



Mineralogical, Petrographic, and Chemical analyses on small perfume vases found in Messina and dated to VII century B.C.

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Abstract

The archaeological excavations carried out in the area of the Messina harbour and in other urban sites during 1926 have brought to sight, among other materials, some small fictile vessels, perfume containers (aryballoi or lekythoi) and exemplars of open forms (VII century B.C.) till now considered to be a Greek production. All the samples have been subjected to a non-destructive X-ray fluorescence analysis (EDXRF). The obtained data, presented in the form log (element/Zr), have allowed the identification of a samples group which presents, with one only exception (sample C3), homogeneous chemical characters. This has been accomplished by using both binary correlations and a multivariate

cluster analysis. Furthermore, in order to identify the production centres of such materials, till now only supposed on a historical and archaeological basis, petrographic, mineralogical and geochemical analyses have been carried out by means of optical microscopy, X-ray diffraction and Inductively Coupled Plasma-Mass Spectrometry (ICP-MS) measurements. The results have put in evidence the presence of different petrographic features. Nevertheless, the samples are enough homogeneous from the chemical point of view with the usual only exception of the sample C3 as already found by EDXRF measurements. The obtained data by means of petrographic and trace-elements analyses have been compared with those reported in the literature and concerning pottery productions both from Messina and the Greek-Corinthian area. A noticeable relationship between the main group of ceramics and the Messina Strait productions has been found, while the sample C3 may be considered, even if with a poor tie, as belonging to the Greek-oriental ceramics group.

Keywords: small perfume vessel, Strait area, Greece, OM, ICP-MS, EDXRF, XRD.

Introduction

Most of the samples analysed come from a recovery occurred in 1926 in the final part of the peninsula that surrounds the harbour of Zankle-Messana. This recovery has been placed in a military area and it were not extended for many reason (Bacci, 2005).

In particular, such samples come from a group of materials certainly related to a votive warehouse suggesting the presence of a sanctuary. The pieces were found in a hole, probably sunk in (sea?) water and, in part, edited by Paolo Orsi in 1929. The discovery, together with these materials, of a gutter has allowed Orsi to understand that they were all really belonging to a sanctuary datable back to VII century B.C.

Due to their significance in the reconstruction of the economical and religious life of the colony, all the pieces were also studied by Vallet (1958), Neft (1987) and Bacci (1998) who stated, in different occasions, the connections of the findings with the first phases of the colony. Moreover, this last author has in progress a general revision of these materials and their related historical, cultural and archaeological problems. For their importance some of them have been exposed in Athens (Mastelloni M.A., 2003) and Reggio Calabria (Bacci 2004, 2005).

From the historical and archaeological point of view, the greater part of pieces have been supposed to be a Greek production. Only recently, due to the find-

ing of ancient furnaces in the Messina area, a production has been suspected imitating the Greek styles. A number of papers concerning the archaeometry of ancient pottery found in Messina and the mineralogical, petrographic and geochemical characterizations of the attested production in this site (Barone et al. 205a) have suggested the use of further experimental methods in order to support the hypothesis of a local production for such important materials.

Our research was carried out to verify the correspondence between typological similar samples and their similar petrografical and chemical composition; to verify, by mineralogical-petrographic and chemical-physical analyses, the historical and archaeological hypotheses on the possible centres of production; to distinguish the local productions from importation pieces and to differentiate them from those produced in others western mediterranean areas.

Samples and Methods

The investigated specimens are small perfume vases, open shape pieces, samples coming from different urban areas drawn from the Messina Museum collection and other pieces of particular significance both imported from Greek-oriental area and local archaic imitations of Greek-oriental items.

In particular the following samples were analysed:

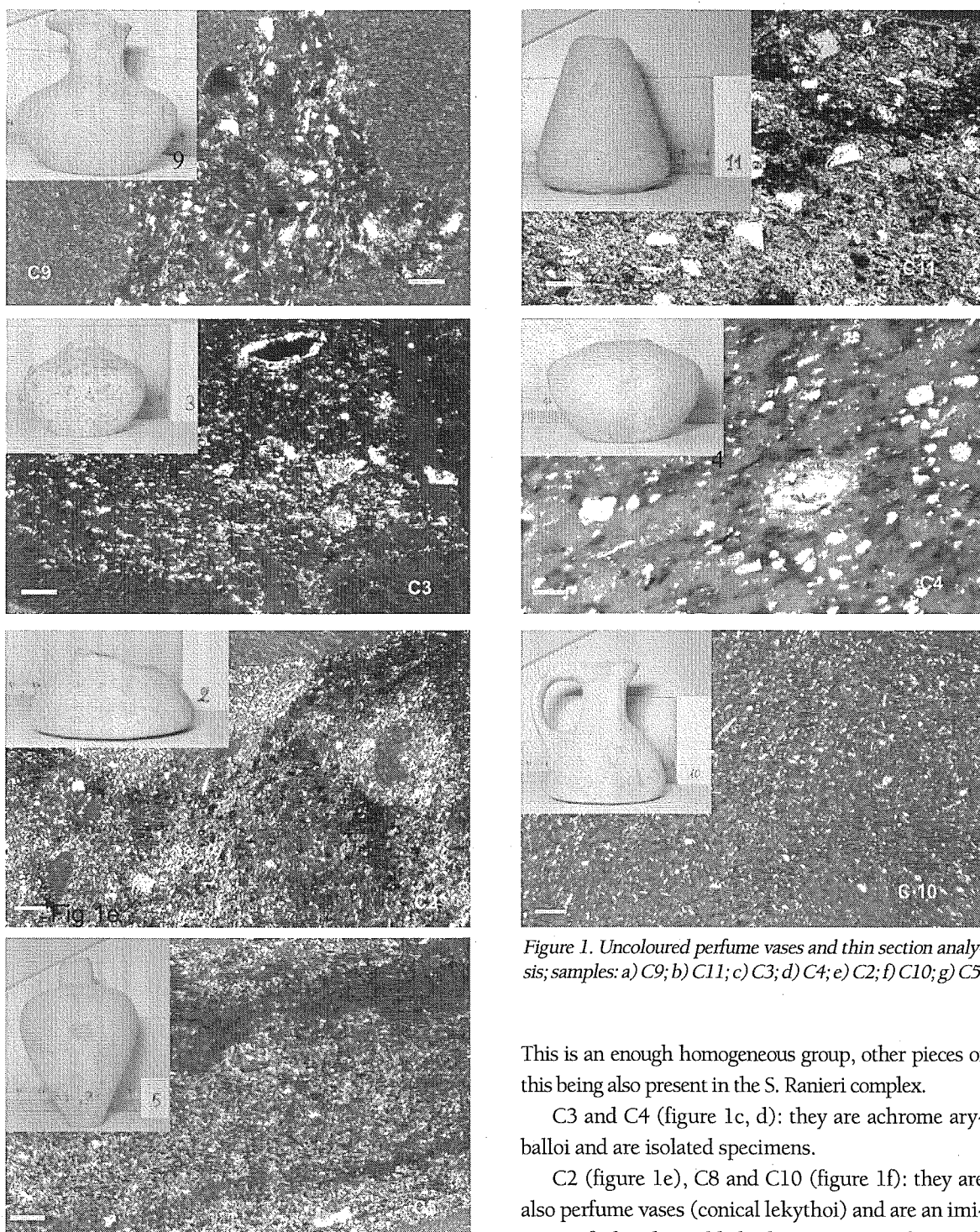


Figure 1. Uncoloured perfume vases and thin section analysis; samples: a) C9; b) C11; c) C3; d) C4; e) C2; f) C10; g) C5.

C1, C7, C9, C11, C12 and CB (figure 1a, b): they are small perfume vases (aryballoi) which imitate in the shape and general style, a pottery class probably produced in Rhodes: the so-called rhodian-cretan aryballoi or KW.

This is an enough homogeneous group, other pieces of this being also present in the S. Ranieri complex.

C3 and C4 (figure 1c, d): they are achrome aryballoi and are isolated specimens.

C2 (figure 1e), C8 and C10 (figure 1f): they are also perfume vases (conical lekythoi) and are an imitation of a hand-moulded achrome pottery class with a stick-finished surface. This class is much diffused and probably has been produced in different zones of Greece, mainly Corinth and Argos.

C5 (figure 1g) and C6: they are ovoidal-type proto-

Corinthian aryballo, but C6 is considered an imitation.

CC: it is referable to the closed-shape pottery, probably a hydria with linear decorations. It is of Aegean or greek-oriental type, enough diffused also in western mediterranean areas.

CA: this piece is a fragment of closed pottery with sub-geometrical type decorations. It seems to be a colonial production whose decoration shows stylistic features very diffused in geometrical and archaical era.

On all the samples, a non-destructive X-ray fluorescence spectroscopy has been carried out. The experimental spectra have been obtained using a Spectra EDXRF system (Mod. QuanX), equipped with an X-ray source (25 keV), an Rh anode and a Si (Li) detector cooled at the liquid air temperature and an energy resolution of about 240 eV. The apparatus was also equipped with a rotating sample-holder to minimize, during the measurements, the effects of non-planar surface of the samples. After the identification of the elements, the intensity of the fluorescence lines have been deduced by a best fit of the spectra, assuming a Gaussian profile and, when necessary, subtracting a linear background under the peaks. A quantitative analysis has been easily carried out, for each element, by comparing its measured intensity (counts) with that one observed in the other samples of the set. All the counts are related to an exposed area of about 346 mm² and a live time of 500 sec for all the samples analysed. Furthermore, when possible, measurements on different areas of the same sample were carried out, in order to have information about the in homogeneity of the mixtures.

Due to their small available quantity, it was possible to carry out the following micro destructive analyses only on few samples (Table I): petrographic analysis in thin section (OM); chemical analysis of trace-elements by a high resolution plasma mass spectrometer HR-ICP-MS (Finnigan-Element 2) carried out on samples solubilized by a microwave digester; mineralogical analysis carried out by a Bruker D5000 X-ray diffractometer utilizing the following working parameters: 40 kV, 30 mA, a Ni-filter, 1 mm, 1 mm, 0.2 mm windows.

Experimental Data and Results

Energy dispersion X-ray Fluorescence Spectroscopy (EDXRF)

In Table 2 the results of the EDXRF analysis are reported as counts per second. This table, with the exclusion of the elements with emission covering the low energy range 1.0-1.5 keV, shows the main constituents as Si, P, S, K, Ca, Ti, V, Cr, Mn, Fe, Ni (but in the samples C1, C8), Cu, Zn, Rb, Sr and Zr, sometime together with traces of chlorine. The presence of chlorine certainly is not related to the clay matrix, but a hypothesis to be confirmed could bring to the seawater content at the discovery time of pieces. The samples C3, C4, C5, C10 and C11 show very low concentrations (not reported in Table I) of arsenic, due to the contaminating effects and/or environment pollution.

A very strong correlations is observed between some elements (for example K-Rb=0.93, Ca-Sr=0.95, Ti-V=0.91, Cr-Ni=0.89); this may be ascribed to the geochemical characteristics of the clay sediment and the mineralogical phases showing that, in spite of the semi-quantitative analysis adopted, the obtained data can be usefully utilized to identify sample groups with homogeneous geochemical characteristics and to obtain a first characterization of them.

The ratios log (element/element) have been deduced from the EDXRF experimental results following the procedure proposed by Aitchinson (1986) and recently utilized by Buxeda (2001) and by Barone et al. (2005b). As a denominator Zr have been used (being a high field-force trace element unlikely mobilized in the post-burial secondary processes). The cluster multivariate statistical analysis (performed by the single bond method and squared Euclidean distances) has been used, has showed a sharp separation between the sample C3 and the homogeneous main group of the other materials (Figure 2a).

Mineralogical and Petrographic analyses

The thin sections analysis has allowed the distinction of the mixtures into two main typologies: The first, coarser, and characterized by inclusion of about 0.2 mm (C7, C9, C11, C12, CB and CC). The second, finer, with inclusion less than about 0.1mm (C2, C3,

Table I

M.I.= Munsell index; OM= optical microscopy; XRD= X Ray Diffraction; EDXRF= Energy dispersion X-ray fluorescence spectroscopy; ICP/MS= Inductively coupled plasma mass.

	typology	M.I.	OM	XRD	EDXRF	ICP/MS
C1	Achrome <i>aryballoi</i>	5YR 5/6	-	X	X	-
C2	Conical <i>lekythoi</i>	10YR 7/3	X	X	X	X
C3	Achrome <i>aryballoi</i>	10YR 8/3	X	X	X	X
C4	Achrome <i>aryballoi</i>	10YR 6/1 e 7/3	X	X	X	X
C5	Ovoidal type proto-Corinthian <i>aryballoi</i>	7.5YR 6/6	X	X	X	X
C6	Ovoidal type proto-Corinthian <i>aryballoi</i> (imitation)	7.5YR 7/4		X	X	X
C7	Achrome <i>aryballos</i>	7.5YR 6/4	X	X	X	X
C8	Conical <i>Lekytion</i>	7.5YR 6/4	-	X	X	X
C9	Achrome <i>aryballos</i> (imitation of Greek- oriental vases)	5YR 6/6	X	X	X	-
C10	<i>Lekytion</i> conica	10YR 7/3	X	X	X	-
C11	Achrome <i>aryballos</i> (imitation of Greek- oriental vases)	10YR 6/4	X	X	X	-
C12	Achrome <i>aryballos</i> di (imitation of Greek- oriental vases)	7.5YR 6/6	X	X	X	-
CVA	Fragment of closet pottery with sub-geometrical type decorations	5Y 7/3	-	X	X	-
CVB		-	X		X	-
CVC	Closet-shape pottery, probably hydria with linear decorations	7.5 YR 7/3	X	X	X	X

C4, C5 and C10).

Samples C7, C9 (figure 1a), C11 C12 and CB (*aryballoi*) show, under crossed nicols, an orange colour mixture which becomes brown when observed by parallel nicols. CB shows a different colouring and a smaller grain in the outside part of the piece. The clayey matrix is heterogeneous and more micaceous in the sample C11 (Figure 1b). The inclusions (mean dimensions of 0.2- 0.6 mm) are not abundant (10-15%) and are constituted by quartz of subrounded or sharp

shapes, plagioclases, K-feldspar, muscovite with rare fibrolithic sillimanite and biotite. CB shows a weak orientation of the present micas. Moreover, oxides and sometime microcrystalline calcites are present. Sample C11 shows vacancies of irregular shape (0.6mm).

C3 and C4 (*achrome aryballoi*) are characterized by different mixtures, but both have a fine grain with inclusion dimensions less than about 0.1mm. C3 presents a brown mixture when observed by crossed nicols and a beige-yellow colour in the case of parallel nicols.

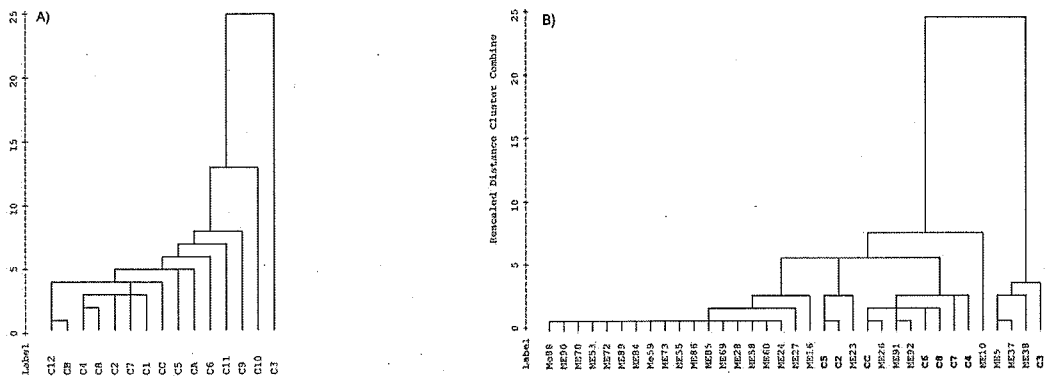


Figure 2. a) Multivariate cluster analysis of EDXRF data ($\log (el/Zr)$); b) Multivariate cluster analysis of ICP MS data ($\log (el/Zr)$) (el =element).

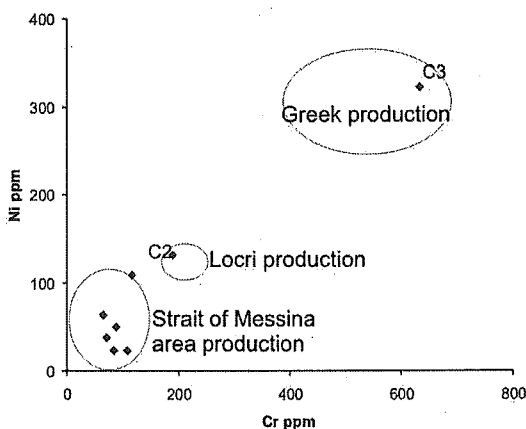


Figure 3. Ni vs Cr diagram (ppm).

The outside portion presents a very fine grain and iron oxides are present. The matrix is heterogeneous and the inclusions are constituted by quartz, siliceous fragments (0.03-0.1mm) and microcrystalline calcite (figure 1c).

C4 shows a light brown-beige mixture (crossed nicols) and a green colour (parallel nicols). The inclusions (20%) have a mean dimension of about 0.1 mm, are constituted by quartz, and zoned plagioclase. Oxides and microcrystalline calcite are also present (figure 1d).

C2 and C10 (lekythion) show a very fine mixture (figure 1e, 1f). The mean dimension of inclusions is about 0.02 mm. The colour is brown-orange (crossed nicols) or yellowish (parallel nicols). The matrix is homogeneous and subrounded quartz (10%), copious muscovite (only in the sample C10) and rare biotite (0.01

mm) are present. Quartz is more abundant (20%) and its dimensions are greater (0.1mm) in the sample C2.

C5 (ovoidal proto-Corinthian type aryballos) has a mixture with a very fine grain (0.02 mm). The matrix is heterogeneous and presents different colorations. The inside portion is dark red (crossed nicols), the outside portion being yellowish. Under parallel nicols the colour is orange-brown. Abundant oxides between the surface and the internal part have been observed in the piece (figure 1g) which is also rich of muscovite but poor of quartz.

CC (hydria) shows a red (crossed nicols) or orange (parallel nicols) mixture. The matrix is homogeneous. The inclusions, whose dimensions are about 0.2 mm, are constituted by microfossils (probably foraminifers) filled by microcrystalline calcite.

Most of the ceramic specimens show inclusions compatible with a Calabrian-Peloritan provenance. Furthermore they are, under many points of view, similar to those produced in the Strait of Messina area (Barone et al. 2002a, Barone et al. 2005a). However, the different types of mixture suggest the presence in this area of different production centres or small workshop characterized by slightly different techniques and raw materials.

The sample C3 shows non local compositional characters leading back to Corinthian B amphoras from Corfù described by Whitbread (1995) and to Corinthian B amphoras from Corinth and Corfù (Barone et al. 2002b, Barone et al. 2004) even if it is different from them due to the characteristics of the texture.

Table II EDXRF data (Major and Minor element analysis as counts per sec).

Elem	Si	P	S	K	Ca	Ti	V	Cr	Mn	Fe	Ni	Cu	Zn	Rb	Sr	Zr
C1	90	43	139	1788	6806	1284	279	100	534	35995	-	460	211	297	1277	623
C2	105	66	405	2466	10287	2149	429	217	727	51260	173	941	258	319	1865	559
C3	100	366	123	727	38670	1570	256	1080	4083	66320	818	2213	405	76	4383	925
C4	114	78	124	2966	9478	1876	381	159	643	40390	57	412	408	405	1453	617
C5	106	28	160	2134	5871	2322	536	345	810	64330	223	1315	287	283	1181	459
C6	68	54	155	1204	6960	1300	297	196	723	36310	58	531	254	227	909	251
C7	78	42	83	2257	6485	1494	291	205	410	47605	38	888	269	341	959	543
C8	109	66	106	1711	6535	1297	309	158	581	28456	-	691	285	243	956	419
C9	192	69	46	4041	7527	3951	894	414	1226	97870	85	1268	773	518	855	690
C10	162	99	31	2572	13140	2916	420	83	859	76570	255	5678	382	349	1379	534
C11	124	63	67	3502	9789	2376	376	135	617	73380	14	720	66	444	1125	692
C12	76	71	36	2359	8969	1620	300	48	666	59745	50	251	341	338	1161	603
CA	186	148	89	1698	14450	2082	395	67	840	67850	61	155	279	162	1263	805
CB	125	118	57	2482	12130	1731	303	72	674	65220	66	352	449	389	1508	719
CC	135	268	51	2700	25220	1829	337	90	2048	61730	64	473	429	280	2389	783

Table III ICP-MS trace element analysis of selected samples, in ppm.

	V	Cr	Co	Ni	Cu	Zn	Rb	Sr	Y	Zr	Nb	Ba	Pb	Th	U	Cs	La	Ce	Pr	Nd	Sm	Eu	Gd	Tb	Dy	Ho	Er	Tm	Yb	Lu	Hf	Ta
C2	108	190	21	131	1,890	108	128.2	638.2	23.4	143.3	15.9	429.6	16.3	12.4	3.1	259.0	30.0	58.3	6.7	25.2	5.2	1.1	4.3	0.7	4.1	0.8	2.7	0.4	2.7	0.4	4.1	1.2
C3	63	633	80	322	560	133	28.0	597.5	17.3	86.2	11.8	143.0	118.1	5.0	2.8	166.8	18.3	34.4	4.0	14.9	3.3	0.8	2.9	0.5	2.7	0.5	1.7	0.3	1.5	0.2	2.1	0.9
C4	61	65	13	63	166	165	160.1	449.8	32.0	205.8	20.2	445.0	51.8	14.9	3.4	9.8	36.8	76.5	8.6	31.7	7.0	1.2	5.7	1.1	5.5	1.1	3.4	0.5	3.1	0.5	5.7	1.9
C5	68	116	25	109	224	75	68.0	264.0	13.6	87.1	13.5	179.3	34.4	7.0	2.8	139.5	17.1	33.3	3.8	14.2	3.1	0.6	2.4	0.4	2.3	0.5	1.6	0.2	1.4	0.2	2.4	0.9
C6	62	84	16	23	107	30	58.5	201.2	12.8	86.1	9.2	237.9	17.0	6.0	1.7	3.8	18.7	38.1	4.2	15.9	3.3	0.6	2.6	0.4	2.2	0.4	1.3	0.2	1.2	0.2	2.3	0.8
C7	82	72	10	38	233	34	123.9	304.0	24.6	191.9	14.4	506.4	43.5	14.3	2.9	129.8	35.5	71.9	8.2	29.9	6.3	1.1	5.1	0.9	4.5	0.8	2.7	0.4	2.5	0.4	5.6	1.4
C8	85	108	6	23	41	30	115.2	364.2	21.8	143.8	15.2	494.7	21.4	11.7	2.6	5.1	36.5	72.8	8.1	29.0	6.2	1.2	4.7	0.8	4.1	0.7	2.3	0.3	2.1	0.3	4.4	1.2
CC	93	88	15	50	65	75	109.4	688.8	25.6	198.0	19.1	488.6	24.1	12.2	3.2	71.3	39.8	78.1	8.7	31.7	6.6	1.4	5.3	0.9	4.5	0.8	2.7	0.4	2.5	0.3	5.6	1.5

The qualitative valuation of the mineralogical composition in analysed ceramics was determined by means of XRD. Quartz and feldspars are present in all samples, while calcite is observable in C1, C2, C3, C4, C5, C7, C10 and C11 and dolomite in C3 and CA. New formation Ca-silicates are observed in C6, C8, C10, CA, CC (diopside) and in CC (gehlenite). The mineralogical composition of the studied pottery indicate a low (<800°C) firing temperature with the exceptions of diopside and gehlenite ceramics that suggest higher one.

Inductively coupled plasma mass analysis (ICP/MS)

The ICP-MS measurements concern only some samples (the only ones that have allowed the drawing of suitable material quantities) representative of each group. This analytic procedure has been used in order to have useful data for the identification of the possible production centres. The results for minor elements are reported in Table II.

The sample C3 shows a different composition with respect to the other analysed samples. In particular, greater concentrations of Cr (633 ppm), Ni (322 ppm), Co, Pb and low Rb, Ba have been detected (Table III). Between the samples of the main group, C2 presents specific characters essentially due to higher Ni (131 ppm), Cr (190 ppm), V, Cu, Rb and Cs.

In the Ni vs Cr diagram (figure 3) the analysed data compared with the Strait of Messina and Locri-an area production and the Corinthian one are reported. These two elements allow distinguishing the Greek-oriental productions from the southern Italy ones; the investigated samples present a composition similar to the one of the Messina area productions (Barone et al 2005a), with the exclusion of the sample C3 that shows a chemical composition similar to the one of the Greek ceramics (Barone et al. 2002b; Farnsworth et al. 1977; Jones 1986) even if with higher Ni and Cr contents. On the contrary, C2 specimens show analogies with Locri (Calabria) ceramics

(Barone et al. 2005a). Further analyses are in progress in order to better qualify this compositional difference.

The obtained chemical data have been compared with reference samples (figure 2b); here a cluster analysis shows a sample subdivision in two groups: C3 belonging to the Greek production group, all the others to the Strait of Messina production zone.

Conclusion

The analyses carried out on a group of important findings dated back to VII century b.C. and discovered in Messina during 1926, have allowed to state that in the examined warehouse, not only materials imported from corinthian and Greek-oriental area are present, as supposed on an archaeological basis, but also groups of locally made pieces that we conventionally define "Strait area" or, in any case, "colonial".

Pottery of different provenance differs in the clay pastes type, tempers nature, minor and trace elements abundances. In some cases, workshops operating in the Strait area have been identified and recognized – for the first time – as producers of imitations of certain types of ceramics (aryballoi and lekythoi) manufactured in Greece or in the greek-oriental area.

In particular, the aryballoi C1, C7, C9, C11 and C12 form an enough homogeneous group (present with a number of exemplars in the peninsula of S. Ranieri in Messina) which imitate – on the whole – a class of greek-oriental vases probably produced in Rhodes but, on the contrary, resulting as a production of the Strait of Messina area.

The C3 and C4 have archaeological and clay paste characteristics alike, but clearly different from the previous samples. However these two aryballoi present different chemical and petrographic characteristics. The data of the sample C3 exclude a Messina area production suggesting a Greek origin. In particular, this sample is comparable with the Corinth or Corfu transport amphorae widely spread in all the Mediterranean. On the contrary, as few notices are reported for the production and commercialisation of such a miniature fictile vessels, further sampling and analysis are necessary.

C2, C8 and C10 specimens are conical lekythoi probably produced in different zones of Greece, (namely Corinth and Argos). The archaeometric analyses show the production in the Messina strait area sites: the site of Locri is heavily addressed for sample C2.

C5 and C6 are protocorinth aryballoi imitations produced in the Strait area. This type of ceramics were largely commercialised, and the local imitations were very diffused in Messina and in other Greek colony: it is worthy noting that the commonest reproduced pottery it regards daily use artefacts as glasses and pouring vases.

The production of the Aegean or eastern Greek hydria sample (CC) and of the sample CA is stylistically conduible to previously reported archaeological Sicelioti centres as Naxos.

Finally, samples referred to imported productions on a typological basis, after archaeometric data have been – on the contrary – interpreted as very refined local productions very similar in technical and stylistic characteristics to the Greek area original ones. This lead to the hypothesis of a local (in the Strait of Messina area) settlement of skilful artisans that brought together production and firing knowledge.

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