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The glass finds from the Vetricella site (9^{th}-12^{th} c.)

Archaelogy and archaeometry

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We examine here the glass finds from the Vetricella site. This site, investigated during the ERC-Avanced nEU-Med project (www.neu-med.unisi.it) is interpreted as the centre of the royal *curtis* of Valli attested in documentary sources from 937. During the excavation, a large number of glass objects dated from the 4^{th} to the 12^{th} centuries were unearthed. The compositions of the early mediaeval Vetricella glass vessels (beakers, goblets, bottles, ampoules/vials), are studied and compared with those of similar finds from various other European sites. With the exception of one glass bottle, made with Syro-palestinian plant ash soda glass, all the other studied glasses belong to Antique or Late-Antique natron glass groups (Mn- and Sb-decolorized, Foy 2 and HIMT). All these objects illustrate the recycling practices of Antique and Late Antique glass in Europe as they are described by Theophilus for the 11^{th} and 12^{th} centuries.

Early Middles Ages, central Italy, glass production and consumption, Glass recycling, chemical analyses

INTRODUCTION

Important changes in the glass manufacturing process occurred in the Mediterranean world at the end of the 8^{th} century. This phenomenon stems directly from a progressive decline of the production of raw natron glass in the Near-East. From the 9^{th} century onward, in Eastern Mediterranean and Mesopotamia, soda-lime glass made from halophytic plant ash becomes the prevailing type of glass before becoming by the 12^{th} century the dominant glass type throughout the Mediterranean. However, natron glass seems to be still used until the end of the 12^{th} century in Europe for common vessels and for more specific productions such as the cobalt blue vessels and churches window panes. In the recent years, the increasing number of analyses carried out on Western European glass dated from this transition period has highlighted both the presence of a great variety of different glass compositions and the importance of natron glass recycling. A large part of the Vetricella glass vessels (cup, beakers, goblets, bottles, ampoules/vials), unearthed on this site, belong to this transition period. The compositions of these objects, which were analysed by LA-ICP-MS at the IRAMAT, Centre Ernest-Babelon (CNRS, France) in collaboration with the nEU-Med project team, is compared with those of Antique and Late Antique glass productions as well as with those of Islamic Near Eastern glasses and of Early Middle Ages European productions. A large part of the results obtained on the cup of blue glass decorated with opaque white glass strands and pellets and red glass trails has already been published.[1] Therefore, these results won't be presented in detail here but will be mainly used as comparative data.

1. Gratuze 2020.

THE VETRICELLA SITE AND GLASS FINDS

Vetricella, Scarlino (GR), is located in Tuscany, in the central portion of the coastal plain crossed by the river Pecora, at the foot of the castle of Scarlino; discovered in 2005 thanks to aerial reconnaissance: three large concentric circles that develop around a quadrangular trace.

The site has been part part of the project: "Origins of a new economic union (7^{th}-12^{th} centuries): resources, landscapes and political strategies in a Mediterranean region", European project within Horizon 2020, ERC Advanced. Host institution is the University of Siena, directed by Professor Richard Hodges (P.I. of the project) and Professor Giovanna Bianchi (Unisi coordinator). It started at the end of 2015 and finished in 2021.

The Vetricella site has become the key-study of the project due to the wealth of data collected in multidisciplinary surveys. The site is interpreted with the center of a royal Curtis, called *Valli*, attested for the first time in 937 AD but with a continuity of life from the 8^{th} to the 12^{th} century.

Glass artefacts found at the site of Vetricella comprise 168 fragments.[2] Identification was not possible for 50 of these. 118 of the 168 sherds have been analysed, of which 83 different forms and 4 semi-finished fragments have been identified.

The identified glass fragments have been divided into seven different forms as well as semi-finished production (fig. 1): I. Cups/Small Cups, 2. Beakers, 3. Goblets, 4. Lamps, 5. Ampoules/vials, 6. Bottles, 7. Gaming pieces, 8. Semi-finished products (shapeless molten glass).

Different types, dated from Late Antiquity to Early Middle Ages were identified for the cups, goblets, lamps and beakers.[3] The large number of goblets from Vetricella (20 fragment of goblets minimus number) is evocative of similar discoveries in the Domus solarata from Forum of Nerva (Roma, later 10^{th} or early 11^{th} centuries).

The study of the glass finds, the typological analysis and the discussion on the stratigraphic provenance have been extensively discussed by the writer in the article in the volume of the project. (L. Castelli, *Glass artefacts from the site of Vetricella (Scarlino, Grosseto)*, in G. Bianchi, R. Hodges (eds.), *The nEU-Med project: Vetricella, an Early Medieval royal property on Tuscany's Mediterranean*, Florence, 2020, Library of Medieval Archeology 28, p. 69-75.)

THE STUDIED GLASS CORPUS

The 43 selected samples were chosen among the different vessel types dated from Early Middles Ages levels. They consist of (fig. 2 and tab. 1):

- 5 sherds belonging to a cup of blue glass decorated with opaque white glass strands and pellets and red glass trails and dated from 10^{th}-12^{th} century. Thirteen sherds from the same cup were previously analysed and published.[4] The fragments of blue Glass were concentrated in the eastern area of the site, near the ecclesiastical building, in contexts dating back to the mid XI century.
- 38 greenish and colourless sherds originating from different vessel types:
- 8 greenish sherds with some red trails originating from 4 beakers type 2 dated from Early Middles Ages,
- 22 sherds originating from 14 goblets type 1 (mostly greenish or pale greenish, except 2 colourless samples) and 8 goblets type 2 (mostly colourless or slightly pale greenish) dated from 10^{th}-13^{th} century. Goblets were positioned in different areas around the tower, in stratigraphy almost all ascribable to the 10^{th}-12^{th} century.
- 2 sherds belonging to two bottles dated as Early Medieval, one pale green and the other darker,
- 4 greenish sherds from 2 ampoules and 1 vials (2 sherds) dated from 11^{th}-12^{th} century
- and 2 sherds from semi-finished products (shapeless molten glass), one dark green and one greenish, possibly indicative of production activities on site.

2. Castelli 2020.
3. Castelli 2020.
4. Gratuze 2020.

TABLE 1. STUDIED CORPUS OF VETRICELLA GLASSES.

US	N. Schedule	TYPE	N. Sherds	Date	N. Sample	Chem. group
127	19	Blue glasses Cup type 2	1	X-XII c.	1	Roman
0	7	Blue glasses Cup type 2	2	X-XII c.	2 A & B	Roman
178	12	Blue glasses Cup type 2	1	X-XII c.	3	Roman
fondo trin-cea B	20	Blue glasses Cup type 2	1	X-XII c.	4	Roman
178	8	Beakers type 2	3	Early Middle Ages	5 A, B, C	Foy 2
130	22	Beakers type 2	3	Early Middle Ages	6 A, B, C	Foy 2
Q.I. 8	74	Beakers type 2	1	Early Middle Ages	7	Foy 2
3006	102	Beakers type 2	1	Early Middle Ages	8	Foy 2
0	21	Goblets type 1	1	X-XIII c.	9	Roman Mn-Sb
Q. I. 8	25	Goblets type 1	1	X-XIII c.	10	Roman Mn-Sb
ripulitura	37	Goblets type 1	1	X-XIII c.	11	Foy 2
sporadico	39	Goblets type 1	1	X-XIII c.	12	Roman Sb-Mn
385	46	Goblets type 1	1	X-XIII c.	13	HIMT/Foy 2
Q. E. 11	51	Goblets type 1	1	X-XIII c.	14	Foy 2
837	54	Goblets type 1	1	X-XIII c.	15	Foy 2
860	58	Goblets type 1	1	X-XIII c.	16	Foy 2
848	57	Goblets type 1	1	X-XIII c.	17	Foy 2
844	56	Goblets type 1	1	X-XIII c.	18	Foy 2
968	60	Goblets type 1	1	X-XIII c.	19	Foy 2
844	79	Goblets type 1	1	X-XIII c.	20	Foy 2
4001	105	Goblets type 1	1	X-XIII c.	21	Foy 2
4001	104	Goblets type 1	1	X-XIII c.	22	Foy 2
127	6	Goblets type 2	1	X-XIII c.	23	Roman Sb-Mn
ripulitura	2	Goblets type 2	1	X-XIII c.	24	Roman Sb-Mn
0	19	Goblets type 2	1	X-XIII c.	25	Roman Mn-Sb
0	28	Goblets type 2	1	X-XIII c.	26	Roman Sb-Mn
160	32	Goblets type 2	1	X-XIII c.	27	Roman Sb-Mn
0	77	Goblets type 2	1	X-XIII c.	28	Roman Sb-Mn
837	81	Goblets type 2	1	X-XIII c.	29	Roman Sb-Mn
2001	89	Goblets type 2	1	X-XIII c.	30	Roman Sb-Mn
ripulitura	36	Bottle	1	Early Middle Ages	31	Syro-palestinian soda plant ash glass
863	59	Bottle	1	Early Middle Ages	32	HIMT
127	12	Ampoules	1	XI-XII c.	33	Foy 2
ripulitura	42	Vials	2	XI-XII c.	34 A & B	Foy 2
848	80	Ampoules	1	XI-XII c.	35	Foy 2
Q. I. 9	38	Semi-finished	1		36	Foy 2/HIMT
ripulitura	70	Semi-finished	1		37	Foy 2

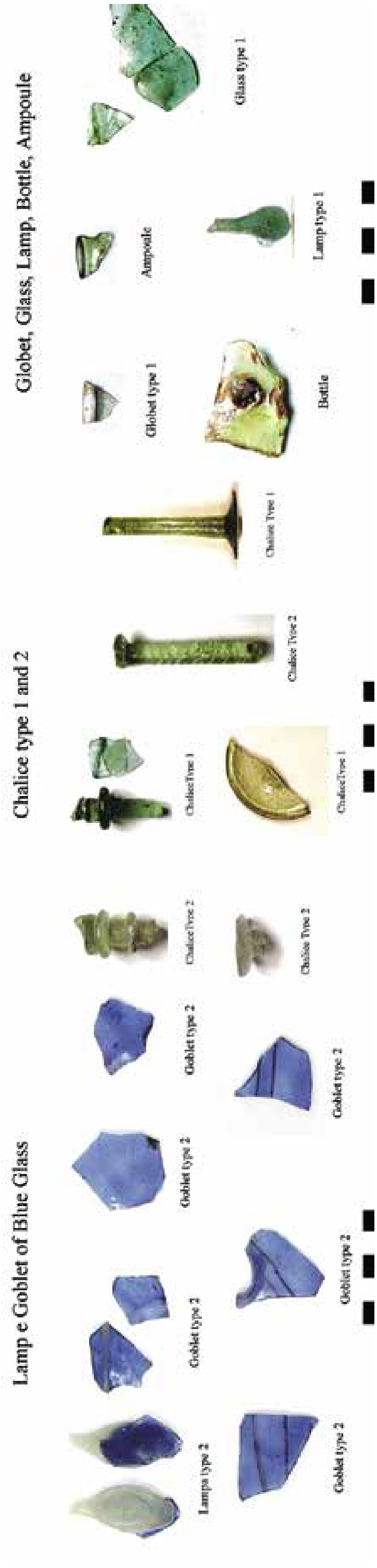

Fig. 1. Some examples of Vetricella Glasses.

Fig. 2. LA-ICP-MS analytical cell (S155 from Resonetics) used for the analyses of the Vetricella glass corpus. Samples in the metallic cylinders are the glass standards.

ANALYTICAL METHOD

The analyses of the glass sherds were carried out by Laser Ablation-Inductively Coupled Plasma-Mass Spectrometry (LA-ICP-MS). The instrumentation consisted of a Resolution M50E UV laser probe from Resonetics/ASI (Eximer ArF laser working at 193 nm equipped with the S155 cell) coupled with a Thermo Fisher Scientific ELEMENT XR mass spectrometer.[5] LA-ICP-MS allows a nearly non-destructive analysis, invisible to the naked eye, of the glass objects. Analytical parameters were as follow: the excimer laser was operated at 5.5 mJ with a repetition rate of 10 Hz, ablation time was set to 50 seconds: 20 seconds pre-ablation, so that transient signal and possible surface corrosion or contamination could be removed, and 30 seconds collection time corresponding to 9 mass scans from lithium to uranium. The signal was measured in counts/second, in low resolution mode for 58 different isotopes. The fifty-eight elements include all major, minor (except sulphur) and trace elements which are usually present in glass samples.[6] Blanks were run periodically between series of 20 analyses. Spot sizes were set to 100 µm (although reduced down to 70 µm when saturation occurred for element such as manganese or copper). During analysis live counts were continuously observed: when element spikes signifying the presence of inclusions were observed, results were discarded and a new site selected. From one to eight areas were analysed per sample in order to analyse the different part and different coloured glasses present on the sherds; homogeneity and agreement between runs carried out on the same type of glass were consistently good. However, some samples show variations in composition due either to the presence of red trails, with significantly different composition, within the glass or to imperfect mixing of glasses of different compositions (insufficient temperature and melting time). In order to take these variabilities into account, we have chosen to represent all the analyses performed on the graphs rather than the average compositions of the objects.

Calibration was performed using five reference standards: NIST610, Corning B, C and D, and APL1 (an in-house reference glass used for chlorine determination), were run periodically (every 15 to 20 samples) to correct for eventual drifts. The standards are used to calculate the response coefficient (k) of each element. The measured values were normalised against ^{28}Si, the internal standard. Concentrations are calculated assuming that the sum of the concentrations of the measured elements is equal to 100 weight percent. In total, 58 elements were recorded. For the major and minor elements accuracy and precision were within 5% relative and within 10% for most trace elements.

RESULTS

Identification and provenance of glass groups

According to their MgO (from 0.40% to 0.96%) and K_2O (from 0.36% to 0.93%) contents (fig. 3), with the exception of one bottle, all the analyzed glasses from Vetricella are mineral soda-lime glass, that is glass obtained by melting a siliceous sand with a mineral soda flux such as natron (fig. 3).

One of the studied bottle (N 31) is characterized by higher MgO (2.57%) and K_2O (1.91%) contents and was made with soda originating from halophytic plant ashes such as *Salsola sp or Salicornia sp*. We can notice that the second bottle (N 32) is characterized by the lowest potash (0.36%) content and, with the exception of the white opaque glasses, the highest magnesia content (0.91%) compared to the other natron glasses.

A more detailed study of the potash and magnesia contents of natron glasses (fig. 3 right) shows that the different groups of objects are characterized by increasing magnesia and potash contents from the goblets type 2 to the white opaque glasses. Four main groups can be considered:

- In the first one (I) we find all the goblets type 2 plus two goblets type 1 (N 10 and 12). These glasses have the lowest potash and magnesia contents;
- The second group (II) contains most of the analyses carried out on the cobalt blue glass of the cup plus one goblet type 1 (N 21);

5. Gratuze 2016.
6. Gratuze 2016.

– The third group (III) brings together all the remaining goblets type 1, most of the beakers (except two analyses carried out on the red glass of beaker 5) as well as all the analyses carried out on the red glass trails of the blue cup with some analyses carried out on the white glasses;

– In the last group (IV), we find mainly the white opaque glass together with the analyses done on the red glass of beaker 5 and on the red glass of waste 37 as well as the two analyses carried out on the waste 36.

If we refer to the classification of late Antique and early Middle Ages glasses, according to their TiO_2, Al_2O_3 and SiO_2 contents (fig. 4), the Vetricella's glasses can be classified among the following glass groups: Foy 2, Roman Mn, Roman Sb and HIMT, that is mainly Egyptian glass groups or mixing between Egyptian and Syro-palestinian (Roman Mn) for the common vessels.[8] Whit the exception of the natron bottle which belongs to the HIMT group (fig. 4), all the other natron glasses belong to Foy 2 glass group or to groups obtained

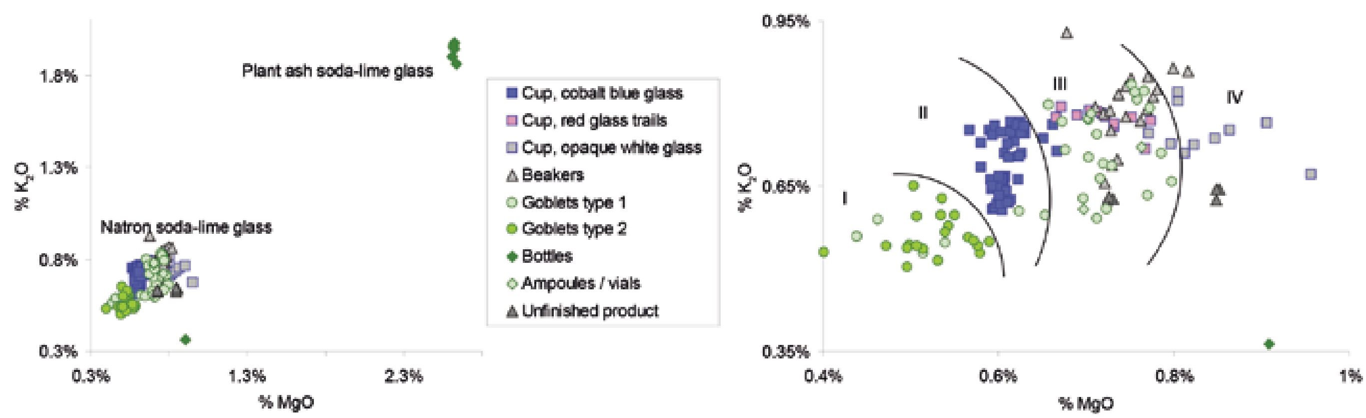

Fig. 3. Distribution of the analysed glasses according to their magnesia and potash contents, on the left, all the glasses are represented, on the right, focus on the natron glass group. All the analytical results are plotted some glasses are represented by 2 to 5 points.

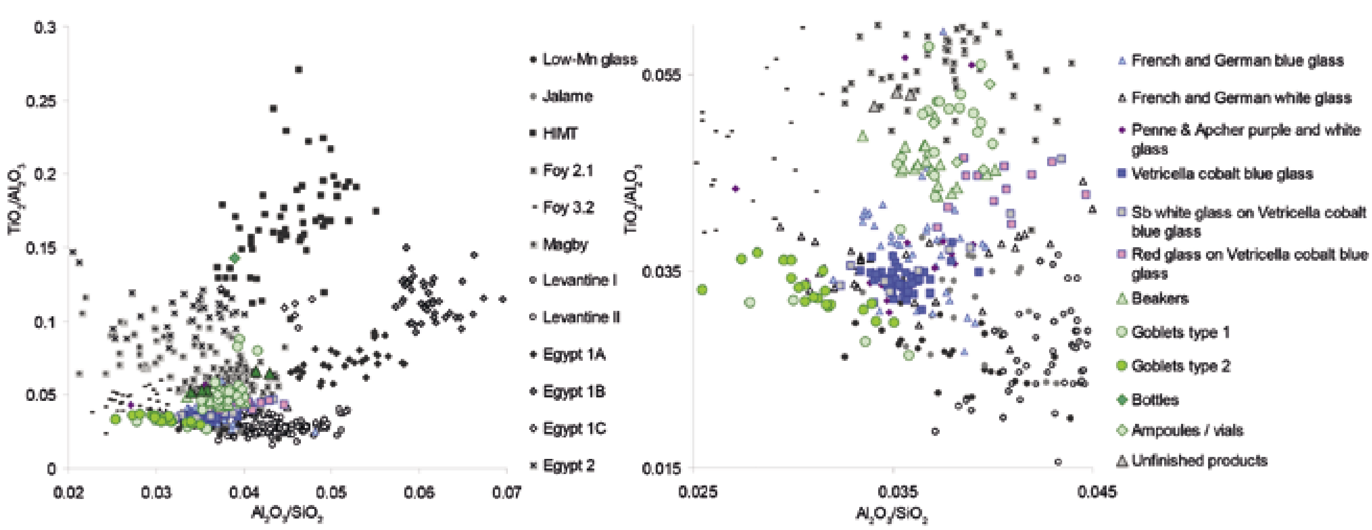

Fig. 4. Distribution of the analysed natron glasses according to their Al_2O_3/SiO_2 and TiO_2/Al_2O_3 ratios. Comparison with Antique, late Antique and early Mediaeval groups of glass.[7] On the right, focus on the main Vetricella's natron glass group. All the analytical results are plotted some glasses are represented by 2 to 5 points.

7. Pactat – Gratuze 2022.

8. Freestone 2021.

by mixing Roman Mn and Sb decolourised glass, or Foy 2 and other groups (Roman Mn/Roman Sb/HIMT, fig. 5 and 6).

According to their SrO/CaO ratios, the glass of the blue cup has probably a Syro-palestinian origin (fig. 7) and, as it has already been discussed in a preceding paper[9] the glasses used to make that cup originates probably from the recycling of Roman tesserae if we refer to their high contents in antimony (fig. 6). We can also notice that despite their distribution on different diagrams(fig. 4 and 5), the red glasses from the cup cannot be classified as Foy 2 glass but have probably also a Syro-palestinian origin (fig. 7).

On figure 7 we also observe that the plant ash glass used to make the bottle N 31 has similar SrO/CaO ratio to the natron glasses used to make the cup. It probably indicates that this glass originates from Islamic Syro-palestinian plant ash glass workshops.[10]

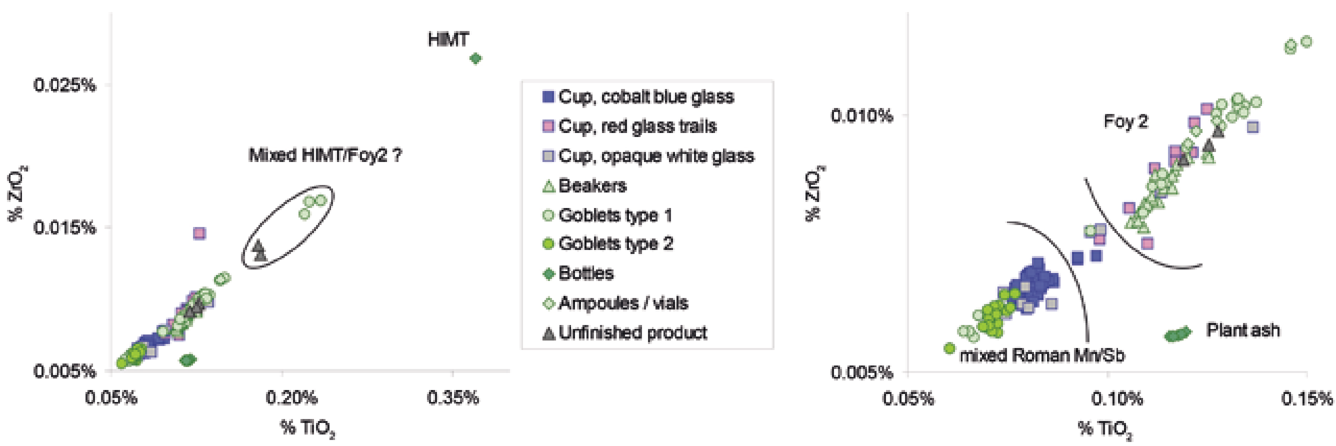

Fig. 5. Distribution of the analysed glasses according to their TiO_2 and ZrO_2 contents. On the right, focus on the lower left part of the diagram. All the analytical results are plotted some glasses are represented by 2 to 5 points.

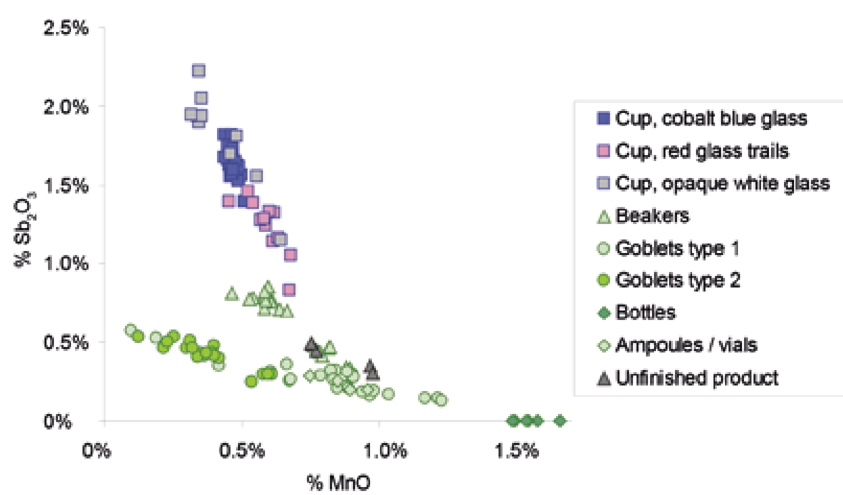

Fig. 6. Distribution of the analysed glasses according to their MnO and Sb_2O_3 contents. The high antimony contents of all the glasses from the cobalt blue cup are probably due to the fact that these glasses originate from the recycling of ancient Roman tesserae.

9. Gratuze 2020.

10. Phelps 2018; Schibille *et al.* 2019.

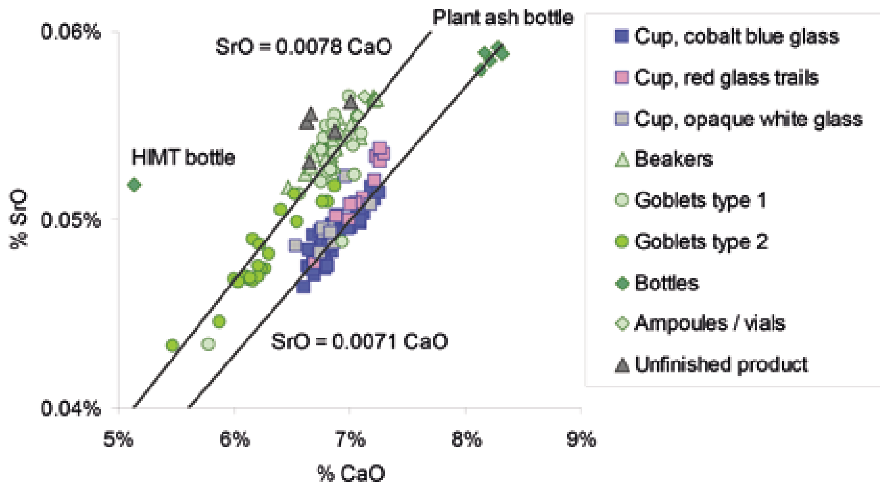

Fig. 7. Distribution of the analysed glasses according to their CaO and SrO contents. With the exception of the HIMT glass, all the other glasses are distributed among two main trends. On the left, we find all the natron glass common vessels with an average SrO/CaO ratio equal to 0.0078 (probably with an Egyptian origin) and on the left the glasses of the blue cup with the bottle plant ash glass with an average SrO/CaO ratio equal to 0.0071 (probably with a Syro-palestinian origin).

Red glass trails: accidental or not?

If we consider now the red glass trails which are present in both the blue cup and the beakers, we can notice on figure 8 that the reduction of copper oxide into metallic copper originate probably from two different processes. In the cobalt blue cup, the red glass is characterized by higher iron, copper and tin contents than the blue glass. It also contains less antimony and more manganese, titanium and zirconium (fig. 5 and 6). Thus the red glasses present in the cup have completely different compositions from the blue glass and originates probably from antique red glass (may be tesserae) mixed voluntary or accidentally with the cobalt blue tesserae recycled to make that cup.

On the contrary in the beakers, the red glass has a composition which appears fairly similar to that of the based greenish glass. It mainly contains a little bit more iron oxide, but presents similar contents of tin and copper as well as all other elements. We can therefore hypothesize that the red glass trails present in the beakers may originate from an accidental reduction, in the crucible, of the copper oxide, originally present in the glass. Its reduction into metallic copper may be due to both, the presence of an excess of iron (contaminations from tools or local heterogeneities) and favourable reductant conditions in the crucible.

Glass recycling

If we now try to estimate the intensity of glass recycling by using the respective contents of copper, tin and lead oxide[11] in the colourless and greenish glasses (fig. 9 top), we observe that Foy 2 glasses (beakers, goblets type 1 and ampoules/vials) present the highest contents for theses oxides and were therefore probably made with the most recycled glasses. On the other hand, goblets type 2 and some goblets type 1, made with mixed Roman Mn and Sb glasses show among the lowest contents of Sn and Cu and content of lead oxides just above the accepted recycling threshold value of 100 ppm level. But the lowest contents for these three oxides were measured for the two bottle made with HIMT and soda plant ash glass. If these results are not surprising for the soda plant ash glass whose production period is probably contemporaneous of Vetricella's occupation period, it is more puzzling for the HIMT glass whose production period dated from the 4th and 5th centuries. These results seem to show that most of the glass used by the glassmakers, who produced the tableware found at Vetricella, did not come from multiple recycling and was probably easily accessible.

11. Ceglia *et al.* 2019.

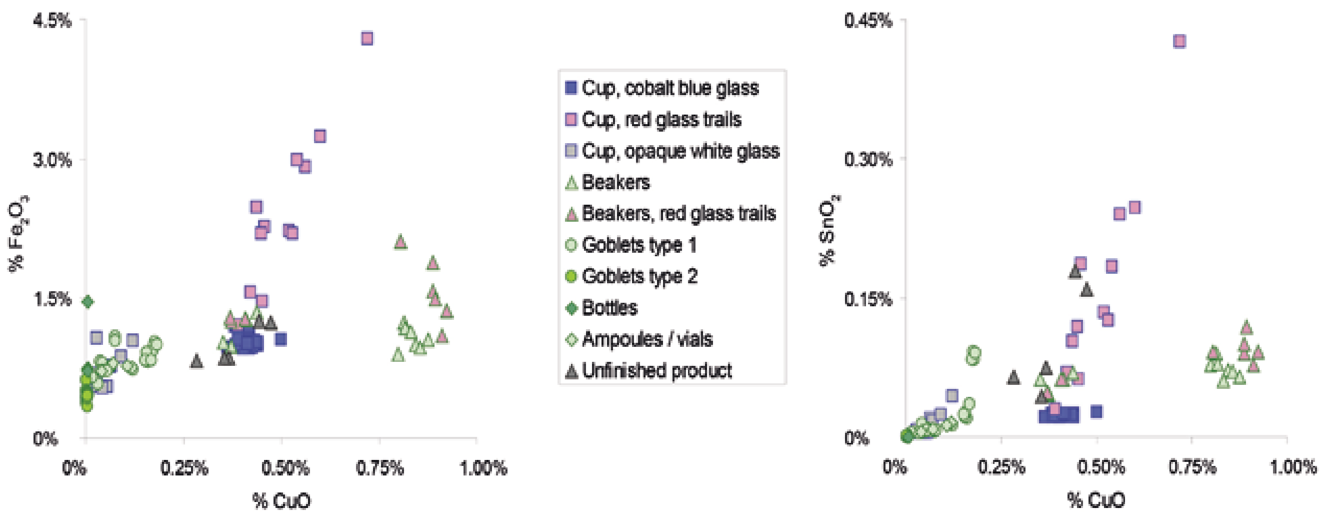

Fig. 8. Distribution of the analysed glasses according to their CuO and Fe_2O_3 contents on the left and CuO and SnO_2 contents on the right. The red glass from the cup exhibits higher copper, iron and tin contents, while the red glass from the beakers exhibits mainly higher iron contents, illustrating that the reduction of copper in these red glasses was not obtained from the same colouring processes.

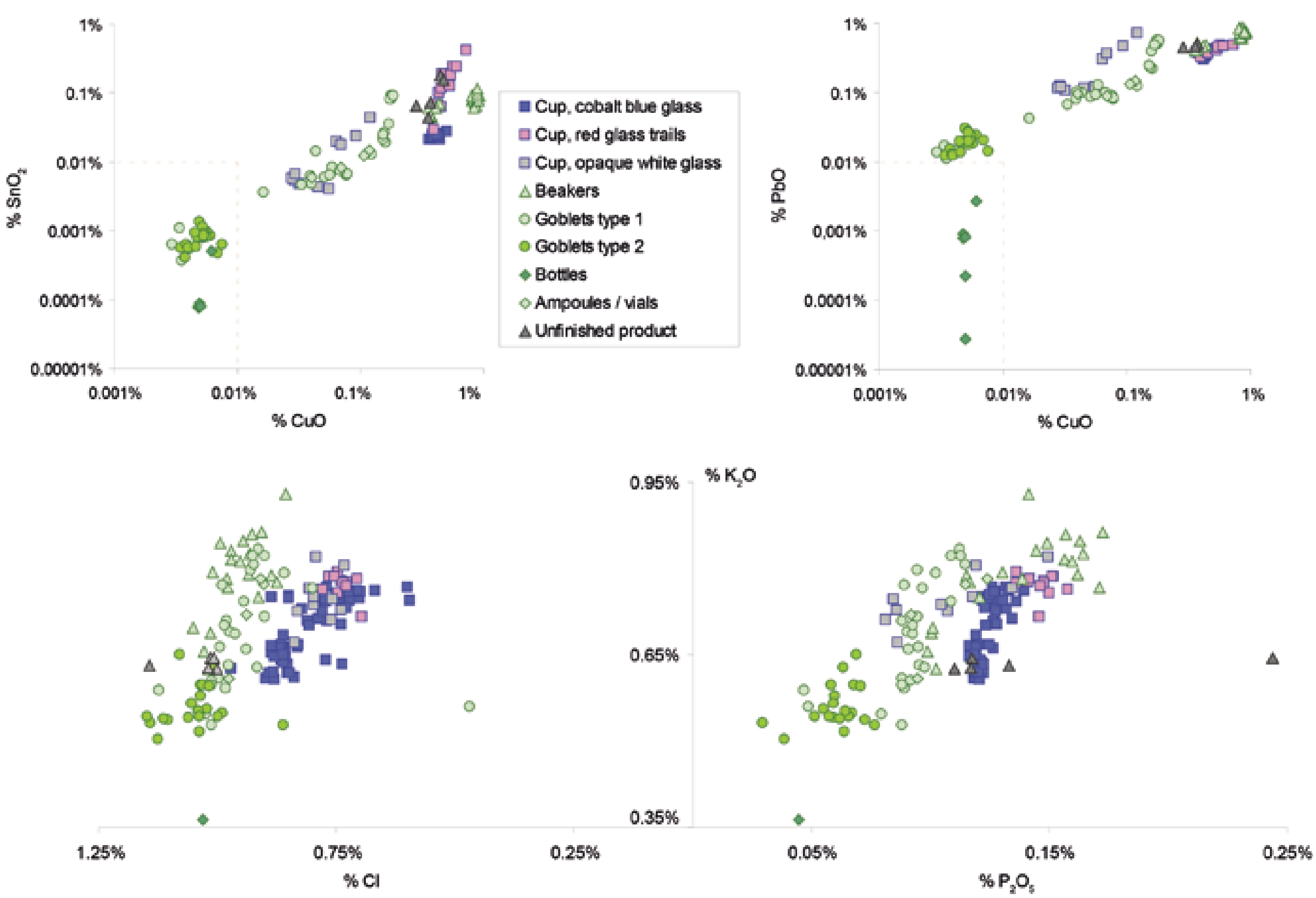

Fig. 9. Distribution of the analysed glasses according to: at the top, their CuO and SnO_2 contents on the left and CuO and PbO contents on the right and at the bottom to their Cl, K_2O and P_2O_5 contents. In colourless glasses, contents in copper, lead and tin oxides are usually used as indicator of intensity of recycling (> 0.01%). These criteria do not apply to the cobalt blue cup glasses as these elements originate from the colouring process. Recycling tend also to increase potash and phosphorus contents (ashes contamination) and decrease contents of volatile element such as chlorine. Applying these criteria to our corpus we observe that the bottles (HIMT and soda plant ash glasses) and in a less extent goblets type 2, were made from less recycled glass than goblets type 1, beakers and ampoules/vials.

Part of this "fresh" glass could have come from architectural glass (window panes) recovered from ancient buildings. Of course, we cannot use the same parameters to estimate the intensity of glass recycling of the cobalt blue cup glasses as these metallic elements are part of the colouring process. For these glasses we can however study the relationship between potash, phosphorus and chlorine (fig. 9 bottom). The contents of first two elements which are present in the furnace atmosphere tend to increase accordingly to the re-melting events while the contents of Cl tend to decrease due to the volatility of this element. Similar results are obtained for all these elements for natron glasses. Goblets type 2 contain the lowest potash and phosphorus contents associated with the highest chlorine contents, while beakers which had the highest contents of metallic impurities have also the highest contents in potash and phosphorus and the lowest contents in chlorine. With these parameters, evidence of recycling is also observed for the coloured glasses of the cobalt blue cup: potash and phosphorus show a positive correlation while a negative correlation is observed between potash and chlorine. If we interpret in term of recycling events the two groups of compositions, observed among the different analysed fragments of the cup and discussed in the preceding paper,[12] we may hypothesize that a sets of fresh tesserae was added into a crucible containing already melted tesserae and that the two sets were incompletely mixed during melting prior to the making of the cup.

These criteria cannot apply to plant ash soda glasses as they contain higher and more variable amount of potash and phosphorus.

CONCLUSION

With the exception of one bottle made with contemporaneous Syro-palestinian soda plant ash glass, all the other studied Vetricella's glasses were made from recycled Antique natron glasses produced before the 7th century.

The natron recycled glasses form two main groups:

- On one side we have the cobalt blue molded glass cup decorated with white opaque glass strands and pellets and red glass trails. Despite a sligltly different typology, its composition is similar to the other vessels of that type found throughout Europe during Ottonian period.[13] The compositions of these vessels illustrate the recycling practices of ancient coloured glass tesserae in Europe, as described by Theophilus for the 11th and 12th centuries (Chap. XII).
- On the other side we have all the colourless or greenish vessels (goblets, beakers…). Their compositions correspond to the recycling of more common glass (windows, vessels, chunks of raw glass…) and can be classified among the more common late antique glass groups: Foy 2, Roman Mn and Sb decolorised glass and HITM. Mixing of glasses belonging to these groups was also observed. It was also noticed that Beakers and Goblets Type 1 were mainly made with Foy 2 glass while Goblets Type 2 were maily made with glass obtained by mixing Roman Mn and Sb decolorised glass.

From the different collected information obtained on these vessels (typologies, chemical compositions, discovery context), we can hypothesize that goblets probably originate from local Italian production while the blue glass cup originate more probably from Northern European production centers.[14]

As it was shown by other types of finds found in the site, Vetricella was the center of a royal court inserted in a large-scale exchange system. The importation of the blue glass cup from northern European production center fits well in this context. Within this frame, we may also reconsider the presence of the Syro-palestinian soda plant ash glass bottle as resulting from the import of Near Eastern goods of which the bottle would have been the container.

12. Gratuze 2020.

13. Pactat – Gratuze 2022; Gratuze 2020.
14. Pactat – Gratuze 2022; Gratuze 2020.

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