08	7.38	17.61	1.58
<i>std</i>			0.25	0.01	0.01	0.08	0.02	0.02	0.03	0.06	0.05
SPO-61	2C altered?	green	80.10	0.38	5.83	2.81	0.25	2.82	3.77	2.14	1.89
<i>std</i>			0.49	0.02	0.06	0.15	0.03	0.03	0.08	0.14	0.06
SPO-Con	2C altered?	green	80.14	0.34	4.47	2.28	1.27	3.57	3.43	2.97	1.54
<i>std</i>			2.22	0.03	0.56	0.16	0.09	0.28	0.18	0.21	0.10
SPO-69	Outlier	light brown	67.05	0.12	3.25	0.99	0.82	3.72	4.71	17.93	1.53
<i>std</i>			0.37	0.02	0.06	0.12	0.05	0.03	0.06	0.13	0.06

4.1.1 First Group

The samples of the first group show SiO₂ varying from 65.50 to 74.21 wt% and Na₂O from 12.35 and 22.57 wt% except for the two samples, US122-76 and US122-Y, showing lower Na₂O (from 8.52 to 8.86 wt%). The concentrations of MgO and K₂O are below 1.5 wt% for all glass fragments (Figure 4b) confirming the use of natron as a flux (Silvestri, 2008; Silvestri et al., 2005a, 2008). Only the two samples US122-bottle and US124-49 display MgO contents around 2 wt%, however, the high Na₂O and the low K₂O contents indicate the use of natron as flux (Figure 4a).

Due to the major element concentrations, the samples of the first group can be subdivided into two sub-groups and two outliers (Tables 1 and 3).

Figure 4: **a)** Ternary plot $\text{CaO} - \text{Na}_2\text{O} - \text{K}_2\text{O} + \text{MgO}$. The four main groups of historical glass are drawn on the plot: (1) mineral silica-soda-lime glass typical of the 1st millennium BCE–1st millennium AD; (2) vegetable silica-soda-lime glass of the medieval period; (3) mixed alkali glass of the late Bronze Age and medieval period; (4) potash glass of the medieval period (Gratuze, 2004; Hellemans et al., 2019). **b)** K_2O vs. MgO diagram: the square area discriminates the natron glasses.

The subgroup 1A includes nine samples (Tables 1 and 3) characterized by CaO around 5 wt% (from 4.86 to 6.11 wt%) and Al_2O_3 around 3 wt% (2.73–3.73 wt%). In addition, as regards FeO , these samples display values varying from 1.56 to 3.41 wt% and are typically dark green in colour.

The subgroup 1B includes three samples showing CaO varying from 7.22 to 7.72 wt% and Al_2O_3 from 2.55 to 2.91 wt%. They vary in colour from light green to colourless and display similar contents of FeO (around 0.6 wt%) and TiO_2 (around 0.2 wt%). Moreover, within this subgroup two samples (US124-47 and US124-49) contain MgO >1 wt%, the other one (US124-48) contains MgO < 1 wt%.

The samples US122-bott and US122-75 are outliers, indeed, US122-bott shows the lower contents of CaO (4.02 wt%) and Al_2O_3 (2.38 wt%), on the contrary US122-75 contains the higher content of CaO (8.31 wt%) and lower of FeO (0.21 wt%) and TiO_2 (0.07 wt%).

As regards the origin of the raw materials used for glass production, it is worth remembering that in the Roman and early medieval periods the glass was produced in primary production centers and then circulated to secondary glass workshops all over the Mediterranean. Previous studies (Freestone, Ponting, & Hughes, 2002; Freestone, 2005) have shown that the different concentrations of Na_2O , K_2O and MgO allow us to obtain information about the flux but do not allow us to trace the type of sand used as a raw material (Freestone, 1994). On the contrary, the contents of CaO and Al_2O_3 that are directly related to the quantities of feldspars and carbonate (calcite-aragonite) present in the sand, reflect both the possible compositional variations and the different proportions of the raw materials used in the different production centers. Therefore, the variable levels of CaO and Al_2O_3 suggest the use of different types of sands containing feldspar as impurities (Freestone, 2005). At the same time, high concentrations of FeO, MnO and TiO_2 reflect a silica source rich in accessory minerals (De Juan Ares, Vigil-Escalera Guirado, Caceres Gutiérrez, & Schibille, 2019b).

In terms of the major element concentrations generally observed in the glasses of the Roman and early medieval periods, Freestone's (2005) pioneering paper distinguished 5 natron-types glasses: Levantine I, Levantine II (produced using coastal sand of Siryo-Palestinian region), Roman blue-green, HIMT (High Iron, Manganese, Titanium) and Egyptian. The Levantine I glass was produced at Jalame (from 4th century AD) and Apollonia (from 6th to and 7th century AD), while the Levantine II glass was produced at Bet Eli'ezer, near Hadera, Israel during the 8th century (Brems et al., 2018; De Juan Ares et al., 2019b; Phelps et al., 2016). Then later the Levantine group (Gliozzo, Braschi, Giannetti, Langone, & Turchiano, 2019) has been related to the Foy's "Group 3.3" (Foy, Picon, Vichy & Thirion-Merle, 2003). Recent studies (Jackson & Paynter, 2016; Schibille, Sterrett-Krause, & Freestone, 2017) allowed the separation of the main glass groups proposed by Freestone (2005) into sub-types. The Roman glass was divided in the Roman-Sb, Roman-Mn and Roman mixed Sb-Mn, this last probably obtained from the recycling of the two end members (Jackson & Paynter, 2016; Schibille et al., 2017). Similarly, the HIMT glass has been divided into numerous sub-types. Gliozzo et al. (2019) distinguished the HIMT1 corresponding to Foy's "Group 1" (Foy et al., 2003), and the HIMT2 glasses corresponding to Foy's "Group 2" (Foy et al., 2003). Ceglia et al. (2015), Ceglia, Cosyns, Schibille, & Meulebroeck (2019) and De Juan Ares, Schibille, Molina Vidal, & Sanchez De Prado (2019a), considering the different FeO/ TiO_2 and FeO/ Al_2O_3 ratios, sub-divided the HIMT1 in HIMTa dated to the 4th and 5th century AD and HIMTb (characterised by higher Fe) attributed to the beginning of the 5th century AD. In addition, to distinguish the high lime HIMT glass, widely distributed in the late 6th–7th century AD, Ceglia et al. (2015) introduced the acronym HLIMT corresponding to some glasses of Foy's "Group 2" (Foy et al., 2003). Moreover, within the Foy's "Group 2", De Juan Ares et al. (2019b) distinguish the Magby subgroup, a plant ash glass characterized by elevated magnesia levels dated to the 6th–7th century AD. As regards the production place of HIMT glasses, it has been hypothesized to have an Egyptian origin (Ceglia et al., 2015; Gliozzo et al., 2019). In the end, as for the glass produced in Egypt, there are other two groups: Egypt I (8th century) and Egypt II (8th–9th century AD) (De Juan Ares et al., 2019b; Schibille et al., 2017).

As concerns our samples their provenance has been investigated comparing the results with the significant chemical signatures distinctive of the previous groups. In the diagram $\text{Al}_2\text{O}_3/\text{SiO}_2$ vs. $\text{TiO}_2/\text{Al}_2\text{O}_3$ proposed by Schibille et al. (2017) (Figure 5), the glasses of the sub-group 1A plot near the HIMT1 type. The two samples US122-76 and US122-Y display concentrations coherent with the HIMT1 group. However, this attribution is uncertain, indeed, as previously stated, these glasses show a very low content of Na_2O and a high content of SiO_2 (around 74 wt%) (Table 3), probably as a result of a superficial de-alkalinisation (Barbera et al., 2012; Huisman, Pols, Joosten, van Os, & Smit, 2008; Silvestri, Molin,

Salviulo, 2005b). The glasses of the subgroup 1B show chemical characteristics that can be related to the HIMT2 type.

Figure 5: Binary diagram $\text{Al}_2\text{O}_3/\text{SiO}_2$ vs. $\text{TiO}_2/\text{Al}_2\text{O}_3$ for the samples of the first group.

Bibliographic data from: Brems et al., 2018; Ceglie et al., 2015, 2019; De Juan Ares et al., 2019a; Foy et al., 2003; Gliozzo et al., 2019; Phelps et al., 2016; Schibille et al., 2017, 2019.

Indeed, differing from the HIMT1 glasses, the samples show lower contents of FeO , TiO_2 and Mn . Furthermore, CaO and Al_2O_3 contents vary around 7–8 wt% and 2.5–3 wt% respectively. As for the two outliers, the sample US122-bott shows similarity with the 1B group, whereas the sample US122-75 plots in the area of Levantine glass.

With the aim of evaluating the attribution to the Levantine I or Levantine II, the US122-75 glass has been compared with the bibliographic data (Brems et al., 2018; Ceglie et al., 2015; Gliozzo et al., 2019; Phelps et al., 2016; Schibille et al., 2017) by using the diagram proposed by Phelps et al. (2016). The diagram (Figure 6) highlights the similarity between the US122-75 sample and the Jalame-Levantine I source dating to the 4th century AD. To confirm these results the samples have been compared with the natron-based glass reference groups using three ternary diagrams (Figure 7) proposed by Gliozzo et al. (2019). The three diagrams of Figure 7 confirm that the samples of the group 1A plot near the HIMT1 and Foy1 groups coherently with a possible date of the 5th century AD. The three samples of the group 1B show similarity with the HIMT2 and Foy2 groups. The samples US122-bott and US122-75 could be respectively of HIMT1 and Levantine provenance. However, US122-bott sometimes plots between HIMT1 and HIMT2 groups, therefore, it is difficult to establish an unambiguous assignment of its provenance. Likewise, the sample US122-75 displays (see Figure 7) slight chemical differences with Levantine glass and some similarities with the Mn-Roman group dating to the 4th century AD (Schibille et al., 2017). This result illustrates the difficulty in assigning a univocal attribution to the find while it dates in both cases to the 4th century AD.

Figure 6: $\text{Na}_2\text{O}/\text{SiO}_2$ vs. $\text{CaO}/\text{Al}_2\text{O}_3$ binary plot. The sample US122-75 has been compared with the three Levantine known primary production sites: Levantine I (Jalame 4th century AD; and Apollonia 6th–7th century AD) and Levantine II (Bet Eli’ezer 8th century AD).

Figure 7: Ternary plots for the samples of the first group compared with the bibliographic data of natron-type glasses distributed in the Mediterranean area and Europe for most of the first millennium AD. Bibliographic data from: Brems et al., 2018; Ceglie et al., 2015, 2019; De Juan Ares et al., 2019a; Foy et al., 2003; Gliozzo et al., 2019; Phelps et al., 2016; Schibille et al., 2017, 2019.

4.1.2 Second Group

The samples of the second group contain MgO and K₂O at levels higher than 1.5 wt% confirming that they are typically plant ash glasses (Figures 4a and 4b). They can be subdivided into three sub-groups and an outlier. The subgroup 2A includes seven samples characterized by Na₂O varying from 11.54 to 13.77 wt%, K₂O from 1.83 to 3.17 wt% and MgO from 2.24 to 3.37 wt% with a peak of 5.20 wt% for the sample SPO-72.

The other two samples, US18-5 and SPO-73, form the sub-group 2B. They show higher Na₂O contents (respectively 16.09 and 17.61 wt%), K₂O around 1.5 wt% and MgO from 4.08 to 4.98 wt%. The SPO-69 sample is chemically very similar to the sub-group 2B but shows a lower CaO content, therefore it can be considered an outlier. The other two samples (SPO-con, SPO-61) form the sub-group 2C, indeed, they contain a very low concentration of Na₂O (2.14–2.97 wt%) and very high content of SiO₂ (around 80 wt%) (Figure 4a, Table 3). These characteristics allow us to hypothesise that the samples are interested by a superficial alteration with an important de-alkalinisation (Barbera et al., 2012; Huisman et al., 2008; Silvestri et al., 2005b). In addition, they display a high FeO content (>2 wt%) and are green coloured.

Considering their archaeological context (see Materials paragraph), the samples of this group have been dated between the 12th and the 13th century AD. As stated above, numerous studies (De Juan Ares et al., 2018; Henderson, McLoughlin, & McPhail, 2004; Henderson, Chenery, Faber, & Kröger, 2016; Phelps et al., 2016; Schibille et al., 2019) showed that, towards the end of the first millennium AD, the fluxing agents changed gradually from natron to plant ashes, rich in potassium in central Europe and rich in sodium in the eastern Mediterranean. The chemical results of the samples presented here indicate that the samples of the second group can be classified as “vegetable silica soda lime glass” (see Figure 4a). However, within the soda-rich plant ash glasses the different magnesium and potassium contents have been associated with different glassmaking traditions (De Juan Ares et al., 2018). The plant ash glasses, considering their chemical features, can be subdivided into Mediterranean (Egypt, Syria-Palestine) and Mesopotamian (De Juan Ares et al., 2018). Schibille et al. (2019) distinguished four compositional plant ash categories that date to the second half of 10th century AD and the early 11th century AD: Levantine, Egyptian1, Egyptian2 and Mesopotamian.

With the aim of assigning the possible source provenance, the results of our samples were compared with the significant chemical signatures distinctive of the previous groups using two binary plots Al₂O₃/SiO₂ vs CaO/Al₂O₃ and MgO/CaO vs. Al₂O₃ (Figure 8). The studied samples display significant differences with the Levantine and Egyptian-Syrian glasses collected from the urban centers (Beirut, Damasco Cairo) dating between the 9th and 14th century (Henderson et al., 2004, 2016).

In general, the samples of the 2A subgroup, despite demonstrating higher Al contents, plot near the Egyptian 2 plant ash glass (Schibille et al., 2019). In addition the samples US18-3, SPO-70 and SPO-71 can be distinguished for their higher Al concentrations.

The chemical characteristics of this group can be related to those of the HMEIG (High-Mg Early Islamic glass) as defined by Angelini et al. (2019), i.e. plant ash glasses dated in a span of time from 840 to 1000 AD.

The samples of the 2B subgroup, based on their chemical features, can be related to the Mesopotamian glasses (Schibille et al., 2019) as confirmed by the MgO concentrations reaching higher than 3.5 wt%, the value suggested by Schibille et al. (2019) to identify a Mesopotamian provenance. The 2C glasses are clearly different from all bibliographic data but, as stated above, they are particularly altered. In the end, the outlier SPO-69 is similar to the 2A group but it differs for the MgO/CaO ratio.

Figure 8: a) $\text{Al}_2\text{O}_3/\text{SiO}_2$ vs. $\text{CaO}/\text{Al}_2\text{O}_3$; b) MgO/CaO vs. Al_2O_3 binary plots. The samples of the second group have been compared with the bibliographic data: *Schibille et al., 2019; ** Henderson et al., 2004 (glasses collected in the Islamic site of Tell Fuhkhkhar, Jordan 11th century AD); Henderson et al., 2016 (glasses deriving from urban centres in the area between Egypt and Syria dating between 9th to 14th century AD).

4.2 Trace Element Concentrations

Table 4 lists the composition of trace element concentrations in ppm determined by LA-ICP-MS. Each datum represents the mean value of three analyses. Only two samples were not analysed (US124-48 for the first group and US18-3 for the second group) due to their small size and rough surface.

Table 4: Trace element concentrations in ppm determined by LA-ICP-MS.

First group	SubGroup/ Color Type	V	Cr	Co	Cu	Ni	Rb	Sr	Y	Zr	Nb	Sn	Sb	Ba	Pb
US122-7	1A HIMT1 green	108.9	73	10.6	97	38.8	6.57	468	17.66	249	6.18	3.06	1.40	245	42
<i>std</i>		5.42	1.23	0.08	5.12	2.34	0.06	11.2	0.18	3.5	0.08	0.41	0.14	9.8	4.4
US122-9	1A HIMT1 green	92.7	84	12.0	83	42.6	6.12	392	16.81	285	7.19	1.59	0.84	173	34
<i>std</i>		4.72	5.04	0.07	4.08	2.54	0.05	12.0	0.15	4.7	0.08	0.11	0.09	11.2	3.3
US122-10	1A HIMT1 green	44.3	97	5.88	8.14	10.1	5.08	438	12.21	361	8.65	1.71	0.71	196	9.64
<i>std</i>		2.72	4.50	0.06	1.13	0.94	0.05	14.6	0.13	5.2	0.17	0.10	0.08	10.4	1.3
US122-77	1A HIMT1 green	92	68	8.07	82	32.7	5.20	411	14.02	204	5.14	2.15	1.53	204	26.5
<i>std</i>		4.94	5.32	0.85	1.96	1.18	0.08	4.02	1.11	8.72	0.18	0.03	0.44	0.25	0.54
US122-78	1A HIMT1 green	86	64	9.35	79	33.3	5.06	430	14.65	232	5.80	2.40	1.16	219	27.8
<i>std</i>		3.72	3.50	0.72	2.13	1.44	0.35	12.6	0.33	6.2	0.17	0.10	0.08	10.4	1.3
US124-50	1A HIMT1 green	102	83	11.1	83	44.4	6.80	373	14.80	259	7.04	1.23	n.d.	184	17.6
<i>std</i>		0.98	10.38	0.88	0.23	0.72	0.62	9.81	0.62	1.13	0.04	0.06	-	2.08	1.50
US129-4	1A HIMT1 green	70	112	14.4	58	19.4	6.06	511	12.06	332	7.76	5.32	2.62	479	64
<i>std</i>		3.27	2.88	0.69	2.09	1.66	0.29	11.6	0.27	4.2	0.15	0.16	0.09	9.3	1.2
US122-76	1A HIMT1 green altered?	91	74	9.75	80	32.7	7.82	453	15.66	235	5.59	1.98	1.34	224	28.5
<i>std</i>		6.25	4.23	0.71	5.04	2.74	0.36	18.3	0.29	11.2	0.91	0.12	0.07	12.5	3.8
US122-Y	1A HIMT1 green altered?	88	93	13.2	77.2	44.3	8.20	394	15.98	276	8.24	2.14	1.08	170	33.2
<i>std</i>		7.32	6.42	1.62	6.14	3.61	0.74	15.2	1.23	15.2	4.03	0.24	0.05	14.7	4.6
US124-47	1B HIMT2 colourless	30.0	n.d.	10.8	43.2	18.6	9.20	756	7.96	98	3.46	8.98	97	270	60
<i>std</i>		2.80	-	1.05	6.16	2.47	1.13	18.36	0.91	10.82	0.23	0.72	9.15	15.17	7.45
US124-49	1B HIMT2 colourless	35.2	19.1	23.8	52	35.5	9.05	820	9.65	102	3.53	6.60	60	269	110
<i>std</i>		2.30	1.23	1.40	0.45	6.21	0.30	16.59	0.04	0.31	0.11	0.08	10.14	16.07	15.92
US122-75	Mn-Roman/colourless Levantine I?	14.8	16.0	3.03	16.5	n.d.	11.6	455	6.81	53	2.39	2.45	0.78	257	66
<i>std</i>		1.60	1.34	0.28	1.13	-	1.13	26.48	0.28	3.82	0.30	0.28	0.09	17.62	3.51
US122-bott	outlier light green	29.8	23.6	6.22	31.3	19.5	4.37	440	6.88	81	2.27	1.08	0.79	176	9.85
<i>std</i>		0.93	1.05	0.47	1.39	2.03	0.43	19.38	0.37	6.62	0.34	0.14	0.13	11.35	1.47
First group	SubGroup/ Color Type	La	Ce	Pr	Nd	Sm	Eu	Gd	Tb	Dy	Ho	Er	Tm	Yb	Lu
US122-7	1A HIMT1 green	16.2	18.8	3.72	16.8	3.17	0.89	3.48	0.54	3.27	0.66	1.81	0.29	2.18	0.24
<i>std</i>		0.18	1.29	0.15	1.15	0.39	0.11	0.13	0.02	0.22	0.05	0.22	0.04	0.02	0.04
US122-9	1A HIMT1 green	16.2	20.2	3.91	15.9	4.09	0.79	3.38	0.57	3.20	0.58	1.37	0.28	2.13	0.30
<i>std</i>		0.25	1.57	0.14	1.26	0.28	0.10	0.12	0.02	0.21	0.04	0.21	0.03	0.02	0.03
US122-10	1A HIMT1 green	11.7	28.6	3.23	11.2	2.36	0.44	3.11	0.46	2.72	0.59	0.95	0.16	1.12	0.23

<i>std</i>			0.27	2.05	0.22	1.23	0.24	0.08	0.13	0.03	0.27	0.03	0.09	0.01	0.03	0.04
US122-77	1A HIMT1	green	13.1	17.8	3.32	12.8	3.01	0.80	2.18	0.35	2.38	0.51	1.87	0.16	1.36	0.27
<i>std</i>			0.79	1.05	0.17	1.85	0.54	0.33	0.37	0.01	0.25	0.10	0.21	0.02	0.32	0.04
US122-78	1A HIMT1	green	14.6	17.5	3.49	14.3	2.59	0.61	2.95	0.48	2.50	0.64	1.64	0.16	1.88	0.24
<i>std</i>			0.27	2.05	0.22	1.23	0.24	0.08	0.13	0.03	0.27	0.03	0.09	0.01	0.03	0.04
US124-50	1A HIMT1	green	14.4	18.9	3.67	15.4	3.24	1.09	3.07	0.52	1.68	0.53	1.99	0.35	1.54	0.22
<i>std</i>			0.35	1.43	0.38	0.87	0.43	0.18	0.30	0.05	0.12	0.01	0.32	0.06	0.18	0.01
US129-4	1A HIMT1	green	11.0	22.9	2.56	10.6	1.62	0.83	3.34	0.25	1.22	0.38	1.50	0.24	1.86	0.37
<i>std</i>			0.29	1.95	0.22	1.24	0.12	0.06	0.11	0.02	0.21	0.04	0.08	0.01	0.02	0.04
US122-76	1A HIMT1	green	14.6	18.2	3.41	14.1	2.64	0.88	3.67	0.53	3.38	0.50	1.17	0.22	2.27	0.30
<i>std</i>	altered?		0.23	1.35	0.16	1.27	0.11	0.04	0.12	0.04	0.18	0.05	0.07	0.04	0.17	0.03
US122-Y	1A HIMT1	green	16.0	20.7	3.84	16.8	3.62	0.76	3.18	0.43	3.51	0.57	1.56	0.26	1.62	0.25
<i>std</i>	altered?		1.32	4.37	0.14	1.38	0.12	0.03	0.71	0.03	0.98	0.04	0.37	0.04	0.12	0.02
US124-47	1B HIMT2	colourless	7.85	15.3	1.92	8.89	1.96	0.32	2.25	0.27	1.03	0.27	0.97	0.22	n.d.	0.30
<i>std</i>			1.19	1.72	0.06	0.81	0.14	0.03	0.14	0.02	0.08	0.03	0.07	0.01	-	0.03
US124-49	1B HIMT2	colourless	9.23	14.9	2.02	8.75	1.68	0.39	1.86	0.17	1.32	0.25	0.97	0.18	1.41	0.30
<i>std</i>			0.05	0.83	0.30	0.81	0.27	0.04	0.37	0.03	0.21	0.02	0.06	0.01	0.02	0.02
US122-75	Mn-Roman/	colourless	5.99	12.2	1.34	6.41	1.48	0.65	1.49	0.15	0.75	0.32	0.68	n.d.	0.68	0.15
<i>std</i>	Levantine I?		0.15	0.62	0.07	0.19	0.06	0.03	0.22	0.01	0.06	0.03	0.08	-	0.04	0.02
US122-bott	outlier	light green	8.46	11.1	2.13	7.13	1.87	0.41	1.03	0.22	1.11	0.31	1.38	0.16	1.83	n.d.
<i>std</i>			0.35	0.88	0.16	0.94	0.26	0.03	0.15	0.03	0.13	0.00	0.18	0.02	0.06	-

Second group	SubGroup/ Type	Colour	V	Cr	Co	Cu	Ni	Rb	Sr	Y	Zr	Nb	Sn	Sb	Ba	Pb
SPO65	2A	light brown	19.1	59.6	19.5	128	15.7	20.4	588	6.25	100	4.67	29.7	9.50	179	119
<i>std</i>			2.06	4.34	1.23	11.06	1.31	2.08	14.82	0.82	10.52	0.13	3.16	0.97	9.14	9.23
SPO-66	2A	colourless	27.3	16.6	9.88	122	16.6	17.3	724	6.43	94	5.37	3.87	26.0	159	68
<i>std</i>			1.96	1.84	0.98	9.06	1.46	1.80	31.82	0.62	8.42	0.92	0.46	1.27	10.25	5.13
SPO-70	2A	light brown	34.4	25.1	15.3	179	21.3	17.5	544	9.19	86	6.14	4.03	2.13	179	50.0
<i>std</i>			2.31	1.22	0.64	11.45	2.20	1.33	18.23	0.65	9.26	0.71	0.38	0.21	11.12	2.33
SPO-71	2A	colourless	32.3	27.7	15.1	296	19.0	18.0	543	8.54	94	6.35	7.37	3.35	175	49.5
<i>std</i>			2.11	2.12	1.45	12.36	1.61	1.98	12.62	0.83	9.32	0.24	0.76	0.31	12.24	6.47
SPO-72	2A	light brown	22.9	18.8	7.38	18.3	21.7	7.64	642	7.98	125	4.63	2.87	1.14	123	336
<i>std</i>			1.72	1.21	0.33	14.08	2.43	0.63	3.56	0.12	0.33	0.52	0.37	0.02	6.59	11.42
SPO-Z	2A	colourless	19.5	21.0	16.3	67	10.8	20.1	538	6.46	86	4.38	16.87	5.34	158	113
<i>std</i>			2.02	1.72	1.98	4.26	0.99	1.33	21.72	0.42	8.35	0.36	1.41	0.64	13.28	6.33
US18-5	2B	colourless	18.3	18.5	11.6	26.9	7.98	13.7	530	6.08	143	3.94	3.45	2.08	110	30.9
<i>std</i>			0.35	2.55	0.11	0.08	0.57	0.64	5.75	0.41	6.72	0.03	0.37	0.17	2.20	0.91

SPO-73	2B	colourless	12.1	29.2	5.76	65	10.9	10.8	497	4.52	78	2.57	5.79	1.31	281	42.2
<i>std</i>			1.31	3.22	0.43	3.46	1.10	1.23	15.83	0.37	6.26	0.21	0.61	0.14	12.73	0.33
SPO-61	2C altered?	green	27.0	27.9	6.00	22.8	21.1	9.37	665	10.4	124	6.17	2.78	0.52	152	283
<i>std</i>			2.76	1.93	0.64	2.75	1.47	1.22	22.79	0.76	8.46	0.63	0.32	0.04	13.41	14.05
SPO-Con	2C altered?	green	26.0	29.9	7.03	96	12.8	6.90	274	8.05	157	5.35	9.84	0.93	111	86
<i>std</i>			1.64	2.67	0.73	11.22	1.32	0.76	18.64	0.65	13.59	0.57	0.62	0.06	10.52	9.17
SPO-69	Outlier	light brown	36.2	24.1	8.38	133	16.3	12.9	264	7.76	78	5.46	3.35	0.42	160	57
<i>std</i>			2.38	1.21	1.04	12.26	1.24	1.23	13.26	0.24	7.76	0.43	0.32	0.03	9.79	7.33

Second group	SubGroup/ Type	Colour	La	Ce	Pr	Nd	Sm	Eu	Gd	Tb	Dy	Ho	Er	Tm	Yb	Lu
SPO65	2A	light brown	9.19	17.9	2.01	8.39	1.36	0.28	1.83	0.15	1.37	0.20	0.70	0.07	0.55	0.19
<i>std</i>			0.96	1.88	0.14	0.13	0.15	0.02	0.15	0.01	0.05	0.03	0.10	0.001	0.01	0.02
SPO-66	2A	colourless	11.6	24.2	2.65	10.5	2.44	0.49	1.37	0.23	1.55	0.25	1.19	0.12	0.40	0.06
<i>std</i>			0.96	1.62	0.23	1.13	0.14	0.02	0.16	0.02	0.06	0.02	0.12	0.01	0.01	0.003
SPO-70	2A	light brown	12.4	23.6	2.65	10.0	1.96	0.53	1.46	0.24	2.26	0.37	0.58	0.09	1.25	0.10
<i>std</i>			0.58	1.31	0.16	1.04	0.11	0.03	0.16	0.02	0.13	0.02	0.02	0.01	0.07	0.01
SPO-71	2A	colourless	12.3	24.8	3.01	11.3	3.11	0.67	1.87	0.23	1.77	0.31	0.61	0.14	0.83	0.16
<i>std</i>			0.87	1.68	0.25	0.14	0.15	0.04	0.17	0.02	0.07	0.02	0.06	0.01	0.03	0.02
SPO-72	2A	light brown	11.7	22.8	2.61	12.0	2.01	0.43	2.27	0.17	1.07	0.26	0.90	0.20	1.07	0.06
<i>std</i>			0.06	0.53	0.28	0.27	0.29	0.12	0.11	0.04	0.16	0.08	0.32	-	0.01	0.01
SPO-Z	2A	colourless	8.80	18.6	2.17	8.11	1.00	0.36	0.62	0.17	0.74	0.23	0.49	0.13	1.08	
<i>std</i>			0.72	1.44	0.18	0.62	0.11	0.03	0.07	0.01	0.03	0.02	0.03	0.01	-	0.01
US18-5	2B	colourless	8.77	17.0	2.10	8.88	1.70	0.27	1.95	0.25	1.63	0.26	0.80	n.d.	0.59	n.d.
<i>std</i>			1.22	2.53	0.27	0.91	0.06	0.02	0.17	0.04	0.06	0.04	0.12	-	0.01	-
SPO-73	2B	colourless	6.46	13.7	1.80	6.71	1.42	n.d.	1.12	0.12	0.84	0.22	n.d.	0.18	n.d.	0.13
<i>std</i>			0.42	0.74	0.19	0.82	0.12	-	0.13	0.01	0.04	0.02	-	0.02	-	0.01
SPO-61	2C altered?	green	11.8	23.3	2.56	11.7	2.54	0.66	2.20	0.32	1.42	0.40	0.75	0.10	1.34	0.23
<i>std</i>			0.73	2.39	0.21	1.04	0.17	0.05	0.18	0.01	0.08	0.03	0.07	0.01	0.04	0.02
SPO-Con	2C altered?	green	8.59	19.7	2.91	11.5	1.02	0.74	2.34	0.17	1.44	0.35	1.52	n.d.	1.52	n.d.
<i>std</i>			0.83	2.22	0.19	1.07	0.12	0.06	0.16	0.02	0.09	0.02	0.08	-	0.06	-
SPO-69	Outlier	light brown	9.42	19.5	2.34	8.68	2.45	0.83	1.56	0.24	1.16	0.26	0.89	n.d.	1.71	0.10
<i>std</i>			0.86	2.46	0.24	0.97	0.24	0.11	0.11	0.02	0.12	0.02	0.07	-	0.11	0.01

The trace element concentrations allow a complete chemical characterization of glasses. In addition, they are very useful to: i) identify the recycling, ii) individuate the colouring or decolouring agents, iii) define the attribution to a compositional glass group. Numerous studies demonstrated that the recycling of glass was a widespread practice since the Roman times (Freestone, 2015; Gliozzo et al., 2019; Schibille et al., 2017). Nonetheless, the identification of recycled glass is often a difficult task, especially if we consider that the primary glass was sometimes coloured by the recycling of other glass fragments. Gliozzo et al. (2019) suggest that the contents of Cu, Sb and Pb are possible indicators of recycling, the recommended limits are: natural <100 ppm, recycled >100, intentionally added >1000.

As regards the glasses presented here, in the first group all samples show Cu, Sb and Pb contents lower than 100 ppm, highlighting the absence of recycling (Table 4). Only the sample US124-49 shows Pb=110 ppm, however, considering the standard deviation (std=15.92) we can also assume, the absence of recycling for it.

In contrast, many glasses of the second group show signs of recycling. Indeed high Cu contents (from 122 ppm – to 296 ppm) have been measured in five samples: SPO-65, SPO-66, SPO-69 SPO-70 and SPO-71 and similarly high Pb contents (from 119 ppm – to 336 ppm) have been measured in SPO-65, SPO-72, and SPO-61.

As concerns the colour, the vitreous finds of the 1A subgroup are all green coloured. Previous studies (Barca et al., 2016, 2019; Basso et al., 2014) showed that, in Roman times, glass was coloured by introducing into the frit transition metals such as cobalt as oxide for dark blue, iron oxide for green, blue or amber and copper as oxide (CuO) for turquoise, blue or green (Paynter & Kearns, 2011). However, our samples display very low content of transition metals (Co and Cu) showing concentrations lower than 100 ppm (Table 4). However, these glasses contain high iron concentrations ($\text{FeO} > 2 \text{ wt\%}$), which can be considered the responsible of the green colour.

The low content of antimony measured in both glass groups leads us to consider that the decolouring agent used was only the manganese. The addition of Mn as decolourant is frequently associated with Co, Cu, Ni, Sb, Sn and Pb. However, the low values of these elements can be attributed to background concentrations in the sand's raw materials (Brems et al., 2018) with an exception made for the sporadic increase observed for Cu and Pb in some samples of the second group correlated to the recycling.

In particular, the trace element concentrations of the first group confirm the distinction in two compositional sub-groups, 1A and 1B. The contents of Nb, Y, Zr, and TiO_2 have been compared in binary plots. The glasses of the subgroup 1A (HIMT1) show high contents of these elements in line with a sand batch that was rich in heavy minerals and/or clays (Figure 9) (Brems et al., 2018; De Juan Ares et al., 2019a; Freestone et al., 2002; Gliozzo et al., 2019). The samples US122-Y and US122-76, considered of doubtful attribution because of their low Na_2O contents, show compositions in agreement with the other samples of the sub-group 1A, thereby confirming the hypothesis that these glasses were subjected to only by a superficial de-alkalinisation.

The samples of the subgroup 1B (US122-47 and US122-49), compared to the subgroup 1A, display lower values of Nb, Y, Zr, and TiO_2 as expected for HIMT2 glasses (Schibille et al., 2017) (Figures 9). As for the two outliers, the sample US122-75 shows very low contents of Nb, Y, Zr, and TiO_2 and high Sr (455 ppm) consistent with a possible Roman-Mn/Levantine I provenance (Phelps et al., 2016) and the sample US122-bott displays variable contents of heavy metals making it impossible to assign a match with the known glass sources.

As stated above, the vitreous finds of the second group show signs of recycling, a procedure widely spread in the Egyptian plant ash glasses (Schibille et al., 2019). In general, the three glass groups can be distinguished by their trace element contents. These differences are highlighted by plotting the trace element average values of the three sub-groups and the outlier in a spider diagram (Figure 10). The plot highlights the differences/similarities among the subgroups. The SPO-69 glass shows a trend similar to the 2A group, from which it differs for the low content of Sr (264 ppm), consistent with the low CaO content, thereby confirming the possibility that it is an outlier produced using different lime rich sands.

5 Conclusions

The chemical characterization carried out on vitreous specimens allowed us to identify the type of raw materials used for their production.

The major and trace element concentrations confirmed the chronological distinction of the samples into two compositional groups. In addition, the glasses of the first group were subdivided into two subgroups 1A and 1B attributable to the HIMT1 and HIMT2 types respectively, with two outliers. The outlier US122-75 is particularly interesting as despite the dubious attribution (Jalame or Roman-Mn), the chemical features allows a consideration of it as the older find of the first group, dating to the 4th century AD.

Figure 9: Binary diagrams for the first group glasses: a) Zr ppm vs. Y ppm; b) Zr ppm vs. Nb ppm c) Zr ppm vs. TiO₂ wt%.

Figure 10: Spider diagram showing the average of trace element patterns of the second group.

The vitreous finds of the second group are plant ash glasses dated on the base of archaeological context to the 12th–13th century AD. Notwithstanding, their attribution to known glass sources has been complicated by recycling processes, their chemical characteristics being similar to the HMEIG type as defined by Angelini et al. (2019) i.e. High-Mg Early Islamic: plant ash glasses dated from 840 to 1000 AD. However, considering that on the typological attribution the studied finds date to the 12th–13th century AD, we can hypothesise that the HMEIG glass production continued until 1100–1200 AD.

Both groups include some vitreous finds characterised by very low content of Na₂O attributable to an intense superficial de-alkalinisation process. This hypothesis is supported by the macroscopic and microscopic observation of the bad state of preservation of glasses buried in the soil for a long time, and by the trace element concentrations determined using the LA-ICP-MS that permits the investigation of a thickness of about 70/80 mm. Because the sum of the measured elements is recalculated to 100 wt%, the loss of Na₂O results automatically in an increase in the other elements. Therefore, in these glasses the high SiO₂ contents are the result of closed-sum effects as confirmed by comparing the recalculated data with those determined by EPMA-WDS.

It is also interesting to highlight that all the vitreous fragments of the first group (4th–6th century AD) were recovered together with coal residues and metallic wicks inside a trench excavated by cutting an *opus tessellatum* mosaic floor. The absence of combustion of the trench walls allows us to hypothesise that symbolic ceremonies were executed by burying the sacred objects after the extinction of a ritual fire. The deposit of different types of artifacts inside the religious buildings when the edifices were subjected to restorations or rededication, was a practice well known in northern Italy from the late-medieval to modern times (Gavagnini & Roascio, 2006). However, the finding of a whole vitreous jar (dated 6th–7th century AD) in a trench under the floor of the Santa Maria church in Vezzano Ligure (La Spezia), indicates that the propitiatory offering for the floor construction was a ritual practiced since the Byzantine times. The discovery of vitreous fragments used during the sacraments of the baptism under the floor of Cosenza Cathedral, clearly represents the first case of the execution of this ritual in Calabria.

The finding of a hidden deposit of sacred objects that are no longer usable inside the synagogue of Bova (Reggio Calabria) is an exception because this evidence refers to a different type of religious ceremony, that is to say the Jewish *genizah* (Costamagna, 1991). Therefore the Cosenza Cathedral case study throws light on the possibility of the existence of this practice in the Late Roman period in the Calabrian archaeological context.

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