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Preliminary chemical characterization of Roman glass from Pompeii

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Abstract - Eleven fragments of Roman glass were analysed by XRF (X-ray Fluorescence) and LA-ICP-MS (laser ablation inductively-Coupled Plasma Mass Spectrometry) to determine major, minor and trace elements, including rare earth elements. The fragments come from the archaeological site of Pompeii (Naples, Italy) and belong to various types of blue-green glass objects.

Analytical investigation of glass was performed in order to determine which melting, colouring and opacifying techniques were adopted to prepare these materials.

The results indicate that the samples, dated to the first century A.D., before A.D. 79, by archaeological suggestions, are soda-lime-silica glass, obtained by using pure sand, *natron* and recycled glasses. Colouring, decolouring and opacifying agents were not used.

Riassunto - Undici frammenti di vetro romano rinvenuti nel sito archeologico di Pompei (Napoli), sono stati analizzati attraverso due metodologie di indagine: analisi XRF e analisi LA-ICP-MS. I frammenti studiati sono datati al 79 d.C. (secondo le indicazioni archeologiche) e appartengono a varie tipologie di oggetti in vetro.

Scopo del lavoro è stato quello di risalire alle caratteristiche tecniche di produzione del vetro (utilizzo di opacizzanti, coloranti ecc.) e della materia prima utilizzata.

I risultati delle analisi hanno messo in evidenza una produzione di vetro piuttosto semplice ottenuta dalla fusione di sabbia pura, del *natron* e di vetro riciclato senza l'aggiunta di altri componenti coloranti, decoloranti o opacizzanti.

Key words: *Roman glass; Pompeii; XRF; LA-ICP-MS; natron.*

Introduction

In Roman time, glass production was widespread and covered many fields. In particular, various discoveries have confirmed the spread of glass, mainly in everyday life, thanks to the possibility of re-using materials and to the low cost of glass-making techniques. In Campanian cities, buried by the eruption of Vesuvius (A.D. 79), glass is found in *domus* in equal if not greater percentage of ceramic tools.

As inferred from writings of the period, such as Pliny's *Naturalis Historia*, from archaeological excavation and chemical analysis of Roman glass from I century B.C. to the VII century A.D., the mixture of raw materials for glass melting included only two major components: sand and *natron*, mixed in suitable proportions.

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Various studies have aimed at identifying the origin of the raw materials and where the glass was produced, according to compositional variation and specific isotope ratios (Wedepohl and Baumann, 2000; Freestone *et al.,* 2003). A common model is that the 'raw glass' was produced in primary workshops in the Middle East, especially Palestine (Huisman *et al*., 2009). Strabo and Pliny, ancient writers, mention the Levant, especially sand from the river Belus, as a major source of raw materials (Humphrey *et al.,* 1998).

The second model proposes local glass-making and working centers employing raw materials locally available and/or imported. For example, Silvestri *et al.* (2006) showed that the Campanian sand is also suitable for glass-making, but only after selective grinding carried out according to Pliny's text, which modifies the pristine ratios among single components.

Huisman *et al*. (2009) suggest that possible diversification of primary production in the Roman period, is demonstrated when large variations between the analysed glass of the same period of time can be found.

In the Pompeii glass, analysed by Vallotto and Verità (2002), modest changes in the concentration of sodium oxide (Na_2O: from 15.5% to 18.5%) have been observed, with a sand/natron ratio (5/2) relatively constant. These results suggest that most of the analysed finds were melted with sand from the same locality and that there was a glass production center in Pompeii. Most of the studied samples from Pompeii showed a remarkable similarity with sand from the river Bélus; the discovery in the Roman city of raw glass with this composition, confirms that the local workshops used raw glass from Middle East. Is not excluded that other sands, anyway different from those of Volturno River, have been probably used for other finds. (Vallotto and Verità, 2002).

This work aims to provide a further contribution to the study on Pompeii glass. The main objective is the chemical characterization of glass fragments by combining two methods of analysis, to identify technological aspects and compositional variations among differing glass objects.

Materials And Methods

The studied glass fragments, found in different *insulae* and houses of the archaeological site of Pompeii, belong to various types of glassware: *balsamaria*, jars, cinerary urns, jugs and a sample of raw glass (Fig. 1). They are dated back to the first century A.D., before A.D. 79, year in which there was Vesuvius eruption. They are opaque decorations free and show the same blue-green

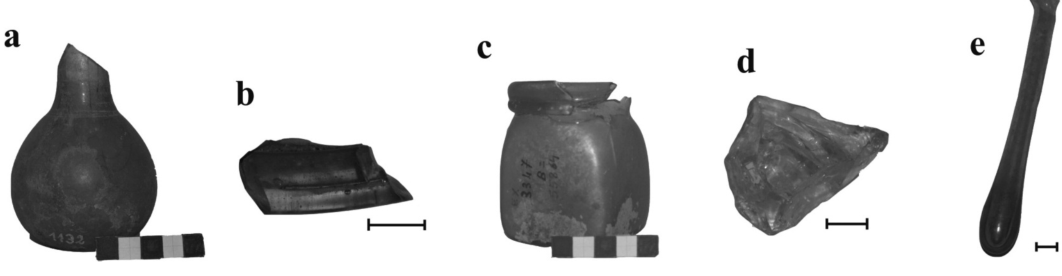

Fig. 1 - Some analysed glass samples - a) *Balsamaria* (UP1); b) Rim of jug (BRP1); c) Jar (BP3); d) Raw glass (RG); e) Drop-shaped *balsamaria* (UP2). (scale is in centimeters)

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

Sample	Type	Date	Colour	Provenance
RG	raw glass	pre-79 AD	blue-green	Insula Occidentalis
BP1	jar	pre-79 AD	blue-green	*Incertae*
BP2	jar	pre-79 AD	blue-green	Menandro house
BP3	jar	pre-79 AD	blue-green	House n. 32
BR4	jar	pre-79 AD	blue-green	Insula 3, Regio V
UP1	*balsamaria*	pre-79 AD	blue-green	Fauno house
UP2	drop-shaped *balsamaria*	pre-79 AD	blue	*Incertae*
UP3	*balsamaria*	pre-79 AD	blue-green	*Incertae*
UP4	*balsamaria*	pre-79 AD	blue-green	*Incertae*
BRP1	jug	pre-79 AD	blue	*Incertae*
OLP1	cinerary urn	pre-79 AD	blue-green	*Incertae*

hue apart two fragments with a more intense blue color. The features of the investigated glasses are summarized in TABLE 1.

Fragments, subjected to chemical XRF and LA-ICP-MS analysis, were previously cleaned of superficial deposits in an ultrasonic bath with Millipore water.

The determination of the major element bulk composition of each sample was determined by X-ray fluorescence (XRF) on a Bruker S8 Tiger spectrometer. The samples was ground to a fine powder in an agate mortar to prepare pressed pellets with boric acid as support. Accuracy was tested on International standards.

LA-ICP-MS analysis was carried out on an Elan DRCe-Perkin Elmer/SCIEX plasma mass spectrometer coupled with a model UP213, Nd-YAG laser (New Wave) to determine trace elements and REE. Small pieces of about 2x2 mm were sampled from each glass and fixed on slides, with the fresh side facing upward. Calibration was carried out on standard glass NIST SRM 612 (50 ppm) produced by the National Institute of Standards and Technology, also to check the quality of analyses the standard glass NIST SRM 610 (500 ppm) was analysed as "unknown". Laser ablation of samples was carried out in the ablation cell with a beam creating a crater of about 50 microns and the vaporized material, was transported by a helium-argon flow to the ICP, where it was quantified (Gunther and Heinrich, 1999).

Lastly, the concentration of SiO_2 for each glass fragment determined by XRF was used, for internal standardization. To assess the accuracy of the analytical data, the mean value of analyses on standard NIST 610, used for quality control, was compared with those reported in the literature (Pearce *et al.*, 1997; Dulski, 2001; Gao *et al.*, 2002). Accuracies expressed as percent

Table 2

Results of XRF analysis performed on 11 Pompeii glass samples. Elements are expressed in wt %.

	RG	BP1	BP2	BP3	BP4	UP1	UP2	UP3	UP4	BRP1	OLP1
SiO_2	66.3	69.2	68.9	69.6	69.3	68.9	69.9	70.1	68.1	70.0	70.2
TiO_2	0.05	0.05	0.09	0.09	0.05	0.09	0.09	0.06	0.08	0.09	0.08
Al_2O_3	1.97	2.08	2.07	2.02	2.06	1.86	2.04	1.90	1.81	1.95	1.98
Fe_2O_3	0.33	0.29	0.61	0.50	0.30	0.51	0.51	0.34	0.43	0.51	0.46
MnO	0.49	0.23	0.39	0.50	0.22	0.48	0.52	0.53	0.60	0.45	0.50
MgO	0.58	0.52	0.62	0.66	0.52	0.60	0.59	0.57	0.53	0.62	0.58
CaO	7.69	7.46	7.35	6.92	7.56	6.84	6.50	7.38	6.82	6.61	6.39
Na_2O	21.9	19.7	19.2	19.0	19.5	20.0	19.0	18.4	20.9	19.0	19.1
K_2O	0.55	0.44	0.71	0.66	0.45	0.64	0.66	0.57	0.57	0.65	0.65
P_2O_5	0.07	0.10	0.14	0.13	0.10	0.12	0.12	0.10	0.10	0.12	0.12
Cl	0.34	0.54	0.41	0.45	0.56	0.44	0.44	0.50	0.47	0.45	0.47

differences between measured and certified values were always less than 10%, and most plotted in the range +/-5%. Contents of Cl cannot be measured by LA-ICP-MS, so XRF analysis has been used to detect these components.

Results

Table 2 and Table 3 show XRF and LA-ICP-MS results on 11 fragments of Pompeii glass. Major elements are expressed in weight per cent of oxides and trace elements in ppm.

Analysed samples are soda-lime silica glass with a SiO_2 content raging from 66,3 to 70,2 wt%, Na_2O concentration from 18,4 to 21,9 wt% and CaO from 6,39 to 7,69 wt%. Al_2O_3 varies from 1,81 to 2,08 wt% instead Fe_2O_3 and MnO occur in concentration 0,29 - 0,61 wt % and 0,22 – 0,60 wt % respectively.

The low potassium and magnesium contents (K_2O = 0,60 ± 0,09 wt%, MgO= 0,58 ± 0,04 wt%) suggest the use of natron as flux for all samples, according to the typical Roman production technology (Sayre and Smith, 1967; Henderson, 1985, 2002; Silvestri *et al*., 2005). Titanium is determined in low concentration for all samples (no more of 0,09 wt%).

Only few trace elements are present in significant quantities in the Pompeii analysed glass: Sb (up to 916 ppm), Ba (up to 283 ppm), Pb (up to 326 ppm), Sn (up to 72 ppm), Sr (385-519 ppm), Cu (up to 374 ppm) and Zr (up to 65 ppm).

As regard the Sb, Cu, Pb and Sn contents, differences are showed among the glass objects. In particular the raw glass (RG) and two jars (BP1 and BP4) contain lower concentrations of Sb (up to 7,66 ppm), Cu (up to 6,73 ppm), Pb (up to 7,19 ppm) and Sn (up to 1,16 ppm) than the other glass samples.

The high amounts of Cl (determined by XRF) in the analysed samples, about 0,46 ± 0,06 % , are also due to *natron* that contains NaCl as a contaminant (Shortland, 2004).

Low levels of Fe_2O_3, Co (between 1,93 and 29 ppm), Sb and Mn confirms the no use of colorizing and decolorizing agents.

Discussion

Major elements

As regards major elements, the samples show the homogeneous composition of Roman glass (mean percentages: 69% SiO_2, 19% Na_2O and 7% CaO), identified as soda-lime silica glass (Sayre and Smith, 1967; Henderson, 1985, 2002; Silvestri *et al*., 2005).

TABLE 3

Results of LA-ICP-MS analysis performed on 11 Pompeii glass samples. Trace elements are expressed in ppm.

	RG	BP1	BP2	BP3	BP4	UP1	UP2	UP3	UP4	BRP1	OLP1
Cr	20.1	10.14	17.4	14.6	10.1	18.9	22	19	18.7	22.8	15.5
Sc	2.89	1.32	3.26	3.52	2.01	3.72	5.03	nd	2.35	4.07	3.03
V	10.8	6.58	12.3	13.2	8.19	11.9	14.4	12.5	13.6	13	12.3
Co	3.31	1.93	11.3	16	2.99	15.1	29	12.7	15.4	21	16.6
Cu	6.73	4.66	332	309	4.30	260	306	28.6	127	374	206
Ni	8.02	4.86	6.37	8.34	5.62	7.30	7.76	8.91	9.25	8.07	7.74
Zn	8.06	11	18.6	20.9	11.1	24.3	19	23.6	26.7	24.5	26.7
As	4.03	2.69	5.29	3.52	2.05	nd	15.6	nd	4.08	6.43	3.86
Rb	8.88	6.38	22.6	9.56	7.45	8.64	12.7	7.62	9.47	11.3	9.27
Sr	519	441	413	412	475	397	472	491	433	463	385
Y	8.23	7.23	7.32	6.90	7.18	6.76	8.59	7.93	7.51	7.98	6.82
Zr	33.7	31.8	49.4	49.3	33.5	53.9	60.3	42.2	44.4	65.4	48
Nb	1.38	1.15	1.82	1.89	1.41	1.81	1.37	1.58	1.66	2.33	1.64
Mo	1.97	1.55	1.21	1.52	1.86	2.18	1.48	1	1.3	2.64	0.96
Ag	nd	nd	0.39	0.31	0.16	0.74	nd	0.36	1.03	0.10	0.20
Cd	5.28	14.3	7	6.4	19	34.36	6.32	nd	7.98	12.5	4.04
Sn	1.16	0.53	62.4	60.3	1.02	37.7	42.9	8.52	24.6	72.2	40.9
Sb	7.66	nd	661	718	0.20	668	894	114	722	916	729
Ba	229	228	225	253	255	233	264	245	263	283	212
La	6.70	6.35	7.56	6.79	6.96	6.64	8.50	7.45	6.93	8.41	6.77
Ce	11.2	11. 5	13.6	12.2	12.3	10.6	14.2	12.2	12.7	15	11.3
Pr	1.63	1.42	1.48	1.55	1.60	1.67	1.87	1.58	1.72	2.09	1.53
Nd	6.52	6.17	6.96	6.85	7.19	7.18	5.89	5.63	7.26	8.34	5.50
Sm	1.47	1.43	1.69	1.14	1.77	2.53	1.56	2.77	2.06	1.65	1.11
Eu	0.30	0.29	0.28	0.30	0.27	0.39	0.98	0.32	0.34	0.26	0.36
Gd	1.09	1.54	1.15	1.14	1.24	1.33	2.50	2.76	1.80	1.65	0.93
Tb	0.23	0.21	0.21	0.20	0.15	0.40	0.86	0.10	0.20	0.16	0.13
Dy	1.10	1.07	1.09	1.20	1.40	1.38	0.32	2.02	1.12	1.66	0.94
Ho	0.20	0.30	0.18	0.28	0.25	0.57	0.77	0.32	0.31	0.25	0.31
Er	0.93	0.50	0.70	0.47	1.07	nd	1.96	0.57	0.98	1.33	0.69
Tm	0.11	0.10	0.09	0.08	0.04	0.33	0.16	0.12	0.13	0.17	0.06
Yb	0.73	0.23	0.87	0.85	0.44	nd	1.40	1.17	0.69	0.33	0.27
Lu	0.09	0.06	0.13	0.12	0.19	0.60	0.33	0.13	0.10	0.12	0.05
Hf	0.60	0.85	1.33	1.30	0.92	1.40	1.88	0.01	1.45	1.87	0.76
Ta	0.17	0.04	0.14	0.14	0.12	0.30	0.32	0.14	0.03	0.21	0.04
Pb	7.19	3.99	326	202	5.32	143	222	31.1	146	202	161
Th	0.88	0.84	1.82	1.21	0.93	1.29	1.61	1.31	1.32	1.51	1.17
U	1.21	1.04	1.09	0.88	1.14	1.04	1.60	1.18	1.07	1.30	1.06

Slight compositional differences between glass samples may be a result of variations in glass production at a single site or the use of chemically similar raw materials at different production sites in the same area.

The MgO versus K_2O plot (Fig. 2a) indicates the use of natron as chemical flux, confirmed by the relatively low MgO and K_2O concentrations, with values not exceeding 1% wt (Mirti *et al.*, 2008).

The SiO_2 versus Na_2O plot (Fig. 2b) shows the considerable homogeneity among the glass fragments. Only the raw glass sample (RG) has lower SiO_2 values (66,3 % wt) and higher Na_2O percentage (21,9 % wt) than the other samples. However the different ratio between SiO_2 and Na_2O, respectively vitrifier and flux, may be explained by considering the different use to which the glass would be put. The intentional addition of Na_2O, favoring glass melting, made raw glass suitable for making objects for casting, such as glass sheet (Verità *et al.*, 2001; Verità, 2004).

The low contents of Al_2O_3 and CaO (Fig. 3a) are indicative of the use of a pure and of good quality, quartz-rich sand, as also shown by the

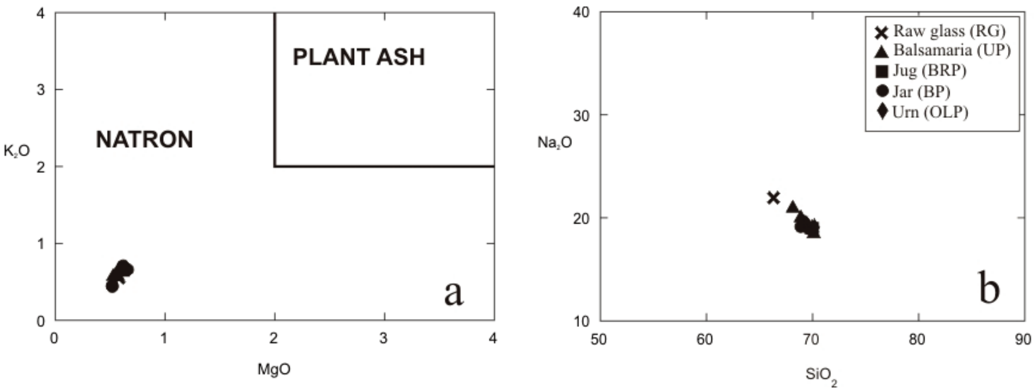

Fig. 2 - Major element concentrations in Pompeii glass - a) MgO versus K_2O; b) SiO_2 versus Na_2O.

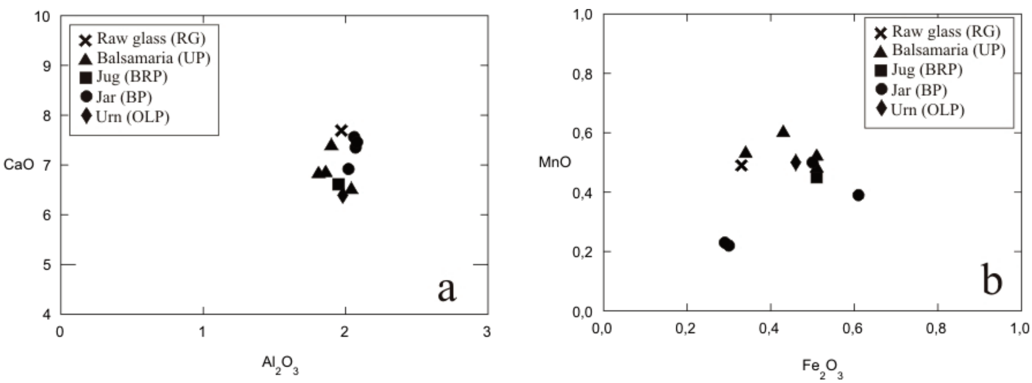

Fig. 3 - Major element concentrations in Pompeii glass - a) Al_2O_3 versus CaO; b) Fe_2O_3 versus MnO.

low Fe_2O_3 (up to 0,62 % wt, Fig. 3b) and trace elements contents (TABLE 3) according to Freestone *et al.*, 2002.

The low percentages of MnO (less than 0.65% wt) show that manganese oxide as decolouring agent was not used as according to common roman glass production (Mirti *et al.*, 2008), but its presence in most of studied samples suggests deliberate addition at some point during the manufacture. Considering that the MnO/FeO ratio in the Earth's crust is about 0,015 (Wedepohl, 1995), instead in the BP1, BP3, UP1, UP2, BRP1 and OLP1 samples it is about 1, the contamination by recycling manganese-decoloured glasses is suggested (Freestone, 2006).

Some correlation exists between the titanium and iron concentrations (TABLE 2), probably introduced into the melting bath as contaminants from sand and flux (Mirti *et al.*, 1993).

Trace elements

The Sb versus Pb diagram (Fig. 4a) shows marked variability among the analysed samples and a high correlation between lead and antimony. The presence of antimony in glass is generally associated with the use of a decolorizer, a procedure which became widespread in glass production in the Middle East from the second half of the first millennium B.C., to the late I century B.C. In Europe, antimony continued to be used beyond the II century AD, together with manganese, which was most frequently used in the I century A.D. (Arletti *et al.*, 2008).

In the studied glass samples, antimony is found in concentrations ranging from a few ppm to about 900 ppm. Its presence as a decolorizer can be excluded, as its content does not exceed 0.2% wt (Jackson, 2005). However the relatively high amounts of Sb in most studied samples could be due by the addition of recycled antimony-decoloured glasses (Freestone *et al.*, 2002).

Even the relatively low contents of Pb confirm that neither lead antimonate or bindheimite ($Pb_2(Sb,Bi)_2O_6(O.OH)$), generally included as decolorizer (Jackson, 2005), was used.

The diagram in fig.4b (Co versus Cu) show in general the low contents of Co (up to 29 ppm) and variable Cu concentrations (below 374 ppm). This confirms that colorizing agents, such as trianite ($2Co_2O \times CuO \times 6H_2O$) found usually in blue glass (Arletti *et al.*, 2008), were not used. The BRP1 and UP1 glass fragments, characterized by a more intense blue hue, show higher contents of Cu and Co, probably due by the employ of recycled glasses (Freestone *et al.*, 2002).

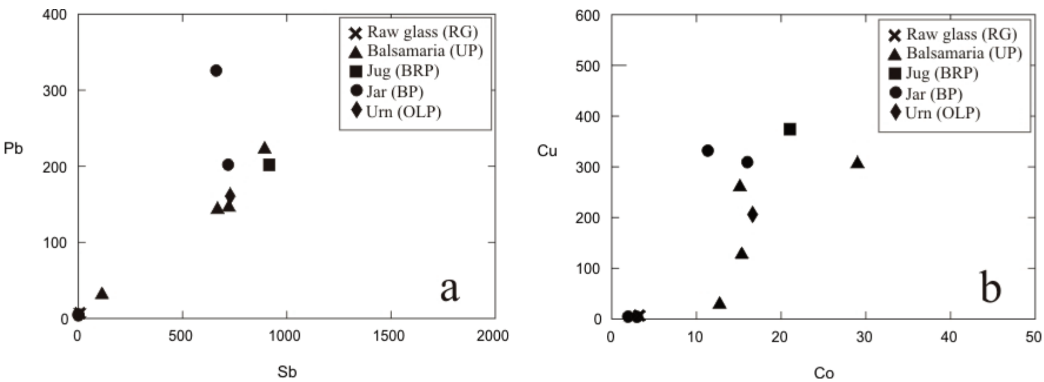

Fig. 4 - Trace element concentrations in Pompeii glass - a) Sb versus Pb; b) Co versus Cu.

Furthermore the presence of Cu, Pb and Sn in different levels and in different relative amounts, could indicate that recycling of bronze slag was a more or less usual practice in glass production (Mirti *et al*., 2009). However the contamination by the manufacture of bronze materials in the same site, cannot be excluded. Independently of the actual origin of copper in glass, it contributes significantly to the development of its blue-green hue.

The two jars (BP1 and BP4) and the raw glass (RG) samples evidence negligible contents of Sb, Mn, Pb, Co and Cu (Fig. 3b, Fig.4a and 4b) suggesting that the analysed glass probably were melted using different proportions of recycled and raw glass or may be related to different secondary productions.

Information on the kind of sand used in ancient glass manufacture can further be obtained by considering the strontium content. The strontium concentration in the samples is not very variable and falls in the range 385-519 ppm and may indicate the use of coastal sand as a source of silica (Van der Linden *et al*., 2009), although isotope analysis is required to confirm this.

Relatively low contents of titanium, zirconium, hafnium and rare earth elements (Table 2 and 3) suggest recourse to a quite pure sand (Mirti *et al.*, 2009).

Conclusions

The preliminary study on Pompeii glass provided information on raw materials and working techniques.

Major elements concentrations defined the glass as silica-soda-lime, with typical homogeneous composition of Roman glass. It was produced with the same pure coastal sand and using *natron*, as a flux.

For the low number of analysed glassware no hypotheses can be put forward concerning the site where the raw glass was produced from raw materials.

The low contents of Fe, Co, Mn and Sb indicate that colouring and decolouring agents were not employed but evidence that recycling of decoloured and coloured glasses was widely practiced in Pompeii town. The presence of Cu, Sn and Pb in glass samples, is attributed probably to the employ of slag from the bronze manufacturing for glass production or as simple contamination.

Trace elements contents could suggest different secondary glass productions in Pompeii or different melting processes in the same workshop.

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