

PROTOHISTORIC VITREOUS MATERIAL FROM SOUTHERN ITALY: CHEMICAL CHARACTERIZATION AND TRACE ELEMENTS STUDY

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INTRODUCTION

This work was focused on the archaeometrical study of protohistoric glass coming from several Bronze and Iron Age sites of southern Italy. The aim was the definition of the raw materials (vitrifying, fluxes, colorants) and of the technology employed for the production of these artifacts, in order to follow their evolution in time. A large amount of archaeological and archaeometrical data about glass of northern Italy is available in literature (*e.g.*, Arletti *et al.*, 2009, 2011a; Towle *et al.*, 2001); on the contrary only a few samples from southern Italy have been analyzed up to now (Angelini *et al.*, 2003). The results of an archaeometrical investigation relative to 132 glass samples coming from Pompei, Vivara, Murgia Timone, Broglio di Trebisacce, Lipari, Roca Vecchia sites, dated from the Early Bronze Age (18th century BC) to the Final Bronze Age (10th century BC), and from Francavilla Marittima, Torre Galli, Cuma, Capua, Sarno and Amendolara, dated to the Iron Age (10th-6th century BC), are here presented. Moreover, 11 samples coming from the Villanovan site of Bologna Fair, dated to the 8th century BC, were taken into account for a comparison.

EXPERIMENTAL METHODS

This study was carried out with a multi-technique approach. The good state of preservation of the glass allowed the removal of small chips of only few hundreds μm^3 . The sampled material was embedded in epoxy resin and polished with diamond paste from 6 to 1 μm for the analyses with Environmental Scanning Electron Microscopy (ESEM), Electron Microprobe (EMPA) and laser ablation inductively coupled plasma mass spectrometry (LA-ICPMS). Finally, isotopic analyses allowed the determination of the Sr and Nd isotopic signatures.

Environmental Scanning Electron Microscopy (ESEM-EDS)

Back-scattered electron images (BSE) and EDS spectra were collected on a low-vacuum ESEM Quanta 200, equipped with an Oxford energy dispersive spectrometer, at the Centro Interdipartimentale Grandi Strumenti of the University of Modena and Reggio Emilia. The analyses were performed using an acceleration voltage of 20 kV and a working distance of 11 mm. The BSE images were collected on all the samples to check the glass matrix homogeneity. EDS analyses were run to obtain qualitative and semi-quantitative chemical analyses. For limited space reason this results will not be discussed in this report.

Electron MicroProbe Analysis

The chemical analyses of major and minor elements (Si, Ti, Al, Fe, Mn, Mg, Ca, Na, K, P, S, Cl, Cr, Co, Cu, Sn, Sb, and Pb) were carried out using a Cameca SX 50 microprobe equipped with four scanning wavelength-dispersive spectrometers (WDS). The reference Smithsonian glass A standard (Jarosewich, 2002) was employed as primary reference sample. The analyses were performed operating at 20 kV accelerating voltage; 2 nA beam current with 10 μm electron beam was used for Na, Si, Al, K whereas for the other elements 20 nA beam current with a spot beam (1-2 μm) was used. The correction program based on the PAP method (Pouchou & Pichoir, 1988) was used to process the results for matrix effects. To test the homogeneity, ten points were analysed on each sample and the mean value was calculated. The elements Co, Cu, Sn, Sb and Pb were also analysed by LA-ICP-MS.

Laser-Ablation Inductively Coupled Plasma Mass Spectrometry (LA-ICPMS)

LA-ICPMS was used to determine the concentration of trace elements. The analyses of the Sarno samples were carried out with a Thermo Fisher X-Series^{II} quadrupole based ICP-MS coupled with a New Wave ablation system with a frequency quintupled ($\lambda = 213$ nm) Nd:YAG laser, at the Centro Grandi Strumenti of the University of Modena and Reggio Emilia. The laser repetition rate and the laser energy density on the sample surface were fixed to 20 Hz and ~ 18 J/cm², respectively. The analyses were carried out using a laser spot diameter of 100 μ m on the same polished fragments used for EMPA. External calibration was performed using NIST SRM 610 and 614 glass as external standard, and ²⁹Si, previously determined by EMPA, as internal standard, following the method proposed by Longerich *et al.* (1996). Standard Reference Material NIST612 (Pearce *et al.*, 1997) was used as a secondary reference sample to check precision and accuracy. All the other specimens (and the Sarno samples too) were analysed by LA-ICPMS at the IRAMAT Centre Ernest-Babelon (CNRS/Univ. Orleans, France) to obtain the trace elements composition. The ablation system consists of a Neodyme:YAG laser working at 266 nm (quadrupled frequency) coupled with a Thermo Electron Finnigan ELEMENT XR mass spectrometer (Gratuze, 2013).

Isotopes analysis

Isotopic analyses of Bologna black samples FiBo5bl, FiBo6bl, FiBo7bl, FiBo19bl, FiBo22bl and FiBo23bl were carried out at the University of Leuven (Belgium). Only these samples were selected for isotopic analyses due to the availability of materials since this analysis requires at least 300 milligrams of glass. The glass samples were finely powdered and subjected to a digestion procedure. The samples were dissolved in a 3:1 mixture of 22M HF and 14M HNO₃ at 110 °C over night. They were then dried and dissolved in aqua regia (3:1 mixture of 12M HCl and 14M HNO₃). The sequential extraction methods for the separation of Sr and Nd from the sample solutions were taken from Pin *et al.* (1994) modified as reported in Ganio *et al.* (2012). Sr was isolated via extraction chromatography using Sr spec resin (Eichrom). The acid 7M HNO₃ was used to rinse the columns and 0.05M HNO₃ to elute the Sr fraction from the resin. Nd was separated via a two-step extraction chromatography using *i*) TRU spec (Eichrom) to separate the REE with 2M HNO₃ and *ii*) Ln spec resin (Eichrom) to separate the Nd from it with 0.25M HCl.

The elemental concentrations of both Sr and Nd were measured with a Xseries^{II} ICP-MS. The obtained concentration results were used to calculate the dilutions for the isotope measurements. The Sr and Nd isotopic ratios were measured with a Thermo Scientific Neptune multi-collector ICP-MS. As standard, NIST SRM 987 SrCO₃ isotopic standard solution for the Sr measurements and a JNdi-1 solution for Nd, were used. All measurement values are corrected for the HNO₃ blank and isobaric interferences. Finally, these are normalised to ⁸⁶Sr/⁸⁸Sr = 0.1194 and ¹⁴⁶Nd/¹⁴⁴Nd = 0.7219 to correct for instrumental mass discrimination.

RESULT AND DISCUSSION

The analyses of major and minor elements were performed on 143 glass. The detailed analysis of the compositions allowed the distinction of six different chemical groups (with sub-groups) and one outsider, based on the flux employed for the glass production and on the peculiar colourant used too. Specifically we have recognised:

1) **MIXED ALKALI GLASSES** were produced with leached plant ashes as flux and are characterised by low MgO (0.5-1.1 wt.%), high K₂O (7.62-9.85 wt.%) and medium levels of Na₂O (5.66-8.26 wt.%). Within this group we have recognised a sub-group of three samples characterised by very high K₂O concentrations (13.44-18.59 wt.%). Mixed alkali glass group is composed by 28 samples: the only two from Broglio di Trebisacce, five from Lipari-Piazza Monfalcone and one from Torre Galli. The three samples of the HIGH-K sub-group were all from Roca Vecchia and exhibited lower Na₂O concentrations (1.01-5.12% wt.%) and higher CaO concentrations (3.59 wt.% on average) compared to the other mixed alkali glasses.

2) **NATRON GLASSES** were produced with an evaporitic salt (known as natron) as flux and are characterized by low levels of K_2O and MgO (both below 1.5 wt.%). This group is composed by 54 samples, containing also some of the iron black glasses (23 samples), high lead opaque yellow glasses and a sample with really high CuO contents (28 wt.%). In this group we found samples coming from: Capua (seven samples), Cuma (seven samples), Francavilla Marittima (nine samples), Sarno (fifteen samples), Torre Galli (five samples), Amendolara (one sample, and Bologna (ten samples).

3) **PLANT ASH GLASSES** were produced with ashes of halophytic plants as flux and are characterised by high levels of K_2O and MgO (0.62-3.76 wt.% and 1.61-7.06 wt.%, respectively). This group is composed by 36 samples: one from Pompei, Vivara, and Murgia Timone, two from Lipari and two Francavilla Marittima, seven from Torre Galli, nine from Capua, and thirteen from Sarno.

4) **HIGH-ALUMINA GLASSES** are characterised by high concentrations of MgO and K_2O (1.85 wt.% and 2.23 wt.%, respectively), which theoretically exclude the use of natron as flux. However the amounts of MgO and K_2O can be related to the use of an impure sand source, rich in Fe-minerals (FeO 3.91 wt.%), clay and feldspars (Al_2O_3 9.65 wt.%). Moreover, taking into account their low amount of lime (CaO 2.52%) the employ of natron as flux cannot be excluded. This group contains only three samples, all coming from Sarno.

5) **COBALT BLUE GLASSES** are characterised by high MgO (2-5 wt.%), high Al_2O_3 (4.36-7.72 wt.%) and low K_2O (0.1-1 wt.%). This group is composed by 12 glasses (4 from Capua and 8 from Sarno) which also showed high levels of CoO (0.11 wt.%) correlating with magnesia and associated to trace elements (such as nickel and zinc) that indicate the use of cobaltiferous alums as source of colorant. Furthermore they showed low contents of CaO (1.3-4 wt.%), K_2O and P_2O_5 (0.08-0.92 wt.% and 0.03-0.08 wt.%, respectively), suggesting the use of natron as source of flux (as lime is a major constituent of plant ash, their employ would typically yield a glass with 5 wt.% CaO or more). Therefore, the high amount of MgO of these cobalt blue samples can be related to the use of cobaltiferous alums as colourant.

6) **IRON BLACK GLASSES** are characterised by the highest FeO values of the entire sample set (3.54-19.89 wt.%). This group is composed by 32 black samples: two from Francavilla Marittima, four from Torre Galli, nine from Cuma, six from Sarno and eleven from Bologna. Twenty-three of them have been already classified as natron glass whereas the others nine showed too high amount of MgO (1.52-3.54 wt.%) to be classified as natron glass (<1.50 wt.% MgO). Anyway, their MgO contents could be related to the use of a very impure sand source (as suggested also by the higher concentrations of Al_2O_3 : on average 2.6 wt.% vs. 1.34 wt.% of the other black samples) rich in Fe-Mg minerals (*e.g.*, pyroxene), rather than to the fluxing agents, making them suitable to be classified as natron based glass. The group showed some peculiar characteristics: high Na_2O (12.32-18.68 wt.%), low K_2O (0.1-1.5 wt.%) and CaO (0.93-6.69 wt.% – with the exclusion of two Torre Galli samples characterised by 16.87 wt.% and 8.94 wt.% CaO , respectively), high TiO_2 (0.08-0.31 wt.%) and FeO (3.54-15.59 wt.%). As in the case of the cobalt-blue glass, the low lime and potassium contents are consistent with the use of natron, as also observed for coeval black glass from France (Gratuze & Picon, 2006) and Italy (Henderson & Arletti, pers. comm.), also showing high FeO (10-14 wt.%), which is responsible for the black coloration.

Finally, one of the studied samples (**SN19b1**) exhibited a very peculiar chemical composition: low Na_2O content (7.73 wt.%), but very high amount of PbO (13.75 wt.%) and high FeO too (12.34 wt.%). This sample remains difficult to understand; in fact, the low contents of both Na_2O and K_2O cannot be directly related to the use of natron or plant ashes. Moreover, the high levels of PbO (13.75%) clearly indicate an intentional addition of the element, probably to allow the melting.

The trace elements analysis was performed on all the transparent samples, with the exclusion of four samples due to their limited dimensions. The trace elements averaged values were normalised to the concentration of the upper continental crust (Wedepohl, 1995). The investigated glass showed how the compositional variations of the sample set are strictly consistent with the grouping found with the major/minor element chemistry, allowing a further subgrouping.

Natron glass can be considered as a mixture of three components: silica sand, lime-bearing material and natron as soda-rich flux. Natron is a relative pure soda source and therefore the trace elements are mainly related to the vitrifying portion of the batch. This principle is valid also for the plant ash glass, even if some elements (such as Cr and Rb) could have been introduced also by the ashes. Glass was often coloured or decoloured by adding a small amount of specific minerals. All these raw materials introduce a number of trace elements to the glass batch (Brems & Degryse, 2013). The trace and rare earth elements in quartz-rich sand are generally concentrated in the accessory- and clay-minerals, with the exception of Sr which is mostly provided by the source of lime: marine shells or limestone for the natron glass, plant ashes themselves for the plant ash glass (Brems & Degryse, 2013).

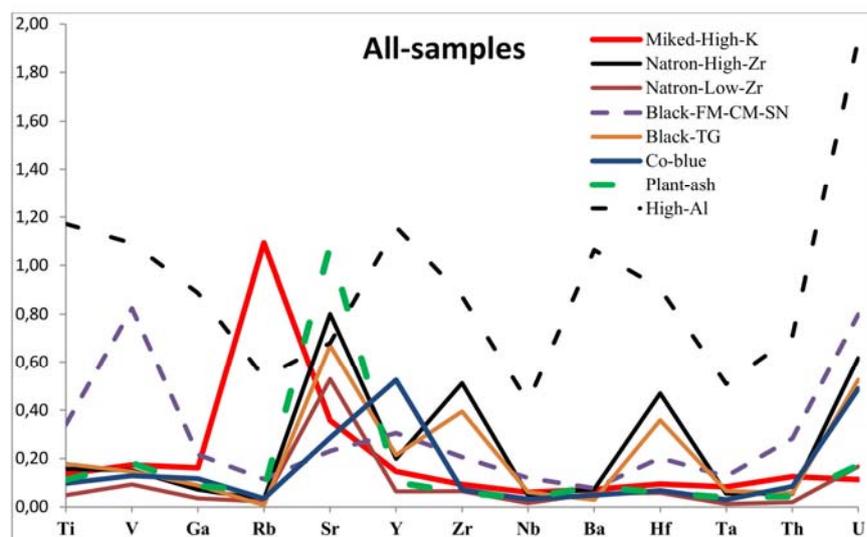


Fig. 1 - Average trace element composition for glass groups normalized to the composition of the upper continental crust (Wedepohl, 1995).

The line graph of Fig. 1 summarizes and compares the different chemical groups found through the trace element study, which correspond to those found also by the rare earth element analysis (Fig. 2). On the basis of these results, we can observe that: *i*) **MIXED ALKALI and HIGH-K GLASSES** form a single group with the highest content of rubidium (Fig. 1), also characterised by a rather flat and strongly depleted REEs pattern (Fig. 2); *ii*) **NATRON LOW Zr-Hf GLASSES** shows the most depleted trace elements pattern (Fig. 1), corresponding to the most depleted REEs pattern of the whole sample set (Fig. 2); *iii*) **NATRON HIGH Zr-Hf GLASSES** are characterised by high levels of zirconium and hafnium (Fig. 1), showing also an higher levels of REEs compared to the natron Low-Zr, with a slightly increasing from LREEs to HREEs (Fig. 2); *iv*) **IRON BLACK GLASSES** from Francavilla Marittima-FM, Cuma-CM, Sarno-SN and Bologna-BO, form a single group, showing high content of titanium, vanadium and yttrium and the lowest Sr content (Fig. 1); they are also associated to relevant levels of REEs and to positive Tb and Yb anomalies (Fig. 2); *v*) **IRON BLACK GLASSES** from Torre Galli-TG, show the same trace elements and REEs pattern of the Natron High Zr-Hf glass (Fig. 1-2); *vi*) **COBALT BLUE GLASSES** are characterised by a depleted trace elements pattern, but enriched in yttrium (Fig. 1); they result strongly depleted in LREE, but significantly less depleted in Middle and Heavy REE (Fig. 2); *vii*) **HIGH ALUMINA GLASSES** are characterised by the lesser depleted trace elements pattern (Fig. 1); they show the highest contents of REEs and increasing pattern from the LREEs to the HREEs, strongly enriched in Yb (Fig. 2); *viii*) **PLANT ASH GLASSES** are characterised by a depleted trace elements pattern and by the highest levels of strontium (Fig. 1); they can be furthermore subdivided on the basis of their cerium contents (Fig. 2) into: a) **PLANT ASH LOW-Ce**, with a strongly depleted and flat REEs pattern and b) **PLANT ASH HIGH-Ce** displaying a slightly less depleted REEs pattern (mostly in light and middle REEs), with a positive Ce anomaly.

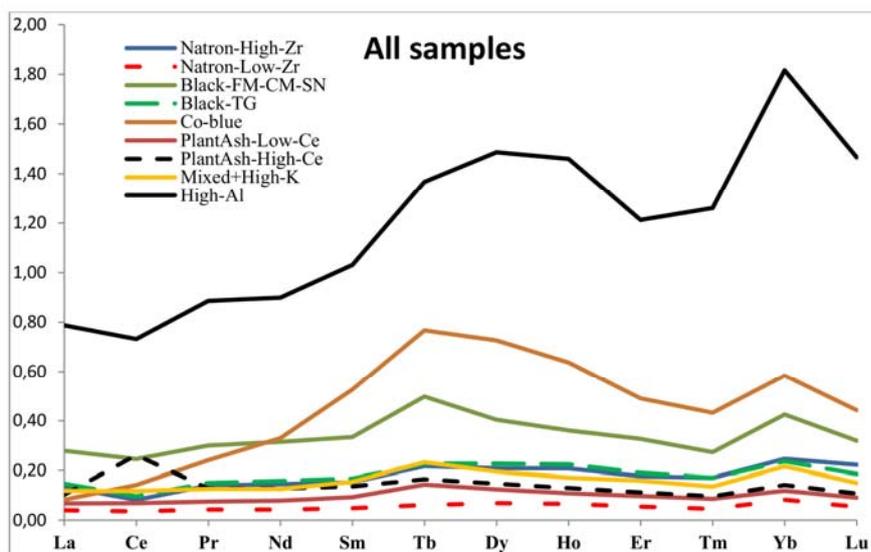


Fig. 2 - Average REEs composition for glass groups normalized to the composition of the upper continental crust (Wedepohl, 1995).

The trace element and REEs data allow us to make hypothesis regarding the raw materials used. Therefore, we can say that the MIXED ALKALI and HIGH K glasses were produced with a sand poor in heavy minerals. The high amount of Rb can be related to the high level of K introduced along with the flux or with the K-feldspars of the sand. The classic NATRON glasses are split into two groups: high Zr-Hf produced with a mineralogically mature sand, rich in quartz and zircon and relatively depleted in other heavy minerals and low Zr-Hf produced with a very pure silica source, perhaps quartz pebbles. The IRON BLACK glasses were produced with very impure sand rich in iron oxides (*e.g.*, magnetite), and heavy minerals (zircon-Y, HREE-garnet, monazite-LREE, rutile-REE); the fact that the black samples from Torre Galli show the same trace and rare earth elements composition of the Natron High Zr-Hf seems to suggest that they were all produced in the same place or with the same vitrifying raw materials. The COBALT BLUE glasses instead show a trace and REEs composition related to the cobaltiferous alum used as colourant, as demonstrated also by other studies relative to the Mycenaean (Walton *et al.*, 2009) and Uluburun (Jackson & Nicholson, 2010) cobalt glasses of the 2nd millennium BC and to the Bologna cobalt glass of the 1st millennium BC (Arletti *et al.*, 2011b). The HIGH-ALUMINA glasses show the highest levels of all trace elements, suggesting the use of a very impure sand source, rich in Fe-minerals, clay and feldspars. The PLANT ASH glasses were produced with a pure quartz source, in the case of the High-Ce plant ash; the higher cerium suggests the use of a different source containing a major amount of minerals enriched in LREEs, such as monazite.

The comparison between our trace results and the trace and REEs reported in literature, and the Sr-Nd isotopic results, allowed making hypotheses regarding the provenance of the Southern Italian glasses. Mixed alkali glasses were imported from the Final Bronze Age production site of Frattesina (Northern Italy); natron glasses, which are among the first natron glasses ever produced (9th-6th century BC), probably came from Egypt; black glass isotopic analyses demonstrated that it was produced in inland Egypt; the cobaltiferous alum employed for the alumina-cobalt blue glass is a distinctive fingerprint for the Egyptian glass production; plant ash glass had a Mesopotamian origin; high alumina glass resulted to be very uncommon among the contemporary glass and could represent part of the Egyptian early natron production.

The samples here analysed are dated from the Early Bronze Age (18th century BC) to the Advanced Iron Age (up to the 6th century BC). As reported in Fig. 3 the Early and Middle Bronze Age (EBA-MBA) materials show a plant ash composition, while during the Final Bronze Age (FBA) the glass mainly exhibits mixed alkali chemistry, both typical of the Bronze Age technologies.

At the transition with the Iron Age (corresponding to the transition between 2nd and 1st millennium BC) natron glass made its appearance. During the Early Iron Age 1-2, the coexistence of the Bronze Age glass-

making traditions (mixed alkali and plant ash glass) and of the Iron Age one (natron glass) is attested. In the Advanced Iron Age (Orientalising/Archaic period) some elements of the Bronze Age tradition still survived, as the ongoing use of plant ash glass, but the predominant aspect is the progressive evolution and standardisation of the natron glass production, which reached the peak in Roman times.

	EBA	MBA2	MBA3	RBA	FBA	EIA1-2	O/A
Pompei-S. Abbondio	Green						
Vivara		Green					
Murgia Timone			Green				
Punta di Zambrone				Pink			
Broglio di Trebisacce					Grey		
Lipari-P. Monfalcone					Green		
Roca Vecchia					Grey		
Cuma						Yellow	Red
Capua						Green	Red
Bologna-Fair						Yellow	Red
TorreGalli						Green	Red
FrancaVilla						Green	Red
Sarno						Green	Red
Amendolara							Red

Fig. 3 - Chronological summary of the glass chemical groups present in the sites of this study. Chemical glass groups: Green = Plant ash; Pink = LMLK; Grey = Mixed alkali; Yellow = black glass; Red = natron; Blue = Al-Co blue; Black = High-Al.

The detailed comparison between the protohistoric vitreous materials from Northern and Southern Italy reported in Fig. 4, allows to demonstrate the existence of different trends, most of all in the Early and Middle Bronze Age 1-2. At that time, in fact, the North of the country was involved in the trades with the Central Europe (presence of mixed alkali glass-typical European recipe), while Southern Italy was already inserted in the Mediterranean trade routes (plant ash glass).

	EBA	MBA2	MBA3-RBA	FBA	EIA1-2	O/A
Southern Italy	Green	Green	Green	Green	Green	Yellow
Northern Italy	Grey	Grey	Grey	Grey	Green	Red

Fig. 4 - Comparison between the protohistoric vitreous materials from Southern and Northern Italy. Green = Plant ash; Brown = HMBG; Pink = LMLK; Grey = Mixed alkali; Yellow = black glass; Red = natron; Blue = Al-Co blue; Black = High-Al.

In the Middle Bronze Age 3-Recent Bronze Age the same chemical types were found both in Northern and Southern Italy, suggesting the circulation of the vitreous materials through all the country. The massive production of mixed alkali glass in Frattesina (Northern Italy) during the Final Bronze Age totally replaced the Near East materials in the North. Frattesina glass spread also to the South, where, however, the continuity in the trades with the Near East (plant ash glass) is also attested. In the Early Iron Age 1-2, the coexistence of FBA Frattesina production and the appearance of materials with a strong Eastern affinity (natron glass) were recorded both in the North and in the South of Italy. Data relative to the Advanced Iron Age confirmed the spread of Mediterranean materials (natron glass) over all the country.

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