Major and trace element characterization of Archaic and Roman pottery from Achaia, Greece

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ABSTRACT Major and trace element data for Archaic and Roman ceramics from Achaia and for local clay materials which were probably used for their production are examined with the aim to discriminate between pottery productions in space and time. A comparison of the compositions of the Roman ceramics with those of the Archaic reveals that the latter tend to have lower concentrations in Cs, Rb, K_2O, Na_2O and CaO and higher in Al_2O_3. Despite their differentiation due to variation in these major and trace element contents the multielement geochemical patterns of the analysed Archaic and Roman sherds are very similar and fit well to the geochemical patterns of the local raw materials.These similarities suggest the use of the same kind of raw materials for their production.

1. Introduction

Very few archaeometric studies dealing with Archaic and Roman ceramics found in northwestern Peloponnese have been published so far. (cf Hein et al., 2002; Jones, 1986, Rathossi et al., 2003a). The present work reviews and reports new results for major and trace element compositions of Archaic and Roman ceramic sherds from Achaia county, Greece and for the compositions of local raw materials, collected from five locations of the region, which were probably used in the past for their production (Figs. 1, 2). Previous analytical results from this on going programme on the Achaia ceramics have been concerned mainly with the technology and composition of Roman ceramic red-painted and unpainted lamps from two workshops and one Lychnomanteion, excavated in the area of Patras (Petropoulos, 1999). Petrographic observations of Roman ceramics from Patras, led us to distinguish five fabric classes, depending on paste texture, quantity of temper and phyllosilicate minerals present.Based on major element composition of the sherds, calcareous clays fired under oxidizing conditions, at temperatures varying

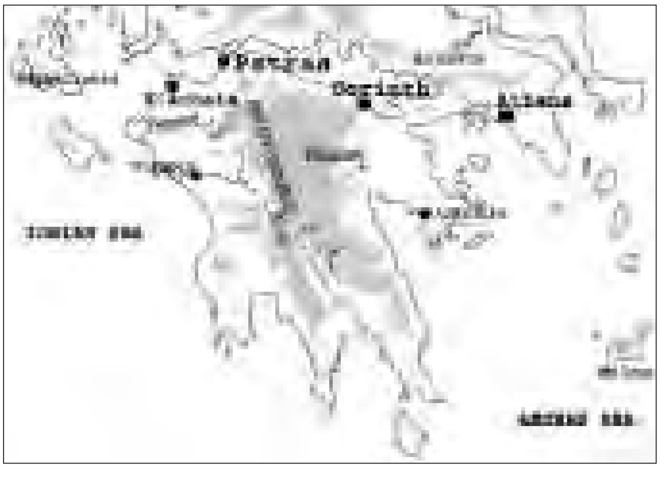

FIG. 1 – Geographical map showing the locations of excavations.

from 800°C to 1050°C, were suggested to be the raw materials used for their production (Rathossi et al., 2003, 2004).

The main purpose of this work, concerning the major and trace element characterization of the Archaic and Roman ceramics and clay materials from the surrounding area, was to establish distinct geochemical patterns for each of the ceramic groups and raw materials which can be used as a "fingerprint" for their identification.

2. Locations of the ceramic samples

2.1. Roman pottery

The studied sherds of Roman lamps were collected from three excavations in the city of Patras (Fig. 1), two pottery workshops and one Lychnomanteion (Petropoulos, 1999).

Workshop A
It was branch of an Italian workshop and produced red painted lamps which were copies of italian lamps. It operated during the second half of the first century AD.

Workshop B
It was a successor to workshop A. It produced unpainted lamps similar to Corinthian lamps and operated from the late first-early second century AD until the early third century AD.

Lychnomanteion
It was located near the port of Patras. It operated from the middle second century AD until the late third-early fourth century AD. The excavation has unearthed a large deposit of unpainted lamps, similar to those of Workshop B, but not of the same high quality.

2.2 Archaic pottery

A deposit of Archaic ceramic sherds, dating from the late seventh-early sixth century BC has been unearthed in the excavation of an Hellenistic settlement (third-second century BC) in the city of Kato Achaia (Ancient Dyme) (Fig. 1). These sherds represent individual wares of Archaic pottery such as: skyphoi, pinakia and krateres, and they display typological influence by Corinthian wares. Macroscopic observation indicated that the fabric and the colours of sherds do not differ from the Hellenistic pottery of Ancient Dyme, and probably they were products of a local workshop. The archaeometric study of this deposit's sherds is very important, due to the fact that it is the only evidence that Ancient Dyme has been inhabited during the Archaic period (Vassilogamvrou, 1998).

3. Geological setting and raw materials

The northwestern coast of Peloponnese comprises the following lithofacies (Fig. 2): The Pre-Neogene basement consists of limestone, flysch and chert of the Pindos and Gavrovo isopic zones of western Hellenides (Aubouin, 1959).

According to Zelelidis et al. (1988) and Doutsos et al. (1988) the Plio-Pleistocene sediments are distinguished into:

1.Grayish fossiliferous lacustrine lagoonal marine sediments consisting of alternating very fine grained sand, silt, silty clay, sandy silt and clay in massive beds, coarsening upwards, with abundant coal. Their mineralogical composition is characterized by the presence of the following sheet silicate parageneses: chlorite-smectite+illite or vermiculite-smectite+illite and mixed layer chlorite-vermiculite. Quartz, albite, K-feldspar and calcite are also present (Tsolis-Katagas, 1992).
2. Yellow terrestrial fluvial deposits. This coarse sequence consists of a thick conglomerate with lenses of sand at the base, followed by conglomerate, sand and minor silt, fine to very fine sandy silt, silt, silty clay. Their mineralogical composition is characterized by the presence of :illite-smectite+illite, quartz, albite and K-feldspar.
3. Alluvial fan deposits. This facies consists of a poorly sorted conglomerate with matrix sand. The clastic material of these formations includes sandstones, cherts and limestone gravels, lithologically connected to the local geology. Mixed layer vermiculite+smectite-illite, quartz, albite, K-feldspar and calcite are present in members of this deposit. The above lithofacies are uncomformably overlain by:
4. Reddish terrestrial fluvial deposits of lower to middle Pleistocene age, consisting of alternating layers of conglomerate, sand and silt fining upwards. Sandy marine terraces are present on the top of the latter deposits.

FIG. 2 – Generalized geological map of northwestern Peloponnese.Numbers refers to sampling locations. Geological boundaries after Zelelidis et al. (1988) and Loftus and Tsoflias (1971).

Nowadays, brick factories operating in the area are using the Plio-Pleistocene sediments for their production. We have collected and studied 63 samples of raw-materials from these deposits supposing that ancient potters could have used these sediments for the production of their ceramic wares. The samples have been collected from three quarries of modern brick factories and from two physical outcrops. Their locations are shown in the geological map (Fig. 2).

4. Analytical methods

Geochemical analyses were carried out using:

a) Inductively Couple Plasma Optical Emission Mass Spectometry (ICP-OES) for major elements and some trace elements (SiO_2, Al_2O_3, CaO, Fe_2O_3, MgO, MnO, TiO_2, K_2O, Na_2O, P_2O_5, Ba, Sr, Y, Zr, Be, V) at ACTLABS, Ancaster, Ontario, Canada. 77 ceramic samples and 39 raw materials samples have been analyzed. Detection limit for major elements is 0.01%. The analytical precision calculated from replicate analysis of one sample is better than ±1%.
b)Neutron Activation Analysis (NAA) for 23 elements (As, Ca, Co, Cr, Cs, Fe, Hf, K, Na, Rb, Sb, Sc, Ta, Th, U, Zn, La, Ce, Sm, Eu, Tb, Yb, Lu) at the NCRS "Demokritos", Athens, Greece. 41 ceramic samples and 29 raw materials samples were analyzed following the procedures described by Kilikoglou et al. (1990).

Due to small size of the available sherds (≈ 3-4 cm in diameter), the total amount of the pulverized sample which has been used for XRD, ICP-OES and NAA analyses was not more than two grams.

5. Results and discussion

The chemical compositions of the raw materials probably used for pottery production and the composition of the ancient ceramics (Table 1-3) can be fingerprinted or classified by basic, geochemical techniques. A large variety of elemental scattergram plots, utilizing the major elements, trace elements and the combination of both has been used. Most of these plots provide clear delinetion of the raw materials and Roman and Archaic ceramics. Roman lamps form a chemically very tight homogeneous group, together with some of the raw materials, mainly those collected from quarries of modern brick factories. Archaic wares form a chemically less tight group of ceramics in which commonly one or two samples (A881, A882) separate (Figs. 3, 4).

There are some very significant plots, such as K_2O-Na_2O, TiO_2-Fe_2O_3, Rb-Al_2O_3, Cs-K_2O, Ta-Sc, Sc-Th, Co-Cr, Y-Th (Figs. 3, 4), which provide sufficient information to distinghuish Archaic wares from Roman lamps and raw materials, but there are no scattergram plots showing clear distiction between Roman lamps from different excavations. The Archaic sherds, compared with the Roman ceramics, have lower Na_2O, K_2O, CaO, Rb and Cs concentrations and are more rich in Al_2O_3, Fe_2O_3 and TiO_2 and in trace elements Y, Th, Cr, Sc, Ta (Figs. 3, 5b). The elements mostly affected by the alteration processes in ceramics are Ca and the alkali metals Cs, Rb, K and Na. Cs and Rb are also considered to be soluble elements and are depleted easily from the glass phase (Schwedt et al., 2004; Buxeda i Garrigó et al.,

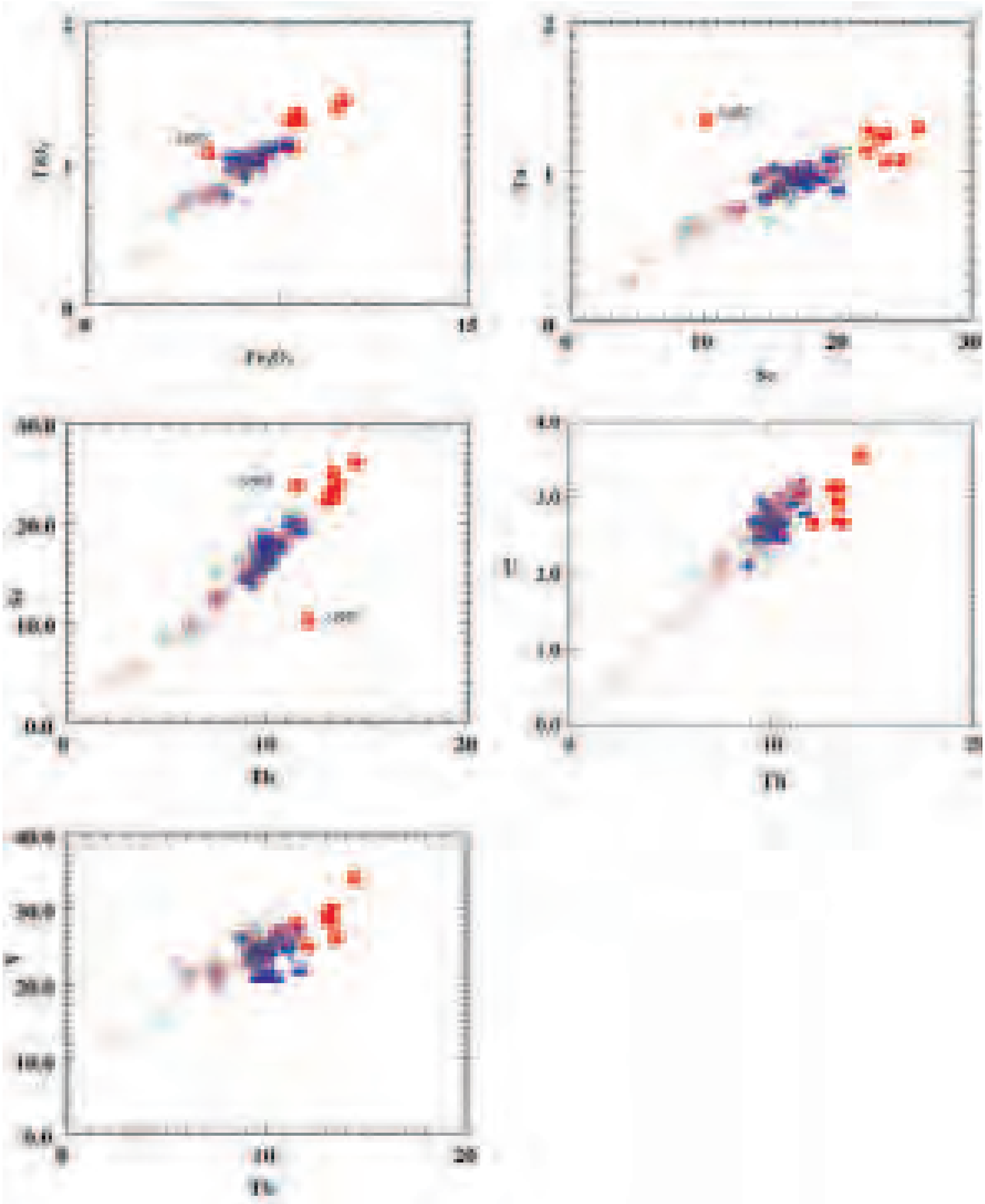

FIG. 3 – Scattergrams showing the variation of major and trace element concentrations in ceramics and raw materials. Major-element oxide concentrations are in wt %, trace-element concentrations are in ppm. Filled circle: Archaic ceramics. Filled diamond: Roman lamps-workshop A. Filled triangle: Roman lamps-workshop B. Half filled square: Roman lamps-Lychnomanteion. Cross: Raw materials (1). Open diamond: Raw materials (2). Open square: Raw materials (3). Open triangle: Raw materials (4). Open circle: raw materials (5). Numbers refer to sampling locations (see geological map, Fig. 2).

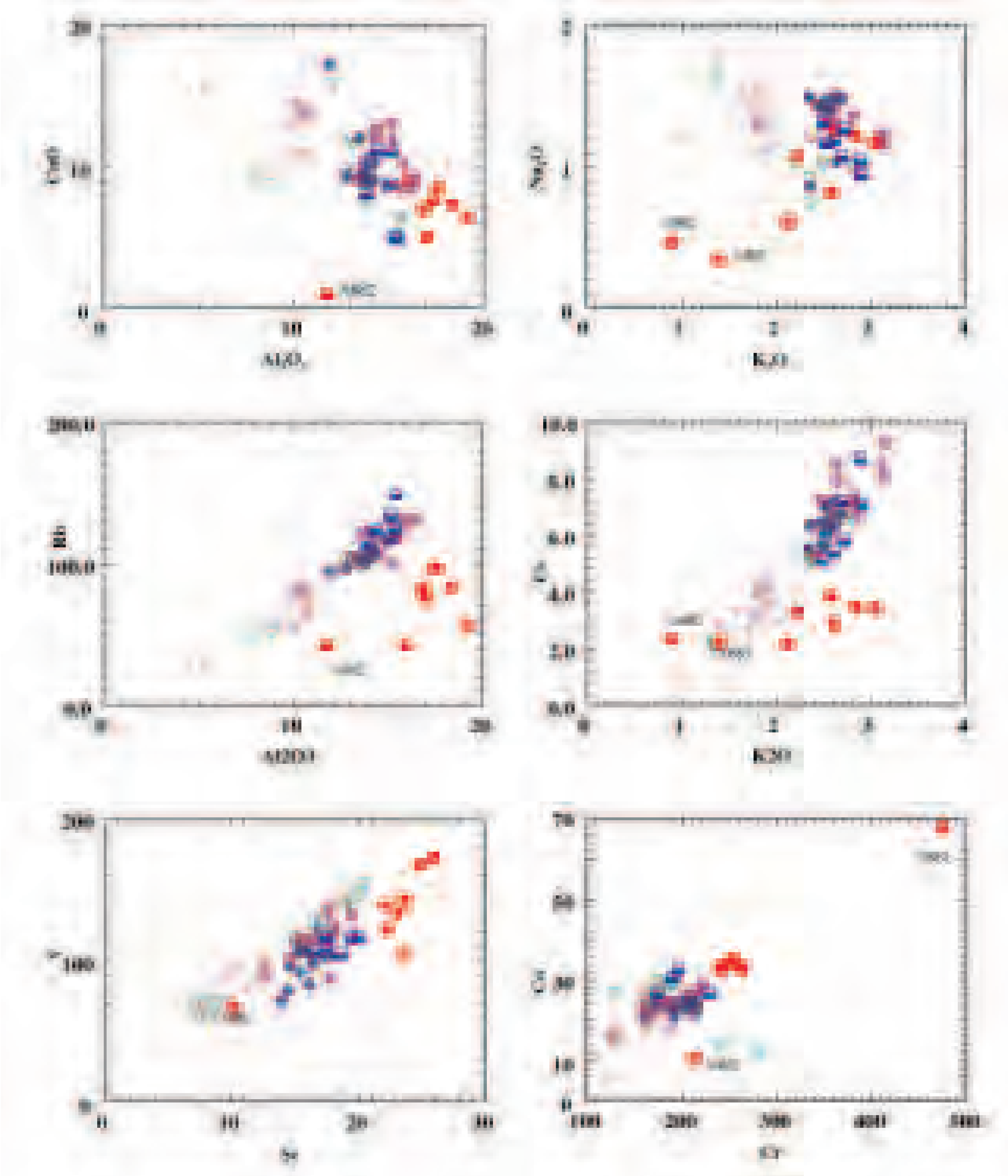

FIG. 4 – Scattergrams showing the variation of major and trace element concentrations in ceramics and raw materials. Major-element oxide concentrations are in wt %, trace-element concentrations are in ppm Symbols as in Fig. 3.

2002; Picon, 1976, 1991). These elements have been found to be reduced in the Archaic samples. However, the latter samples show no signs of any kind of alteration or corrosion, and as they have been fired at low temperatures (T≈800°C), the structure of the mica, which is known to resist to chemical weathering (Lee, 2002), is still preserved and the glass phase is minor. The only secondary phase which has been detected petrographically and by XRD is calcite, but it occurs in Roman samples only. It is therefore evident that the depletion of the Archaic ceramics in these elements could not be attributed to alteration processes occurred during their burial.

Although both Roman and Archaic ceramics could be characterized as fine wares, petrographic observations revealed significant differences in the grain size of the minerals and their distribution between the two types of samples. The mineral grains of the

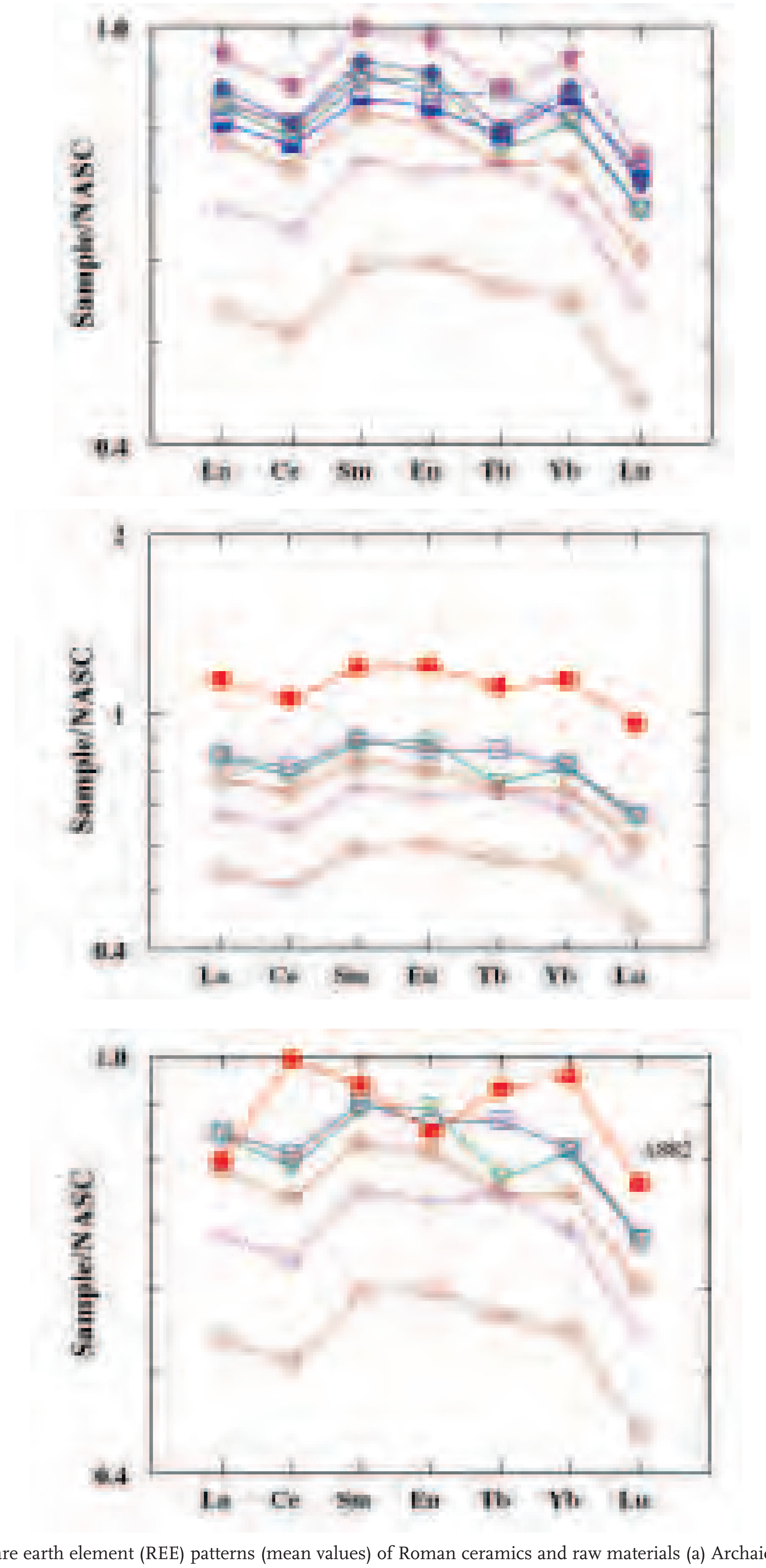

FIG. 6 – Rare earth element (REE) patterns (mean values) of Roman ceramics and raw materials (a) Archaic ceramics and raw materials (b) and of sample A882 and raw materials (c); data normalized to NASC (North American Shales). NASC normalized data are from Cromet et al. (1984). Symbols as in Fig. 3.

Roman ceramics are of silt size (2-20 μm), of very fine sand (50-100 μm) or medium sand size (200-500 μm); but the majority of them are of fine sand size (100-200 μm). The grain size of the archaic samples ranges from silt (2-20 μm) to very fine sand size (50--100 μm). No mineralogical differences have been observed between Roman and Archaic ceramics. Quartz, feldspar, white mica, biotite-like mica, iron oxides and calcite are the main phases present but the Archaic ceramics contain smaller amounts of detrital feldspars. Ilmenite, sphene, zircon, rutile, spinel, epidote, apatite and monazite are accessory minerals which have been identified by SEM in some of the samples (Rathossi et al., 2004).

The concentrations of Cs and Rb are controlled mainly by the presence of K-feldspar and mica where Cs and Rb substitute for K. This observation coupled with their low K_2O and Na_2O content and their high Al_2O_3 content lead us to suggest that during the preparation of the raw materials for production of the Archaic wares, potters followed a procedure, probably "levigation" and obtained the fine fractions of the clays separating the less fine non-plastic particles such as K-feldspar and albite.

The enrichment of elements Y, Th, Cr, Sc, Ta in the Archaic wares is probably due to the silt size fractions since these elements are usually accommodated in clays (Cullers, 1988). The positive correlations observed between TiO_2-Fe_2O_3, Ta-Sc, V-Ta in the scattergram plots (Figs. 3, 4) support a genetic relationship for sphene and ilmenite, and between U-Th, Th-Sc and Y-Th for monazite and zircon. The Archaic ceramics A881 and A882 are outliners in some of the plots. The former contains grog in its matrix and has grains of coarse sand size. Its high Cr and Co content is probably due to the presence of spinel. Sample A882 is a very coarse ware, full of grains of quartz,chert and a few grains of pyroxene and epidote. It is very rich in silica and poor in Al_2O_3 and K_2O and trace elements.

The REE, Th and Sc are considered to be the most confident elements for provenance studies of ancient ceramics because these elements are insoluble and the effects of metamorphism, weathering and diagenesis upon them are minor (Cullers, 1988; Nesbitt et al., 1990; Rollinson, 1993).

The multi element variation diagrams (spidergrams) of trace and some major elements normalized to the composition of north American shales (NASC) (Gromet et al., 1984), show also differences in the relative abundances of the elements between raw materials and Archaic and Roman ceramics. The lines joining the data points of the analyses for each of the ceramic and raw materials groups have patterns showing a striking resemblance to each other (Fig. 5a, b).

On the normalized REE diagrams, the Archaic pottery sherds show an enrichment over NASC whereas the patterns of the Roman pottery and raw materials show little and marked relative depletion over NASC, respectively. With the exception of a Tb negative anomaly observed in the Roman lamps and in clays collected from sampling areas 2 and 4, the patterns of their REE diagrams are very similar (Fig. 6a, b) showing that probably the raw materials used by ancient potters for the production of ceramics are local. From the Archaic ceramics only the sample A882 appears to have different patterns of its normalized REE indicating that it may be an imported product (Fig. 6c).

Despite the differentiations due to some variation in element concentrations, most of the analyzed pottery and raw materials present chemical similarities pointing to the use of the same kind of raw materials for producing the ceramics. Thus, the Archaic ceramics could be connected to the local production even though no kilns have been found so far.

TABLE 1

Major and trace elements ICP-OES analytical results for ancient ceramics and raw material sample (in wt. % and ppm).

Samples ceramics	SiO_2 %	Al_2O_3 %	Fe_2O_3 %	MnO %	MgO %	CaO %	Na_2O %	K_2O %	TiO_2 %	P_2O_5 %	LOI %	TOTAL %	Ba ppm	V ppm	Zr ppm	Y ppm	Sr ppm	Be ppm
Roman WA																		
Mean (n=29)	57,65	13,9	6,29	0,12	3,17	9,21	1,4	2,53	0,75	0,34	4,46	99,81	364	87	167	23	203	1,8
St.Dev	2,82	0,77	0,5	0,01	0,35	1,84	0,19	0,12	0,05	0,27	1,9	0,36	32,47	30	20	7	76	0,6
Roman WB																		
Mean (n=22)	55,22	14,88	6,9	0,12	3,44	10,1	1,29	2,78	0,79	0,31	3,77	99,59	406	115	169	25	201	2,1
St.Dev	2,13	0,87	0,43	0,01	0,27	1,63	0,14	0,23	0,03	0,13	2,18	0,53	57,06	13	19	1	19	0,3
Roman Lych.																		
Mean (n=16)	54,92	14,61	6,77	0,1	3,15	9,17	1,2	2,56	0,79	0,22	6,24	99,73	403,11	119	135	22	163	2
St.Dev	2,84	0,8	0,66	0,02	0,41	2,34	0,18	0,23	0,03	0,05	3,12	0,54	26,91	6	21	1	44	0
Archaic A																		
Mean (n=10)	55,84	16,84	8,17	0,13	3,92	6,88	0,94	2,32	0,95	0,31	3,73	100,04	445,75	131	164	29	171	2,5
St.Dev	6,18	2,01	1,42	0,03	1,3	2,47	0,35	0,69	0,08	0,14	3,34	0,19	81,88	34	39	3	51	0,5
RAW MATERIALS																		
Location TH (1)																		
Mean (n=13)	47,99	10,21	4,74	0,11	2,62	13,52	1,34	1,78	0,57	0,12	16,65	99,63	244,67	89	129	21	201	2
St.Dev	465	0,46	0,71	0,03	0,34	1,52	0,17	0,15	0,03	0,01	2,18	0,48	32,24	8	19	1	18	0
Location K (2)																		
Mean (n=2)	45,78	12,27	5,3	0,09	2,98	12,62	1,06	2,21	0,63	0,12	17,04	100,11	266,5	116	118	21	203	2
St.Dev	10,07	2,18	1,02	0,01	0,75	2,5	0,64	0,58	0,08	0	3,46	0,14	17,68	36	42	0	21	0
Location MP (3)																		
Mean (n=1)	46,53	13,28	5,92	0,107	3,57	9,85	1,21	2,49	0,715	0,11	15,54	99,33	275	127	105	24	105	2
St.Dev																		
Location G (4)																		
Mean (n=10)	48,26	12,91	5,9	0,11	3,18	8,68	1,09	2,31	0,69	0,1	16,71	99,95	306,78	119	126	24	158	21
St.Dev	7,51	2,63	1,37	0,02	0,63	3,18	0,28	0,56	0,12	0,01	3,83	0,24	112,9	33	37	4	36	0,6
Location T (5)																		
Mean (n=4)	57,13	9,48	4,13	0,08	2,27	10,65	1,36	1,66	0,52	0,09	12,78	100,16	210,75	64	61	15	128	1,3
St.Dev	7,67	3,57	1,68	0,02	0,92	4,47	0,18	0,63	0,21	0,03	2,4	0,23	159,24	54	4	11	93	1

Mean concentration values and standard deviation. (WA, WB, Lycn: Workshop A, B, and Lycnomanteion, respectively).

TABLE 2

Trace elements NAA analytical results for ancient ceramics samples (in wt. % and ppm).

Elements	Roman lamps WA		Roman lamps WB		Roman lamps Lycn.		Archaic		Archaic
	Mean (n=12)	St.Dev.	Mean (n=13)	St.Dev.	Mean (n=8)	St.Dev.	Mean (n=8)	St.Dev.	A882
U ppm	2,66	0,24	2,79	0,24	2,67	0,17	2,97	0,31	2,65
As ppm	6,95	1,75	5,66	2,19	11,12	6,07	6,41	3,06	9,2
Sb ppm	0,65	0,13	0,69	0,07	0,95	0,44	0,52	0,05	0,56
Ca %	6,9	1,44	7,11	1,26	5,47	2,22	4,51	1,78	0,7
Na %	0,77	0,12	0,72	0,13	0,8	0,09	0,64	0,27	0,4
K %	2,16	0,16	2,33	0,31	2,19	0,21	1,83	0,57	0,79
Th ppm	9,66	0,54	10,44	0,71	10,35	0,71	13,05	0,89	12,1
Cr ppm	193,25	18,87	197,92	19,61	200,37	15,94	275,12	81,74	215
Hf ppm	4,33	0,48	4,16	0,41	3,93	0,3	4,82	1,1	7,43
Cs ppm	6,44	0,65	7,3	1,06	6,56	1,17	2,92	0,65	2,29
Sc ppm	15,42	0,95	17,37	1,53	18,11	1,14	22,02	4,94	10,2

TABLE 2 [cont.]

Elements	Roman lamps WA		Roman lamps WB		Roman lamps Lycn.		Archaic		Archaic
	Mean (n=12)	St.Dev.	Mean (n=13)	St.Dev.	Mean (n=8)	St.Dev.	Mean (n=8)	St.Dev.	A882
Rb ppm	104,5	5,93	115,85	10,45	126,75	14,48	71,5	22,25	41
Fe %	4,27	0,28	4,73	0,38	4,92	0,43	5,98	1,21	3,22
Zn ppm	100,92	7,94	113,77	12,66	109,12	24,52	131,25	34,82	49
Ta ppm	0,91	0,07	0,96	0,06	0,97	0,06	1,2	0,1	1,33
Co ppm	22,66	1,24	24,8	1,91	26,52	3,4	34,84	15,34	10,7
La ppm	27,92	1,05	29,8	2,75	25,96	3,09	34,91	4,52	25,4
Ce ppm	59,09	3,05	63,58	5,58	56,37	4,82	76,44	3,42	72,2
Sm ppm	5,29	0,24	5,62	0,56	4,89	0,54	6,61	0,67	5,35
Eu ppm	1,12	0,05	1,19	0,12	1,04	0,11	1,41	0,17	1,05
Tb ppm	0,67	0,07	0,73	0,08	0,67	0,08	0,95	0,12	0,79
Yb ppm	2,7	0,12	2,87	0,19	2,65	0,12	3,44	0,36	2,96
Lu ppm	0,34	0,01	0,36	0,02	0,345	0,02	0,44	0,05	0,362

Mean concentration values and standard deviation. Trace elements NAA analytical results for Archaic sample A882.

TABLE 3
Trace elements NAA analytical results for raw materials samples (in wt. % and ppm).

Elements	Raw materials (1)		Raw materials (2)		Raw materials (3)	Raw materials (4)		Raw materials (5)	
	Mean (n=12)	St.Dev.	Mean (n=2)	St.Dev.	Mean (n=1)	Mean (n=10)	St.Dev.	Mean (n=4)	St.Dev.
U ppm	1,85	0,29	2,16	0,34	2,27	2,34	0,51	1,6	0,87
As ppm	3,772	0,79	3,475	0,16	2,93	2,75	1, 79	2,12	0,23
Sb ppm	0,39	0,05	0,48	0,09	0,478	0,48	0,09	0,4	0,12
Ca %	8,97	1,52	9,08	1,89	6,54	6,57	3,08	6,57	2,47
Na %	0,97	0,19	0,79	0,48	0,89	0,83	0,23	0,83	0
K %	1,5	0,3 4	1,92	0,6 4	2,14	1,9	0,49	1,08	0,5
Th ppm	6,52	2,176	8,46	1, 38	9,53	8,94	2, 19	5,6625	3, 03
Cr ppm	158	22, 63	165,5	3, 53	177	190,8	41,954	130,5	22, 13
Hf ppm	3,65	0,861	3,65	0,95	2,88	2,82	1,767	2,58	0,82
Cs ppm	3,649	0,651	5,6	2, 37	5,8	5,05	1, 72	2,85	2, 11
Sc ppm	1,133	1,570	15,6	4,667	17,2	15,92	4, 23	9,57	5, 31
Rb ppm	72,17	11,2	104,7	41,436	111	97,02	28, 97	61,5	35, 44
Fe %	3,05	0,44	3, 78	0,7 8	4, 27	4,09	0,98	2, 57	1,2 4
Zn ppm	73,36	10,424	97	29, 70	112	97,14	24, 84	60,65	34, 04
Ta ppm	0,69	0,078	0,827	0,15	0,86	0,83	0,18	0,57	0,27
Co ppm	18,97	2, 73	22,8	6, 64	25,3	24,05	6,3	14,5	7, 44
La ppm	21,55	2, 56	24,9	3, 96	27,1	26,85	6, 24	17,17	8, 03
Ce ppm	46,49	5, 94	53,5	5, 79	58,9	57,23	13, 47	37,3	18, 16
Sm ppm	4,26	0,51	4,71	0,41	5,15	5,07	1, 03	3,38	1, 48
Eu ppm	0,89	0,1	0,99	0,09	1,07	1,11	0,23	0,73	0,31
Tb ppm	0,63	0,08	0,63	0,09	0,738	0,65	0,11	0,48	0,19
Yb ppm	2,12	0,23	2,3	0,18	2,53	2,51	0,39	1,69	0,71
Lu ppm	0,26	0,03	0,29	0,02	0,322	0,32	0,05	0,21	0,09

Mean concentration values and standard deviation.

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