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Establishing a new reference group of Keay 25.2 amphorae from Sidi Zahruni (Nabeul, Tunisia)

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Revista	Applied Clay Science. Volume 132-133, November 2016, Pages 140-154		
DOI	https://doi.org/10.1016/j.clay.2016.05.027		
Disponible en línia	6/6/2016	**Data de publicació**	11/2016

Per citar aquest document:

S. Baklouti, L. Maritan, Ll. Casas, N. Laridhi Ouazaa, R. Jàrrega, M. Prevosti, C. Mazzoli, B. Fouzaï, S. Larabi Kassaa, M. Fantar, Establishing a new reference group of Keay 25.2 amphorae from Sidi Zahruni (Nabeul, Tunisia), In Applied Clay Science, Volumes 132–133, 2016, Pages 140-154, ISSN 0169-1317, https://doi.org/10.1016/j.clay.2016.05.027.

Abstract

This paper presents the results of the archaeometric study of African Keay 25.2 amphorae from the archaeological site of Sidi Zahruni (Beni Khiar, NE Tunisia), where this pottery was massively produced. A set of 43 amphorae was analysed with a combined approach consisting of thin-section petrography, X-ray powder diffraction (XRPD) and X-ray fluorescence (XRF), to establish a homogeneous reference group for this production. Although all the amphorae are petrographically very similar, three petro-fabrics were identified in terms of grain-size distribution and abundance of inclusions. Detailed digital image analysis, carried out on SEM-BSE images of some representative samples of each petro-fabric, was used to quantify the differences among them. Cluster analysis of XRPD data patterns also revealed groups of samples for which similar raw materials/paste and firing conditions were used, contributing to better assessment of information on the production process. Statistical multivariate treatment (principal component and cluster analyses) of chemical data and comparisons with 10 samples previously attributed to the Sidi Zahruni potteries show that the potsherds analysed here are similar from the geochemical viewpoint. Similar trends in the abundance and ratio of some trace and rare earth elements (REE) also indicate that the Sidi Zahruni amphorae were produced from a local clayey material collected from nearby outcrops of Upper Miocene deposits.

Highlights

- Archaeometric study of African Keay 25.2 amphorae from Sidi Zahruni site, north-eastern Tunisia.
- Comparisons of both bulk chemical composition and trace and rare earth element (REE) patterns of both studied amphorae and those from literature data with local clayey materials allow defining the area from which the clay was supplied at Sidi Zahruni.
- New and consistent “reference group” for Keay 25.2 amphorae is established for Sidi Zahruni.

Keywords

Petro-groups ; Chemical data ; Reference group ; Amphorae ; Roman times ; North Africa

1. Introduction

In the last few decades, Roman amphorae have been archaeometrically investigated in several studies, and have provided valuable insights into socio-economic relations, revealing interactions between different regions (mainly trade routes) and the technological evolution of societies (Buxeda i Garrigós et al., 2002; Prudêncio et al., 2003, 2009; Capelli, 2005; Dias et al., 2010; Fantuzzi et al., 2015a,b and references therein). Problems regarding the provenance of amphorae, due to imitations, export of raw materials and potters' movements, all require the support of petrographic, chemical and mineralogical analysis in addition to classical archaeological methods based on epigraphic and typological studies. Moreover, the definition of reference groups, based on materials collected from the production site, is essential to infer the provenance of amphorae found in consumption sites.

During the Roman Empire, what is now Tunisia (once part of the Roman Province of Africa Proconsularis) was a major centre of production of African-type amphorae, which were extensively traded throughout the ancient world (Peacock et al., 1990). This type of pottery provides clear information attesting to the importance of the main economic connections among Roman regions, as trade routes were essential for transporting foodstuffs (Bes, 2007).

The paste of African amphorae is coarse enough to allow petrographic and mineralogical examination before chemical analyses. This approach has already been used on African materials from consumer sites (Peacock, 1984, 1994), on non-African materials (Schuring, 1984) and on amphorae from production sites in regions with a single source of raw materials (Peacock and Tomber, 1991). Most of the ceramics produced in North Africa, but also in Palestine, are distinguished from other Mediterranean productions by the occurrence of aeolian quartz grains in the paste. But, the lack of specific petrographic markers is a significant complication in determining the provenance of African ceramics. In some cases, the distinction among African pastes, representing specific productions, has been achieved by comparison of petrographic characteristics, typological classifications and reference groups of available workshops (Fantuzzi et al., 2015a,b and references therein). However, the widespread occurrence of similar siliciclastic lithologies in North African regions, i.e., the coarser fraction in clayey materials and sand clasts mostly composed of quartz and non-diagnostic accessory inclusions (feldspar, limestone, sandstone, clay pellets), hinder definite attribution of amphorae to specific production centres in Tunisia (Fantuzzi et al., 2015a,b and references therein). Therefore, provenance studies of amphorae from these regions also require chemical data from a significant number of samples, and statistical multivariate analysis (Baxter, 1994).

The aim of this paper is to establish a reference group, based on petrographic, mineralogical and chemical data, for a distinct class of amphora, African Keay 25.2, representing the prevalent type made at Sidi Zahruni, in the Nabeul region (NE Tunisia) (Fig. 1a). Despite the large diffusion of this amphora type in the numerous localities of the ancient Roman world, only preliminary archaeological and archaeometric studies have been carried out on these amphorae in the last decade (Sherriff et al., 2002a,b; Fantuzzi et al., 2015a,b and references therein) and they are still generally based only on the petrographic analysis of a small number of samples supplied from Sidi Zahruni and other sites of the Nabeul region. Only Fantuzzi et al. (2015a,b) characterised the amphorae also from a chemical viewpoint, but the amphorae they analysed were found in other Roman provinces and were attributed, on the basis of archaeological considerations, to the site of Sidi Zahruni. Proper reference groups for amphorae produced in the various localities of this region, is actually missing, and provenance attributions are at the moment based only on materials supposed to come from the production sites.

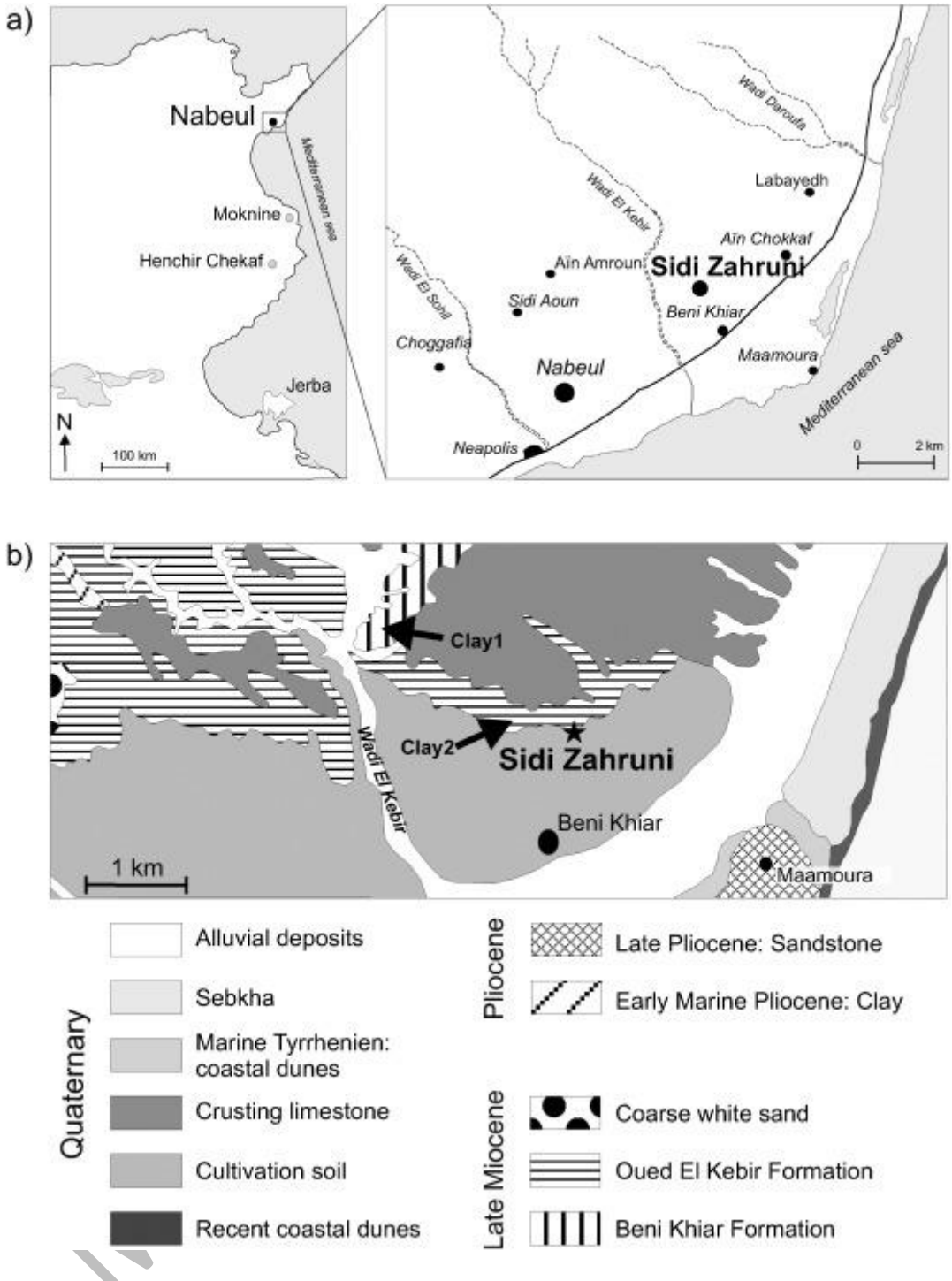

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Fig. 1. a) Location of the production centre of Sidi Zahruni in Tunisia. Other productive sites of Late Roman amphorae in north and central Tunisia are also given; b) Geological sketch of the area of Sidi Zahruni and location of clay sampling points near the site.

The archaeometric analyses here proposed will allow defining a homogeneous compositional reference group for Keay 25.2 amphorae at Sidi Zahruni. Moreover, the comparisons with clays outcropping in the area also aimed at identifying the specific source of the raw materials.

2. Archaeological context

One of the best-documented amphora production area in Africa Proconsularis is that of Nabeul (ancient Neapolis, NE Tunisia), where potteries have also been identified by the stamp C.I.N. [*C (olonia) I (ulia) N (eapolis)*] found on African amphorae of Africana II C (Keay, 1984). In the late 3rd/early 4th century, the classical Africana I and Africana II types were replaced by the cylindrical medium-sized Africana III (or Keay 25) type, which became the most common in the Roman world (Keay, 1984). The African Keay 25 amphora, as classified by Keay (1984) based on discoveries in Catalan sites (NE Spain), and corresponding to Africana amphorae III (Peacock et al., 1989), is a variety of the North African amphorae produced between the 4th and 7th centuries AD. This product, and particularly its most common variant Keay 25.2 (Fig. 2), corresponding to Africana III C (Fantuzzi et al., 2015a,b and references therein), has been found in many regions of Tunisia, mainly in Zeugitana (Nabeul) and Byzacena (Sullecthum, Thaenae, Oued El Akarit) where many potteries have been discovered (Peacock et al., 1989). Archaeological surveys of the Nabeul region have shown the existence of many Roman potteries such as Aïn Amroun, Sidi Zahruni, Sidi Aoun, Briquéteries (Fantuzzi et al., 2015a,b and references therein), Choggafia, Aïn Chokkaf, Labayedh, and many others located near villages. The most important of these is that of Sidi Zahruni (Fig. 1a), with large-scale ceramic production dominated by amphorae, and active mainly between the 5 th and 7th centuries AD. In addition to Keay 25.2 the site produced also other varieties of Late Roman Amphorae, mainly Keay types 35, 55, 56, 57 and 62 A (Fantuzzi et al., 2015a,b and references therein).

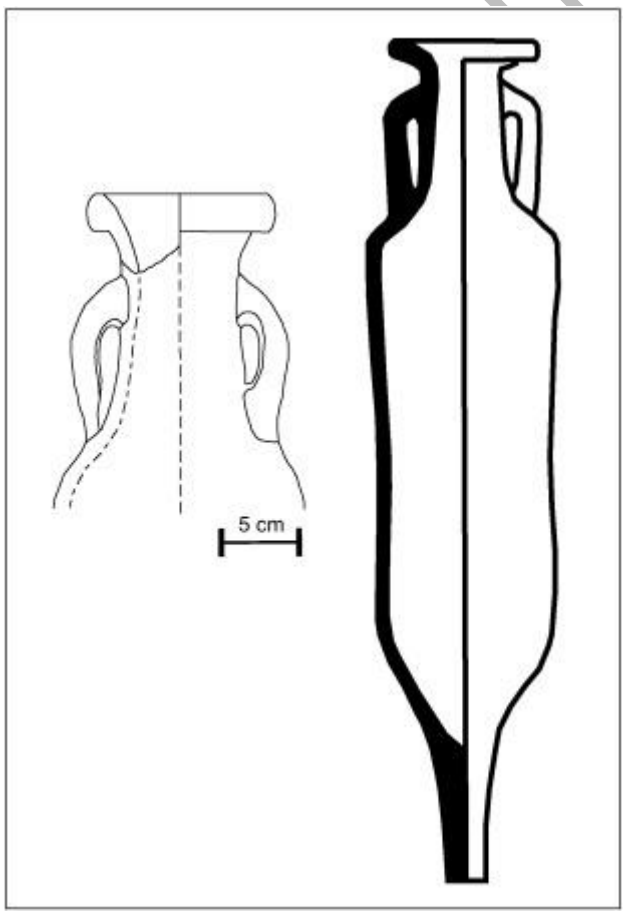

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Fig. 2. Medium-sized cylindrical-shaped Keay 25.2 amphora (Keay, 1984).

3. Geological context

The site of Sidi Zahruni is located near the modern town of Beni Khiar, 6 km north-east of Nabeul, and covers an area of 13 ha between the Wadi El Sohil and the Jebel Al Qola, and crossed by the Wadi El Kebir (Fig. 1). It is at a short distance from the sea, from which it is separated by a system of dunes (recent and Marine Tyrrhenien) and outcrops of Late Pliocene sandstone. Sidi Zahruni is at the base of the southern side of the massif of Jebel Al Qola (75 m high), an anticline consisting of a stratigraphic succession of Tertiary sedimentary rock of marine origin., formed by few meter layers of grey-blue clays, alternating with layers of quartz-sandstone and rare layers of thinner limestone and marl. More in detail, the area surrounding the site is characterised, in addition to Quatrenary deposits (soils, crusting limestone) by the presence of two geological formations: i) the Upper Tortonian-Lower Messinian Beni Khiar Formation, geologically defined near the village of Beni Khiar, close to Nabeul, and consisting of a 15m thick sequence of sandstone and clay, with some intercalations of limestone and marl; ii) the Messinian Oued El Kebir Formation, composed of sandstone, clay, sand, and small banks of limestone. The clay levels within these formations represent the possible clay source exploited during the Late Roman Times for the amphorae production.

4. Materials and methods

A set of 43 potsherds from Keay 25.2 amphorae (labelled CZ) were selected from the site and archaeometrically analysed. In particular, they were collected from the surface, since the site was not excavated up to now, and therefore it cannot be excluded that they may also represent ceramic wastes from the site production. In the area of the site various kilns have been identified. Most of potsherds have a fine-grained paste, reddish-orange to reddish-brown in colour. In some cases, the surface is grey; in others, both paste and surface (samples CZ-1-230, CZ-1-227, CZ-10-6, CZ-5-97) are grey, presumably due to a change in the firing atmosphere. The outermost portions of the amphorae are characterised by a white/cream colour, due to the effect of salt during firing, since salted sea water was used to mix the clay (Sherriff et al., 2002a,b).

Two clay samples (Clay1, Clay2) were collected from the undisturbed clay-rich layers of the nearby Upper Miocene deposits, at a depth of 30–40 cm. In detail, Clay1 comes from the Upper Tortonian-Lower Messinian Beni Khiar Formation, whereas Clay2 was collected from the late Messinian clayey deposit of the Oued El Kebir Formation (Fig. 1b). Both clays are suitable for ceramic manufacture, especially that from the Chabat Al Qola massif, although even nowadays it is exploited by a brick factory located near the archaeological site. Traces of clay exploitation in ancient times are still visible along the slopes of the massif.

All amphora fragments were thin-sectioned and petrographically analysed, and their ceramic fabrics were described following the procedure of Whitbread (1989, 1995). Samples were divided into petrographic groups and subgroups according to the type, abundance and grain-size of inclusions. One representative sample was selected for each subgroup and analysed by scanning electron microscopy (SEM) on a CamScan MX 2500 equipped with a LaB_6 cathode, coupled to an EDS spectrometer, and operating at 20 kV and 160 nA. For each sample, a set of about 120 back-scattered (SEM-BSE) images (resolution 1280 x 1024 pixel) were merged, covering almost the entire surface of the thin section (approximately 46 x 27 mm). The resulting image was processed with open source software (ImageJ 1.44p, National Institutes of Health, USA; MultiSpec 3.1, Purdue Research Foundation) to extract quantitative data of the textural features of the pottery paste, i.e., the abundance of its constituents (inclusions, voids, matrix) and the grain-size distribution of inclusions. Particle analysis of binary images gave the Minimum Feret diameter (MinFeret) of the grains, defined as the shortest distance between any two points along the selected grain boundary, corresponding to the minimum size of the inclusions. Particle grain-size were described according to the MinFeret diameter because it provides results similar to those obtained by sieving in sedimentological studies. The cut-off point between matrix inclusions and grains was set at 10 μm, according to Whitbread (1995). Image treatment followed the method of Dal Sasso et al. (2014).

The mineralogical composition of all samples was determined by X-ray powder diffraction (XRPD) on a Philips PW 3710 diffractometer in Bragg–Brentano geometry, equipped with a Cu X-ray tube, operating at 40 kV and 40 mA in the 4°–70° 2θ range, with a step-size of 0.02° and a counting time of 1 s per step. In order to obtain homogeneous groups with analogous mineralogical composition, cluster analysis of the whole dataset was performed with X'Pert HighScore Plus® software, according to the procedure of Piovesan et al. (2013) for analysis of mortars and of Maritan et al. (2015) for that of pottery.

Quantitative chemical analyses of major, minor (SiO_2, TiO_2, Al_2O_3, Fe_2O_3, MnO, MgO, CaO, Na_2O, K_2O, P_2O_5) and trace elements (Sc, V, Cr, Co, Ni, Cu, Zn, Ga, Rb, Sr, Y, Zr, Nb, Ba, La, Ce, Nd, Pb, Th, U) were performed on both potsherds and clay materials by X-ray fluorescence (XRF) on a Philips PW2400 spectrometer, in order to define the chemical variability of the potsherds and their compatibility with the petrographic subgroups and the local base-clays. Potsherds and clay specimens were reduced to powder in an agate mortar, after removal of the surface layer of potsherds to avoid any contamination, and then prepared as beads from calcined powder and $Li_2B_4O_7$, dilution ratio 1:10. A set of geological standards, as analytically tested by the international scientific community (Govindaraju, 1994), was used for calibration; they were supplied by the following agencies: USGS (United States Geological Survey, Reston, USA), CRPG (Centre de Recherches Pétrographiques et Géochimiques, France), ANRT (Association Nationale de la Recherche Technique, Paris, France), GIT-IWG (Groupe International de Travail - International Working Group, France), RIAP (Research Institute of Applied Physics, Irkutsk, Russia), GSJ (Geological Survey of Japan, Japan), MINTEK (Council for Mineral Technology, South Africa) and WIHG (Wadia Institute of Himalayan Geology, India).

Chemical data were processed with standard statistical tools such as Principal Component Analysis (PCA) and cluster analysis (CA) with Statgraphics® Centurion XVI software. The R Project for Statistical Computing was used to explore the compositional variation matrix, according to the method of Buxeda i Garrigós (1999) to evaluate any contamination. Multivariate analyses were carried out on a subset of elements, excluding those with a low ratio between total variation (vt) and variance (τ.i). Raw data were standardised according to procedures designed by Vitali and Franklin (1986), Baxter (1999) and Buxeda i Garrigós (1999), by log-normal ratio transformation in order to avoid misclassifications due to the different orders of magnitude and range of variation of the variables (Fermo et al., 2008).

5. Results and discussion

5.1. Petrography

Petrographic analysis of thin sections facilitated the identification of three fabrics according to similarities and differences, especially in the nature, shape and arrangement of aplastic inclusions, voids and micromass.

- Petro-group 1: PG1

This group of 17 samples contained about 30% of very poorly sorted aplastic inclusions, displaying almost unimodal grain-size distribution. In some cases, medium sand-sized inclusions also occurred, showing a grain-size gap with respect to the smaller fraction. The inclusions were mainly fine to medium sand-sized quartz, occasionally associated with rare crystals of pyroxene, plagioclase and mica, small fragments of chert, micritic and sparitic limestone, sandstone, and microfossils. The pores were generally small to medium in size (< 1 mm), occurring mainly as vesicles, but also as elongated subparallel channels and irregular-shaped vughs (Fig. 3a). The micromass was generally iron-rich, optically inactive.

- Petro-group 2: PG2

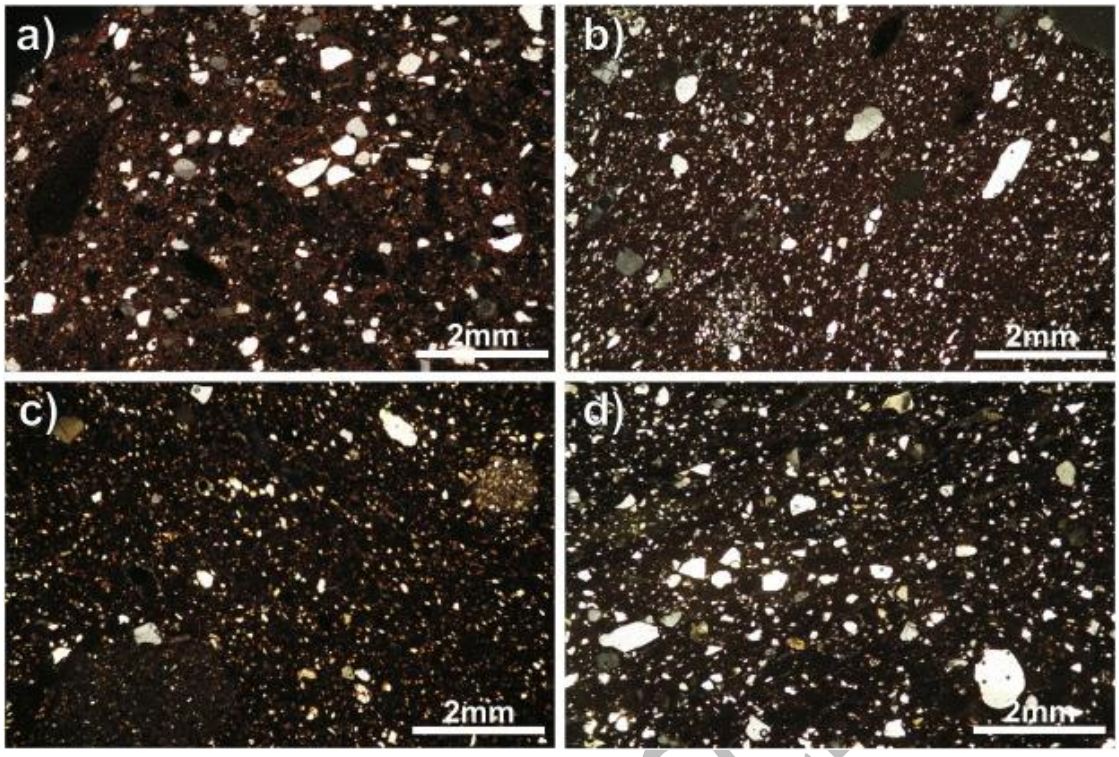

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Fig. 3. Photomicrographs of representative samples of the petrographic groups: a) PG1; b) PG2a; c) PG2b; d) PG3. Cross polars.

This group contained 22 samples, characterised by abundant very fine-grained inclusions (< 300 μm), predominantly composed of angular quartz grains, and a coarser fraction of rare rounded (aeolian) grains of quartz and quartz-sandstone, usually with calcareous cement. Coarse fragments of fine quartz-sandstone and sub-angular to rounded quartz grains were occasionally found. As regards inclusion composition, the samples of this group were similar to those of PG1, but had smaller quartz grains, with bimodal grain-size distribution. Rare fragments of microfossils and iron-rich inclusions were also found in some potsherds. Two distinct subgroups were distinguished: one with higher (PG2a) and one with lower (PG2b) percentages of silt and fine sand-sized inclusions (Fig. 3b,c).

- Petro-group 3: PG3

This was a small group of only four samples, characterised by moderately sorted mainly medium-sized inclusions, with unimodal grain-size distribution, composed mainly of sub-angular to sub-rounded quartz, with rare coarser aeolian quartz grains, up to 700 μm. Compared with PG1 and PG2, the silt and fine sand-sized fraction were lower, and the abundance of coarse sand higher. The groundmass was moderately to very porous, dark reddish-brown in colour, and optically inactive. A few crystals of plagioclase, biotite, pyroxene, opaque minerals chert and quartz-sandstone were also found (Fig. 3d).

The presence of siltstone and sandstone fragments in most of the amphorae was consistent with the clayey materials from the Upper Miocene deposits (Chabat Al Qola massif), which were also rich in sandstone grains.

As the petrographic groups were distinguished mainly according to their grain-size distribution, some representative samples of each petro-group were subjected to digital image analysis (DIA) on scanning electron microscope back-scattered (SEM-BSE) images, to quantify these differences.

The DIA results showed that all the above petro-groups had similar c:f:v (coarse:fine:voids) ratios, as clearly revealed by the histograms of their percentages (Fig. 4a), although their grain-size distribution of inclusions differed (Fig. 4b,c). In more detail, groups PG1 and PG2a appeared to be very similar, although the former was richer in medium sand-sized inclusions. Petro-group PG2b had the lowest percentages of medium and coarse inclusions, whereas petro-group PG3 has the highest ones.

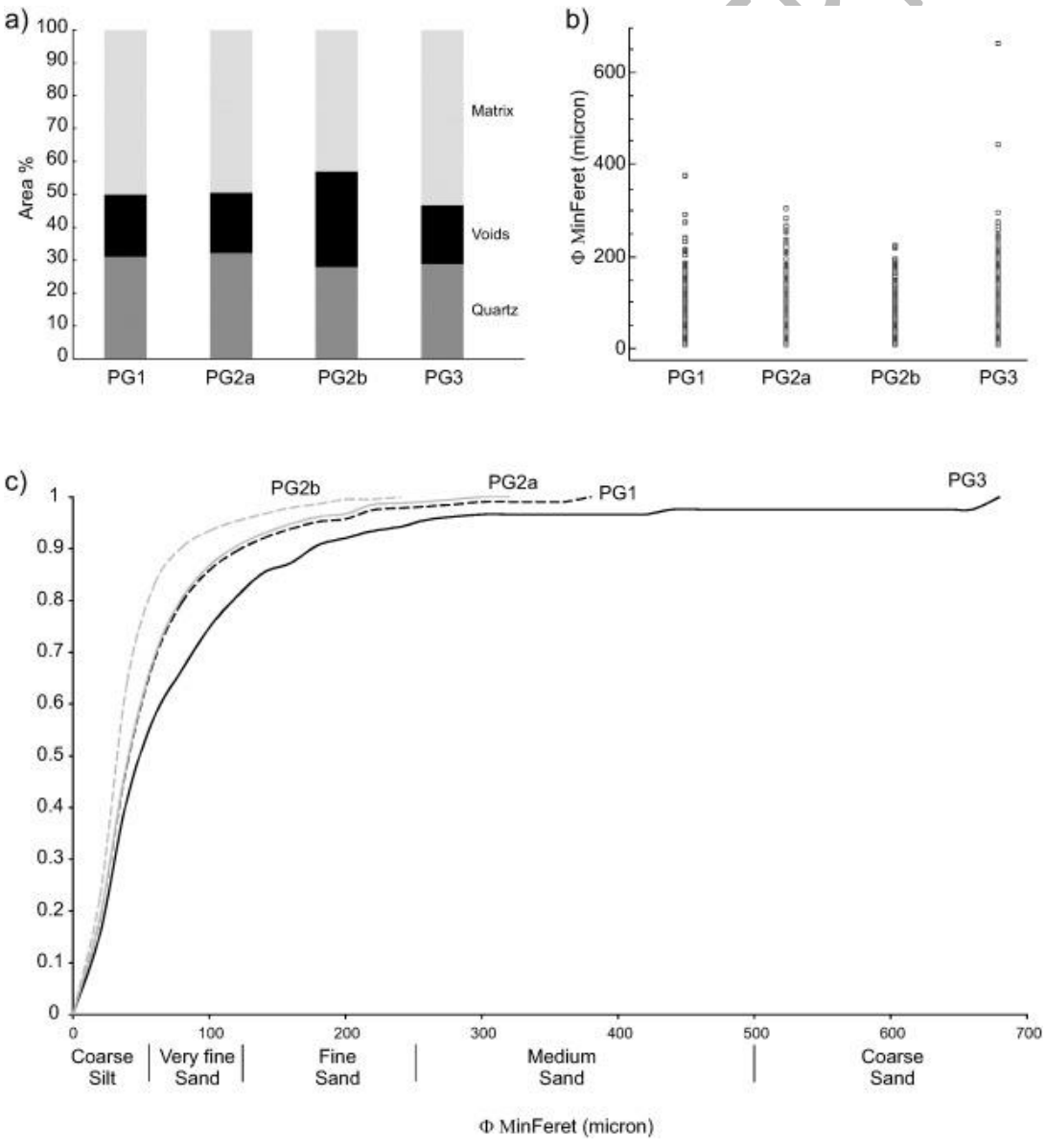

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Fig. 4. a) Histogram of quartz, voids and matrix percentage; b) dispersion diagram of quartz inclusions; c) cumulative grain-size curves of inclusions, as obtained from DIA, for samples representative of the various petrographic groups.

5.2. Mineralogical composition and firing temperature

As regards the mineralogical composition, the cluster analysis of XRPD patterns (Fig. 5) showed that the samples fell into five clusters, with the exception of three sherds (outliers: CZ-4-65, CZ-4-67 and CZ-12-173). The diffraction patterns of the most representative sample of each cluster showed the same mineralogical association of quartz, orthoclase, calcite, anorthite, hematite and diopside; therefore, the clusters differed in their relative abundances of mineral phases. The resulting dendrogram showed that the sherds of clusters 1 and 2 were very similar, due to the presence of calcite and the low anorthite content (Fig. 6a,b), whereas those of clusters 3, 4 and 5 (Fig. 6d,e,f) shared a higher proportion of anorthite. The three isolated samples were composed of quartz, orthoclase, illite and calcite (Fig. 6c), in addition to a relatively small amount of hematite in sample CZ-12–173. The mineralogical similarities between clusters indicated that quartz-rich and moderate-poor carbonate clayey materials were used to produce these amphorae. The small differences in the relative proportions of the mineral phases may have been due to firing conditions (maximum firing temperature and atmosphere) and perhaps on the addition of a temper.

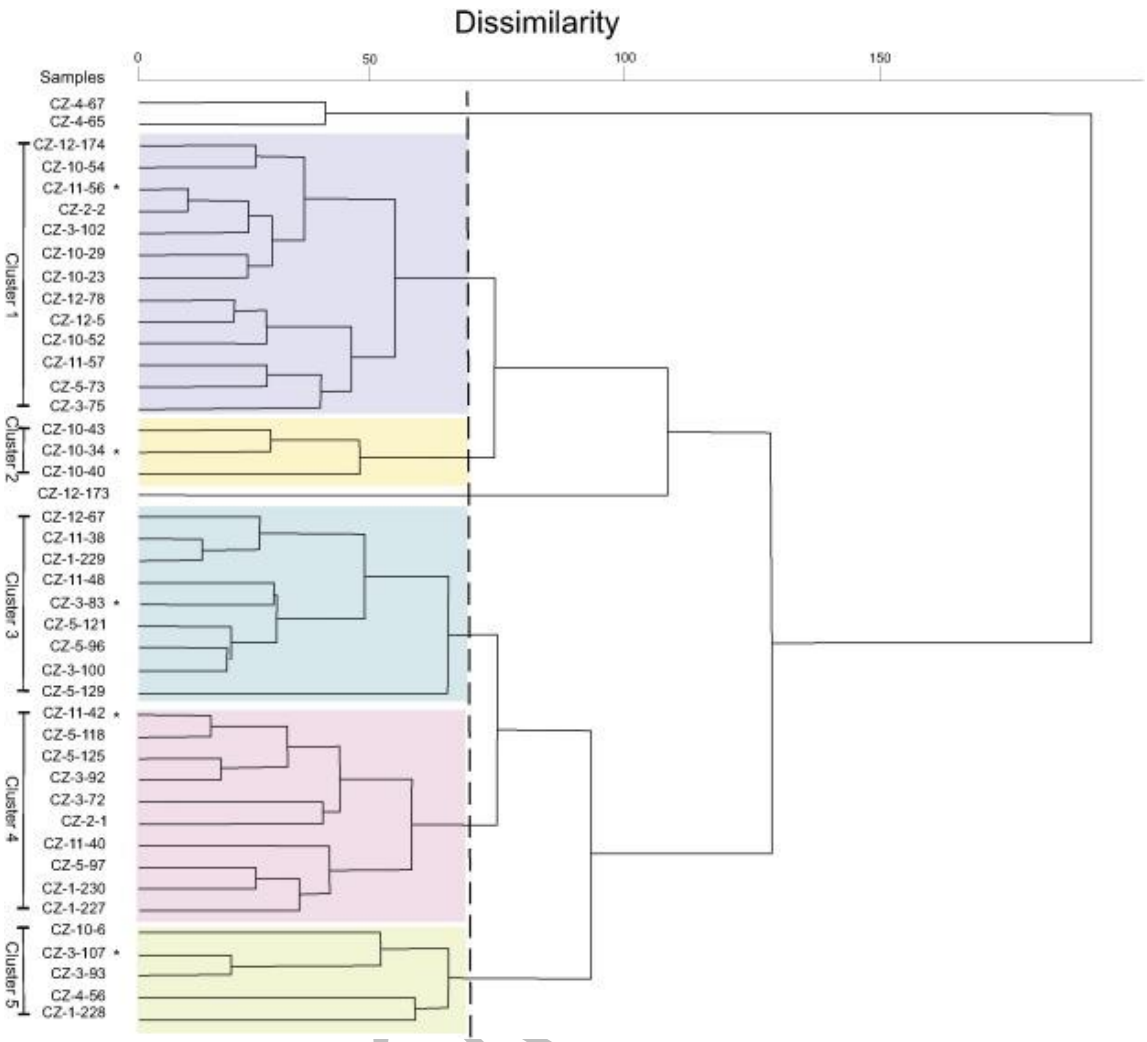

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Fig. 5. Dendrogram from cluster analysis of XRPD patterns of Keay 25.2 amphorae from Sidi Zahruni site, according to Euclidean distance and average linkage method on position and intensity of peaks. *: most representative samples within each cluster.

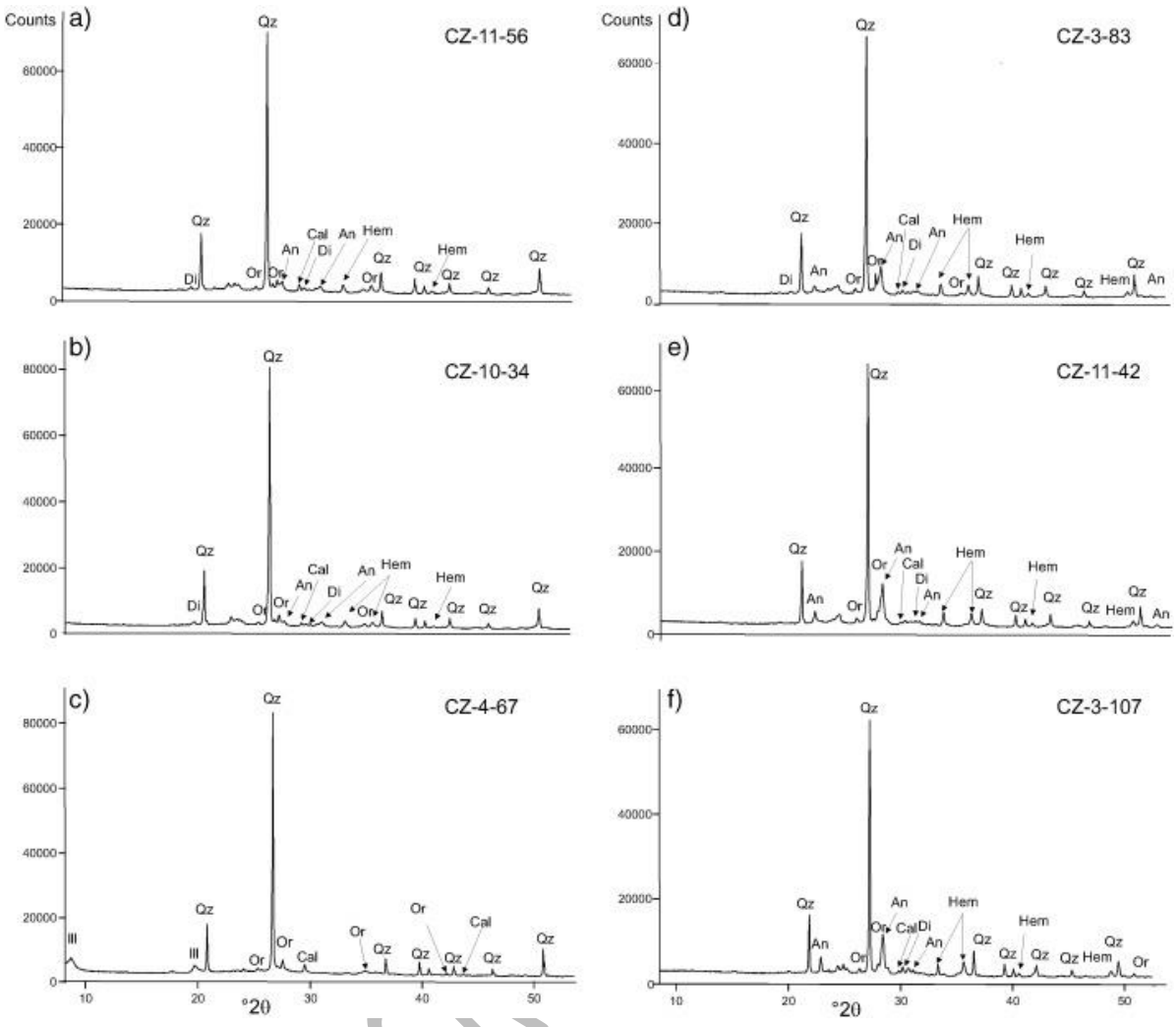

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Fig. 6. XRPD patterns of most representative sample (sample name: top right of each pattern) for each cluster identified in the dendrogram of Fig. 6. Abbreviations: Qz: quartz, Ill: illite, Mi: microcline, Or: orthoclase Cal: calcite, Hem: hematite, Di: diopside, An: anorthite (Whitney and Evans, 2010).

XRPD analysis provided the key to reconstruction of firing temperatures according to mineralogical changes during firing (Maritan et al., 2005, 2006; Nodari et al., 2007; Grifa et al., 2009). Regarding outliers, in which some calcite and illite were detected and newly formed minerals were not identified, the firing temperature was 800–850 °C. According to their optically active groundmass in thin section (Fig. 3a), these samples were relatively low-fired products. In the potsherds of clusters 1 and 2, anorthite and diopside became detectable in the diffraction patterns and calcite was still present in trace amounts, indicating relatively higher firing temperatures, around 850–900 °C. In these samples, also amorphous phase can be recognised, as the background is quite high, especially in the region between 20° and 30° 2θ. The majority of samples (clusters 3, 4, 5) had higher quantities of anorthite and diopside, suggesting firing higher than 900 °C but lower than 1000 °C, since mullite, which would be expected to form after the decomposition of phyllosilicates (Gualtieri et al., 1995; Cultrone et al., 2004), lacks.

XRPD analysis of the two clay samples (Clay 1 and Clay 2), collected near Sidi Zahruni, showed remarkable similarities in their mineralogical compositions, since they were both composed mainly of quartz, calcite and K-feldspar, in addition to clay minerals, illite and kaolinite. Clay1 had a notably higher content of calcite.

5.3. Chemical composition and raw material provenance

The chemical composition of the amphorae was quite homogeneous, although small differences were observed for some elements (Table 1). More in detail, the amphorae are calcareous, although not highly, have a relatively high content of iron, which determine their reddish-brown colour, and of silica due also the nature of their inclusions (mainly quartz), whereas alumina is relatively low (Table 1). The statistical treatment of the chemical data, according to cluster analysis (performed on 26 elements and excluding Sc, U, Sr and CaO due to their high τ values: 19.539, 15.222, 4.787 and 4.618, respectively), shows that the amphorae tended to cluster into two main groups, with the exception of a few samples (CZ-3-93, CZ-4-67, CZ-2-2, CZ-10-6 and CZ-11-40) and the two clays (Clay1 and Clay2), which show a high dissimilarity level (Fig. 7). Most of the potsherds of PG1 tended to group in cluster CL2, separately from those of PG2 and PG3, which clustered in CL1. This was also confirmed by the PCA score plot (Fig. 8), in which almost all the PG1 potsherds had negative values of PC1 and were richer in MgO and Ni, whereas those of PG2 and PG3 mainly had positive values of PC1 and were richer in SiO_2, TiO_2, K_2O, Fe_2O_3 and trace elements such as Ce, Nb, Ba, Zn, Cu, Rb, Zr, Y and Th. As regards clay materials, the two samples were isolated from the potsherds, but Clay2 was closer to the amphorae cloud, whereas Clay1 was chemically different from all the samples because of its high CaO concentration (Table 1). Although many archaeometric studies based on petrographic analysis have been carried out in recent years on Late Roman African amphorae from various workshops in Tunisia and found in many consumption sites in the Mediterranean area (Sherriff et al., 2002a,b; Capelli, 2005; Capelli and Leitch, 2011; Fantuzzi et al., 2015a,b and references therein), only very few African amphorae have been chemically analysed (Sherriff et al., 2002a,b; Fantuzzi et al., 2015a,b). Statistical analysis of these data with those produced in the present research shows a clear distinction between the amphorae produced at Sidi Zahruni and those found in Catalonia attributed to various workshops in the Nabeul area (Sidi Zahruni, Labayedh, Choggafia) and central Tunisia (Moknine and Henchir Chekaf) (Fig. 9). In more detail, the amphorae found in Tarragona (Catalonia, Spain) (*Sidi Zahruni* in Fig. 9), previously defined by Fantuzzi et al. (2015a) as produced at Sidi Zahruni on the basis of typology (Keay 35 A, Keay 35B, Keay 27, Spatheion 1) and petrographic features, do not cluster with those of Sidi Zahruni due to their higher contents of Ce and Nb (Fig. 10). Despite they are compositionally more similar to those of the central Tunisian workshop of Henchir Chekaf, it cannot be excluded that the geochemical differences with the samples collected at Sidi Zahruni are due to the different instrument used to perform the analysis or to the fact that those analysed by Fantuzzi et al. (2015a) come from a consumption site. In addition, the amphorae of Labayedh and Moknine are geochemically different from those of Sidi Zahruni (Nabeul) and Henchir Chekaf (central Tunisia), since they have higher contents of Nb, Ce and Cr (Fig. 10), whereas the amphorae of Choggafia are geochemically different from all the others due to their higher Zr content. Lastly, three amphorae

from Sidi Zahruni (CZ-4-67, CZ-11-40, CZ-2-2) exhibit a higher dissimilarity level (Fig. 9) with respect to the other amphorae analysed, due to their lower Na_2O and Pb contents in the former two and higher Ni concentration in the latter (Fig. 10).

Table 1. Geochemical composition by XRF of analysed samples (ceramic and clays) with the literature data for amphorae attributed to *Sidi Zahruni* (in italic) (Fantuzzi et al., 2015a) and other sites of the Nabeul area (Fantuzzi et al., 2015b). Major and minor elements expressed as wt% of oxides, trace elements as ppm. Petrographic group for samples of Sidi Zahruni is also reported.

Samples	**Petro-group**	**SiO_2**	**TiO_2**	**Al_2O_3**	**Fe_2O_3**	**MnO**	**MgO**	**CaO**	**Na_2O**	**K_2O**	**P_2O_5**	**Tot**	**L.O.I**	**Sc**	**V**	**Cr**	**Co**	**Ni**	**Cu**	**Zn**	**Ga**	**Rb**	**Sr**	**Y**	**Zr**	**Nb**	**Ba**	**La**	**Ce**	**Nd**	**Pb**	**Th**	**U**
Potsherds. *n* = 43																																
CZ-1-227	PG2	69.02	0.87	14.42	5.755	0.03	1.93	4.15	0.62	2.98	0.15	99.92	1.46	13	115	102	17	29	29	103	23	98	217	30	368	18	4344	41	78	34	24	9	3
CZ-1-228	PG1	66.35	0.87	15.39	6.34	0.03	2.65	4.79	0.82	2.60	0.14	99.98	1.00	12	132	113	18	32	19	104	26	94	254	29	257	19	3211	49	69	39	23	9	7
CZ-1-230	PG1	65.41	0.88	15.77	6.36	0.03	2.79	5.04	0.80	2.82	0.14	100.04	0.87	9	121	116	16	32	28	111	28	98	271	29	255	20	3300	51	80	46	24	12	4
CZ-1-229	PG2	66.77	0.87	14.25	5.71	0.03	2.23	6.39	0.76	2.83	0.14	99.98	1.73	2	110	100	17	28	29	103	24	92	298	30	364	17	4433	47	67	36	24	11	5
CZ-2-1	PG2	65.05	0.89	15.05	6.29	0.03	2.25	7.10	0.73	2.66	0.13	100.18	0.92	2	117	110	19	35	27	104	25	98	263	31	343	20	3334	34	81	36	22	13	1
CZ-2-2	PG1	67.52	0.81	14.31	6.26	0.04	2.54	5.11	0.54	2.81	0.14	100.08	1.81	11	119	104	16	68	27	104	24	96	368	28	277	16	3222	34	67	36	36	7	6

Samples	Petro-group	SiO_2	TiO_2	Al_2O_3	Fe_2O_3	MnO	MgO	CaO	Na_2O	K_2O	P_2O_5	Tot	L.O.I	Sc	V	Cr	Co	Ni	Cu	Zn	Ga	Rb	Sr	Y	Zr	Nb	Ba	La	Ce	Nd	Pb	Th	U
CZ-3-72	PG1	64.38	0.88	15.84	6.58	0.06	2.41	6.06	0.49	2.58	0.17	99.45	1.23	2	133	122	19	40	26	109	28	95	435	27	238	18	355	48	77	42	22	11	7
CZ-3-75	PG1	64.61	0.89	15.59	6.23	0.04	3.20	5.80	0.63	2.73	0.13	99.85	1.42	5	132	110	15	33	30	107	26	104	634	31	306	19	336	31	67	46	29	7	4
CZ-3-83	PG1	66.56	0.87	15.15	6.46	0.03	2.23	5.37	0.62	2.68	0.13	100.10	1.14	5	123	108	17	33	28	104	24	98	250	30	301	19	354	42	67	35	26	11	1
CZ-3-92	PG2	65.72	0.91	15.61	6.48	0.03	2.12	4.70	0.62	2.89	0.16	99.24	1.16	7	125	111	18	31	27	111	26	97	245	31	308	18	400	46	74	45	24	11	5
CZ-3-93	PG2	70.39	0.81	12.41	4.91	0.03	1.66	5.61	0.66	2.51	0.11	99.10	1.28	2	103	90	12	24	22	88	22	79	204	29	408	17	363	20	65	38	26	10	1
CZ-3-100	PG2	67.83	0.89	14.55	5.78	0.03	2.00	4.95	0.52	2.72	0.15	99.42	1.56	8	115	106	17	29	30	104	25	95	229	32	356	19	423	35	67	39	28	12	4
CZ-3-102	PG2	66.61	0.90	14.84	5.95	0.03	2.04	6.00	0.56	2.78	0.15	99.86	1.95	2	119	105	17	31	31	104	24	102	260	33	365	19	403	54	71	40	24	12	1
CZ-3-107	PG3	68.31	0.82	12.97	5.60	0.04	1.96	6.57	0.62	2.40	0.14	99.43	1.02	2	110	98	16	26	23	92	21	84	657	27	384	16	342	29	66	42	23	7	7
CZ-4-56	PG2	70.03	0.85	13.95	5.56	0.03	2.00	3.67	0.75	2.71	0.14	99.69	1.27	8	118	98	18	27	22	100	24	94	320	31	345	18	344	38	65	37	24	10	6

Samples	Petro-group	SiO_2	TiO_2	Al_2O_3	Fe_2O_3	MnO	MgO	CaO	Na_2O	K_2O	P_2O_5	Tot	L.O.I	Sc	V	Cr	Co	Ni	Cu	Zn	Ga	Rb	Sr	Y	Zr	Nb	Ba	La	Ce	Nd	Pb	Th	U
CZ-4-65	PG1	70.22	0.87	15.64	5.90	0.03	2.27	1.32	0.57	3.03	0.14	99.99	3.33	20	126	104	16	32	34	108	26	103	280	33	293	18	449	41	77	37	22	13	6
CZ-4-67	PG1	65.62	0.88	15.81	6.51	0.04	2.48	5.18	0.37	2.87	0.21	99.97	6.05	8	128	108	17	38	38	111	27	84	437	30	265	18	514	49	69	28	27	9	4
CZ-5-73	PG2	68.23	0.81	13.37	5.29	0.03	2.33	6.50	0.76	2.60	0.13	100.05	1.56	2	103	92	15	26	32	103	23	92	312	29	354	16	367	41	68	32	23	11	4
CZ-5-96	PG1	66.21	0.84	14.96	6.57	0.04	2.85	4.25	0.61	2.96	0.14	99.43	1.41	6	119	107	14	33	29	102	26	99	650	27	265	16	330	43	75	40	21	8	8
CZ-5-97	PG2	66.70	0.91	15.47	6.16	0.03	2.07	4.98	0.61	2.92	0.15	100.00	0.74	5	131	110	15	33	27	107	27	100	234	31	348	19	365	49	74	39	25	11	3
CZ-5-121	PG3	65.85	0.85	15.09	6.69	0.04	2.89	4.34	0.61	3.01	0.15	99.52	1.70	8	123	111	15	36	28	107	26	96	599	28	245	16	339	33	68	50	28	7	5
CZ-5-125	PG1	65.78	0.90	15.82	6.40	0.03	2.38	4.55	0.69	2.69	0.11	99.35	1.36	11	128	115	18	32	20	103	27	95	308	30	285	18	339	35	80	43	18	15	8
CZ-5-129	PG2	66.36	0.96	16.74	6.64	0.03	2.23	3.38	0.53	2.97	0.17	100.01	1.20	10	140	119	21	34	29	119	27	113	211	33	311	21	334	51	82	52	28	12	3
CZ-10	PG2	64.6	0.8	14.4	7.2	0.0	2.0	7.0	0.6	2.6	0.1	99.9	0.8	2	12	10	19	31	29	11	23	89	91	31	35	18	37	37	64	33	25	3	3

Samples	Petro-group	SiO_2	TiO_2	Al_2O_3	Fe_2O_3	MnO	MgO	CaO	Na_2O	K_2O	P_2O_5	Tot	L.O.I	Sc	V	Cr	Co	Ni	Cu	Zn	Ga	Rb	Sr	Y	Zr	Nb	Ba	La	Ce	Nd	Pb	Th	U
-6		7	5	9	5	5	9	7	8	5	4	4	2		3	1				8			5		4		9						
CZ-10-23	PG2	67.85	0.90	14.78	5.96	0.03	2.07	4.82	0.51	2.78	0.15	99.85	2.01	5	117	98	18	31	27	103	26	100	253	32	378	19	530	33	84	31	22	12	1
CZ-10-29	PG1	69.11	0.82	14.00	5.60	0.03	1.94	5.23	0.44	2.47	0.15	99.79	1.62	7	115	97	14	29	29	100	24	94	246	29	293	17	369	30	66	41	27	12	4
CZ-10-34	PG2	66.84	0.92	15.43	6.14	0.03	2.09	4.61	0.59	2.98	0.16	99.79	1.38	9	131	106	16	32	31	106	25	106	247	33	365	19	430	41	73	40	23	12	6
CZ-10-40	PG2	67.57	0.89	14.29	5.92	0.03	2.04	5.68	0.51	2.70	0.14	99.77	2.93	2	110	100	17	29	33	102	24	99	257	32	407	18	510	33	72	34	24	10	1
CZ-10-43	PG2	69.58	0.82	12.53	5.52	0.03	1.80	6.10	0.56	2.41	0.14	99.49	1.95	2	102	85	16	26	27	93	22	86	242	30	356	16	439	23	72	21	22	12	9
CZ-10-52	PG2	67.53	0.79	12.89	5.41	0.05	2.46	7.74	0.55	2.54	0.14	100.10	3.49	2	99	91	16	26	31	93	22	88	376	28	319	17	502	30	71	31	23	8	1
CZ-10-54	PG2	65.99	0.93	15.53	6.58	0.03	2.16	4.64	0.48	2.77	0.15	99.26	1.58	12	132	112	19	33	32	107	26	100	217	31	347	18	387	41	69	41	33	9	7
CZ-	PG	67.	0.	13.	5.7	0.0	2.3	6.3	0.6	2.	0.1	10	1.8	2	10	10	1	3	2	10	2	8	37	2	30	1	36	4	7	3	2	9	7

Samples	Petro-group	SiO_2	TiO_2	Al_2O_3	Fe_2O_3	MnO	MgO	CaO	Na_2O	K_2O	P_2O_5	Tot	L.O.I	Sc	V	Cr	Co	Ni	Cu	Zn	Ga	Rb	Sr	Y	Zr	Nb	Ba	La	Ce	Nd	Pb	Th	U
11-38	3	89	80	67	4	3	9	2	4	58	3	0.19	1		7	0	5	0	8	1	6	8	8	7	0	6	6	1	6	5	3		
CZ-11-40	PG2	65.24	0.83	14.21	5.90	0.04	2.97	6.65	0.61	2.77	0.15	99.37	1	2	121	104	16	32	31	82	24	82	333	26	282	16	361	38	65	42	8	14	7
CZ-11-42	PG1	64.62	0.88	15.65	6.49	0.04	2.71	5.56	0.64	2.69	0.16	99.44	0.99	10	127	110	15	33	30	110	27	92	301	28	255	19	319	38	70	40	23	11	4
CZ-11-48	PG3	67.19	0.89	15.42	5.87	0.03	2.39	5.06	0.49	2.73	0.15	100.22	1.63	6	129	108	17	31	29	108	28	101	495	30	339	20	352	36	71	44	24	10	1
CZ-11-56	PG1	67.18	0.86	14.78	5.83	0.03	2.50	5.36	0.53	2.80	0.14	100.01	1.83	11	118	105	18	31	30	107	24	100	396	32	355	18	361	56	75	40	23	11	4
CZ-11-57	PG1	64.97	0.85	14.94	6.19	0.05	2.60	7.07	0.65	2.72	0.17	100.21	1.53	2	117	108	18	33	26	110	26	93	345	31	284	18	330	41	60	36	26	9	6
CZ-12-5	PG2	68.04	0.78	12.83	5.23	0.03	2.31	7.47	0.62	2.53	0.11	99.95	1.74	2	106	95	14	26	27	88	22	91	309	29	381	16	361	43	63	38	22	9	8
CZ-12-67	PG2	68.47	0.85	13.66	5.47	0.03	2.11	5.81	0.71	2.77	0.13	100.01	1.61	2	105	99	16	28	26	106	22	90	400	32	438	18	365	44	65	36	25	8	1

Samples	Petro-group	SiO_2	TiO_2	Al_2O_3	Fe_2O_3	MnO	MgO	CaO	Na_2O	K_2O	P_2O_5	Tot	L.O.I	Sc	V	Cr	Co	Ni	Cu	Zn	Ga	Rb	Sr	Y	Zr	Nb	Ba	La	Ce	Nd	Pb	Th	U
CZ-12-78	PG1	67.5	0.81	13.58	5.60	0.04	2.09	7.30	0.56	2.55	0.14	100.17	1.87	2	110	95	16	27	29	97	23	91	364	28	339	18	353	34	77	34	22	11	3
CZ-12–173	PG1	65.19	0.88	15.68	6.45	0.05	2.47	5.111	0.55	2.82	0.17	99.37	2.1	11	125	118	18	36	32	109	26	99	291	30	265	18	325	39	73	52	30	11	3
CZ-12–174	PG2	67.51	0.89	14.58	5.80	0.03	2.111	5.78	0.52	2.70	0.14	100.06	2.11	2	120	99	15	30	33	104	25	100	268	32	382	17	377	31	76	37	31	13	7
CZ-5-118	PG1	64.94	0.90	15.86	6.20	0.03	2.7	5.58	0.89	2.68	0.14	99.92	0.99	10	126	113	16	33	27	112	27	97	305	29	275	21	302	41	71	43	25	9	3
mean*		66.91	0.86	14.69	6.03	0.03	2.311	5.445	0.600	2.73	0.14	99.80	1.67	6.11	119.3	104.7	16.55	31.83	28.41	103.8	24.90	95.16	345.9	29.97	324.5	18	378.1	39	71.3	38.6	24.4	10.2	4.39
SD*		1.61	0.04	1.03	0.47	0.00	0.33	1.20	0.10	0.15	0.01	0.31	0.90	4.34	9.85	8.22	1.70	6.58	3.61	7.43	1.87	6.66	148.2	1.87	50.12	1.37	57.13	7.90	5.67	5.99	4.21	2.24	2.39
Sidi Zahruni n = 10																																	
mean*	*Sidi Zahruni*	68.08	0.87	14.90	5.80	0.05	1.97	4.74	0.50	2.70	0.20	99.86	1.14	–	1115	1059	–	29.40	14.70	80.80	18.40	91.30	233.1	28.20	329.8	21	348	–	81.6	–	21.2	8.20	–
SD*	*Sidi Zahru*	1.17	0.0	0.44	0.16	0.09	0.08	0.96	0.05	0.0	0.03	0.10	0.30	–	4.99	4.50	–	0.84	2.21	4.87	0.96	3.33	43.4	1.03	17.2	0.7	34.3	–	4.3	–	1.2	0.7	–

Samples	Petro-group	SiO_2	TiO_2	Al_2O_3	Fe_2O_3	MnO	MgO	CaO	Na_2O	K_2O	P_2O_5	Tot	L.O.I	Sc	V	Cr	Co	Ni	Cu	Zn	Ga	Rb	Sr	Y	Zr	Nb	Ba	La	Ce	Nd	Pb	Th	U
	ni		1							8													5		1	8	0		2		2	8	
Nabeul area n = 7																																	
CAT221CHOG	*Choggafia*	60.79	0.65	11.07	4.25	0.02	1.18	19.01	0.43	2.07	0.41	–	–	–	105	111	–	24	15	61	14	72	487	20	271	17	329	–	59	–	17	5	–
CAT232LABA	*Laba yedh*	69.89	0.74	14.13	5.71	0.03	1.95	4.45	0.48	2.35	0.14	–	–	–	105	95	–	26	10	67	16	75	192	20	178	18	228	–	65	–	18	6	–
CAT204MOK	*Moknine*	63.03	0.62	10.92	4.01	0.03	1.84	16.69	0.32	2.28	0.14	–	–	–	104	88	–	21	11	53	14	70	251	21	222	17	270	–	60	–	16	6	–
CAT220MOK	*Moknine*	61.38	0.66	12.13	4.46	0.03	2.14	16.42	0.25	2.33	0.24	–	–	–	112	95	–	25	14	68	16	74	241	24	235	18	332	–	69	–	18	7	–
CAT214ECH	*Henchir Chekaf*	63.46	0.82	15.01	5.68	0.04	2.65	8.84	0.49	2.55	0.30	–	–	–	122	113	–	31	17	87	19	89	318	28	274	23	373	–	82	–	20	8	–
CAT224ECH	*Henchir Chekaf*	61.96	0.84	15.12	5.76	0.04	2.84	10.08	0.62	2.39	0.21	–	–	–	121	120	–	31	15	77	18	82	279	27	261	21	296	–	83	–	19	7	–
mean CAT	*Henchir Chekaf*	63.63	0.81	14.56	5.66	0.04	2.56	9.34	0.53	2.46	0.28	–	–	–	113	110	–	31	13	77	17	78	257	25	250	21	279	–	74	–	17	8	–

Samples	Petro-group	SiO_2	TiO_2	Al_2O_3	Fe_2O_3	MnO	MgO	CaO	Na_2O	K_2O	P_2O_5	Tot	L.O.I	Sc	V	Cr	Co	Ni	Cu	Zn	Ga	Rb	Sr	Y	Zr	Nb	Ba	La	Ce	Nd	Pb	Th	U
ECH																																
Clays																																
Clay 1	Clay1	58.73	0.59	9.82	3.64	0.02	1.3	23.56	0.34	1.60	0.09	99.69	20.51	2	101	79	6	24	26	73	18	68	717	19	266	11	230	43	38	37	19	9	4
Clay 2	Clay2	71.46	0.79	11.81	5.43	0.02	1.43	5.80	0.41	2.44	0.09	99.68	11.12	2	106	88	11	24	27	94	20	91	178	26	443	16	475	41	56	27	21	13	3

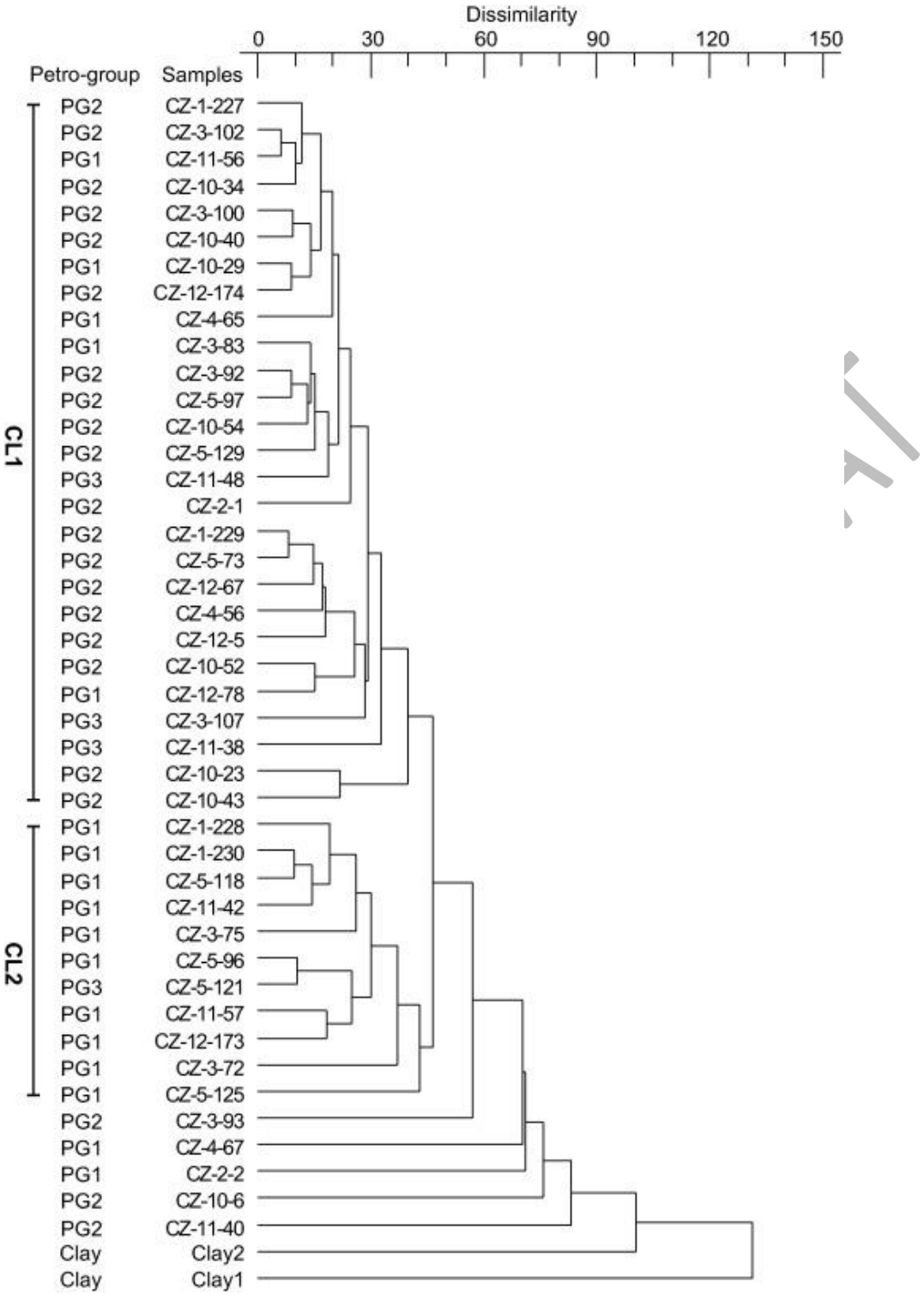

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Fig. 7. Dendrogram from hierarchic cluster analysis (average linkage method, square Euclidian distance) performed on sub-composition SiO_2, TiO_2, Fe_2O_3, MnO, MgO, Na_2O, K_2O, P_2O_5, V, Cr, Co, Ni, Cu, Zn, Ga, Rb, Y, Zr,Nb, Ba, La, Ce, Nd, Pb and Th, divided by Al_2O_3, for studied potsherds and clay materials. Abbreviations: CZ: amphorae from Sidi Zahruni site, AZ: clay materials collected near the site.

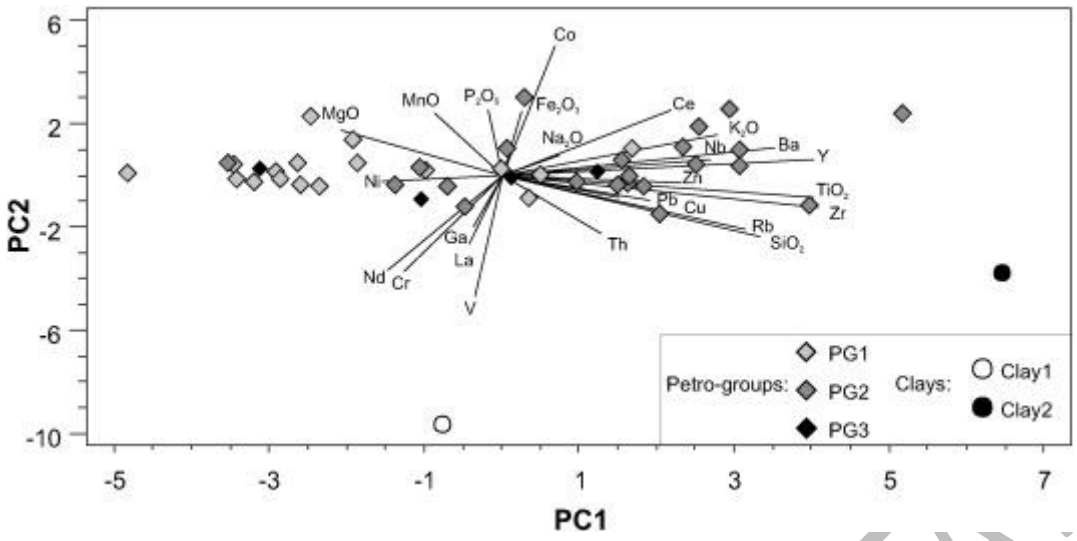

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Fig. 8. Scores and loading plots of principal components 1 and 2 (PC1 vs. PC2) explaining 26% and 14% of total variance, respectively.

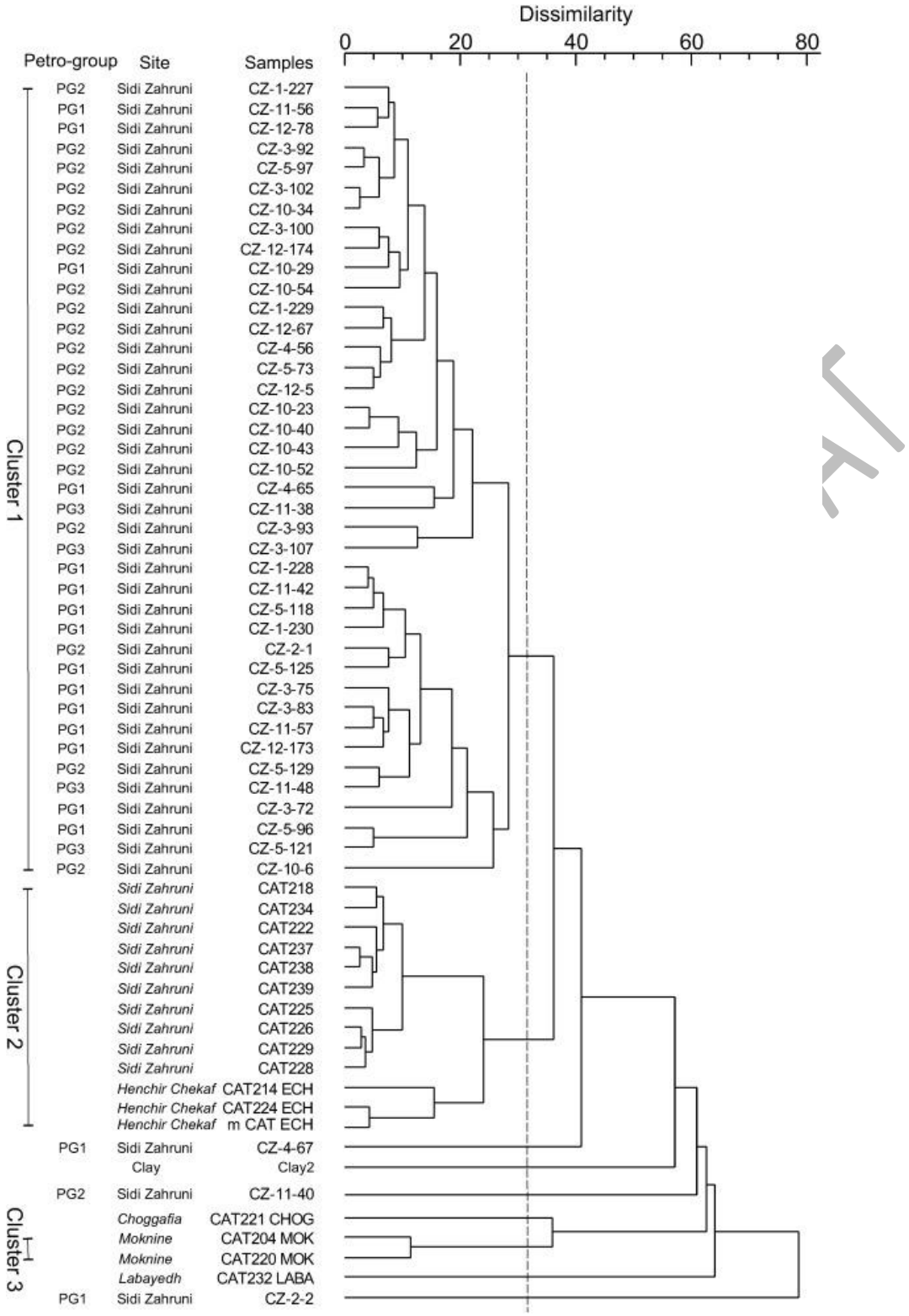

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Fig. 9. Dendrogram from hierarchic cluster analysis (average linkage method, square Euclidian distance) on sub-composition SiO_2, Al_2O_3, Fe_2O_3, MgO, CaO, Na_2O, K_2O, V, Cr, Ni, Zn, Ga, Rb, Y, Zr, Nb, Ba, Ce and P divided by TiO_2. CaO, Sr, MnO, Cu, P_2O_5, and Th were excluded from treatment due to their high τi:6.26, 4.03, 3.96, 3.20, 3, and 2.62, respectively. Amphorae from Sidi Zahruni analysed in this research and from other localities in north-eastern and central Tunisia are included. Samples

attributed to Sidi Zahruni, Henchir Chekaf, Moknine, Choggafia and Labayedh (Fantuzzi et al., 2015a) have the site name in Italic.

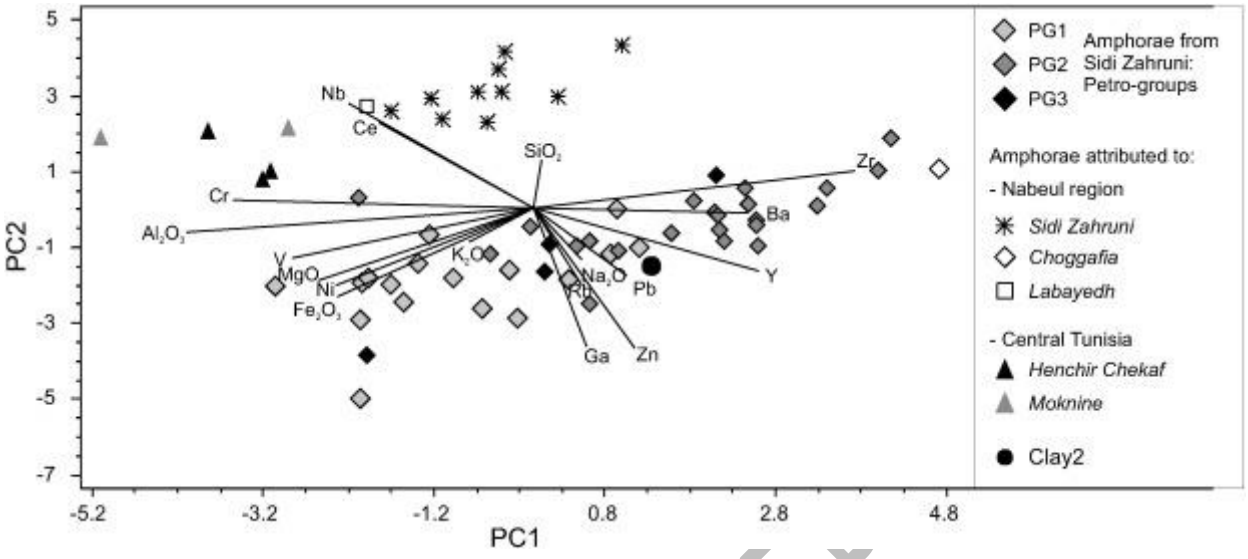

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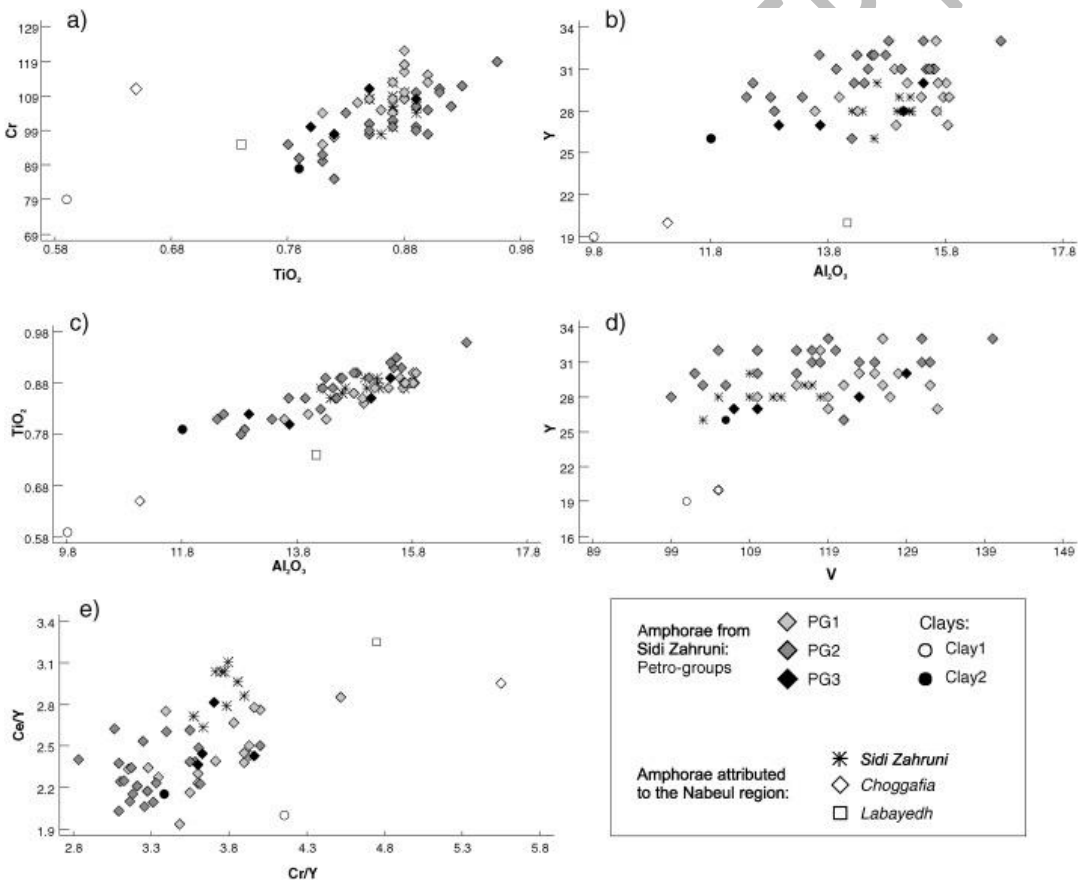
Fig. 10. Scores and loading plots of principal components 1 and 2 (PC1 vs. PC2), representing 26% and 23% of total variance respectively, relative to PCA on amphorae from Sidi Zahruni and from other localities in north-eastern and central Tunisia. Sub-composition as in Fig. 9.

Separating the amphorae into two groups may reflect technological changes, since, the three production centres in the Nabeul region were active at different chronological phases: between the 2nd century and the first half of the 5th, amphorae were produced at Choggafia, and from the 5th to the 7th centuries at Sidi Zahruni and Labayedh (Fantuzzi et al., 2015a,b and references therein). This hypothesis does not exclude the possibility that the potters used various clay materials. According to Capelli and Bonifay (Fantuzzi et al., 2015a,b and references therein), the amphorae from Zahruni are more similar to those of Choggafia than those of Labayedh.

On the basis of these considerations, it cannot be excluded the possibility that differences between the amphorae found at Sidi Zahruni and those attributed to the site (Fantuzzi et al., 2015a) are due to the use of different clay materials or production processes. Since the inclusions are mainly composed of quartz and other major elements concentrations may have been affected by technological changes (addition of temper, levigation of clay), the trace element patterns of the amphorae should reflect those of the clay materials used (Pillay et al., 2000), therefore allowing us to constrain their provenance better (Lee, 2002; Jia et al., 2004). In this case study, one group of trace elements and two major potentially immobile elements (Y, Ce, Cr, V, Al, Ti) were selected to define possible sediment sources (Bhatia and Crook, 1986; McLennan, 1992; Yang et al., 2003, 2004).

Comparisons among potsherds from Sidi Zahruni, those attributed to the site, ones from other workshops in the Nabeul area and the clay materials collected near Sidi Zahruni all show that only one clay (Clay2) always overlaps with potsherds from

Sidi Zahruni and is attributed to the site, whereas the other (Clay1) is also isolated from the potsherds from Choggafia and Labayedh (Fig. 11). This indicates that the amphorae from the latter workshops were produced with a different clay material from that of Sidi Zahruni. Therefore, Clay2, collected from the Upper Miocene clay deposits of the Oued El Kebir formation, was most probably the one used to produce amphorae in the Sidi Zahruni workshop, independently of the type of the final product. This is also confirmed by the distribution of trace elements normalised to average continental crust, which demonstrates that the amphorae from Sidi Zahruni and those attributed to the site (Fantuzzi et al., 2015a) both show patterns very similar to those of Clay2 (Fig. 12). Small differences in some trace element concentrations, essentially between amphorae attributed to Sidi Zahruni and Clay2 (Fig. 12c), may reflect the small natural inhomogeneity of raw materials from the same geological formation along sedimentary clayey deposits. From an archaeological viewpoint, this is a satisfactory hypothesis: due to the longer production period of the centre (about 200 years), various levels from the same outcrop could have been exploited.



1. Download high-res image (490KB)
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Fig. 11. Binary variation diagrams of: a) TiO_2 vs. Cr; b) Al_2O_3 vs. Y; c) Al_2O_3 vs. TiO_2; d) V vs. Y; e) Ce/Y vs. Cr/Y for the amphorae from Sidi Zahruni, the clays collected near the site and the amphorae attributed by Fantuzzi et al. (2015a) to Sidi Zahruni, Choggafia and Labayedh.

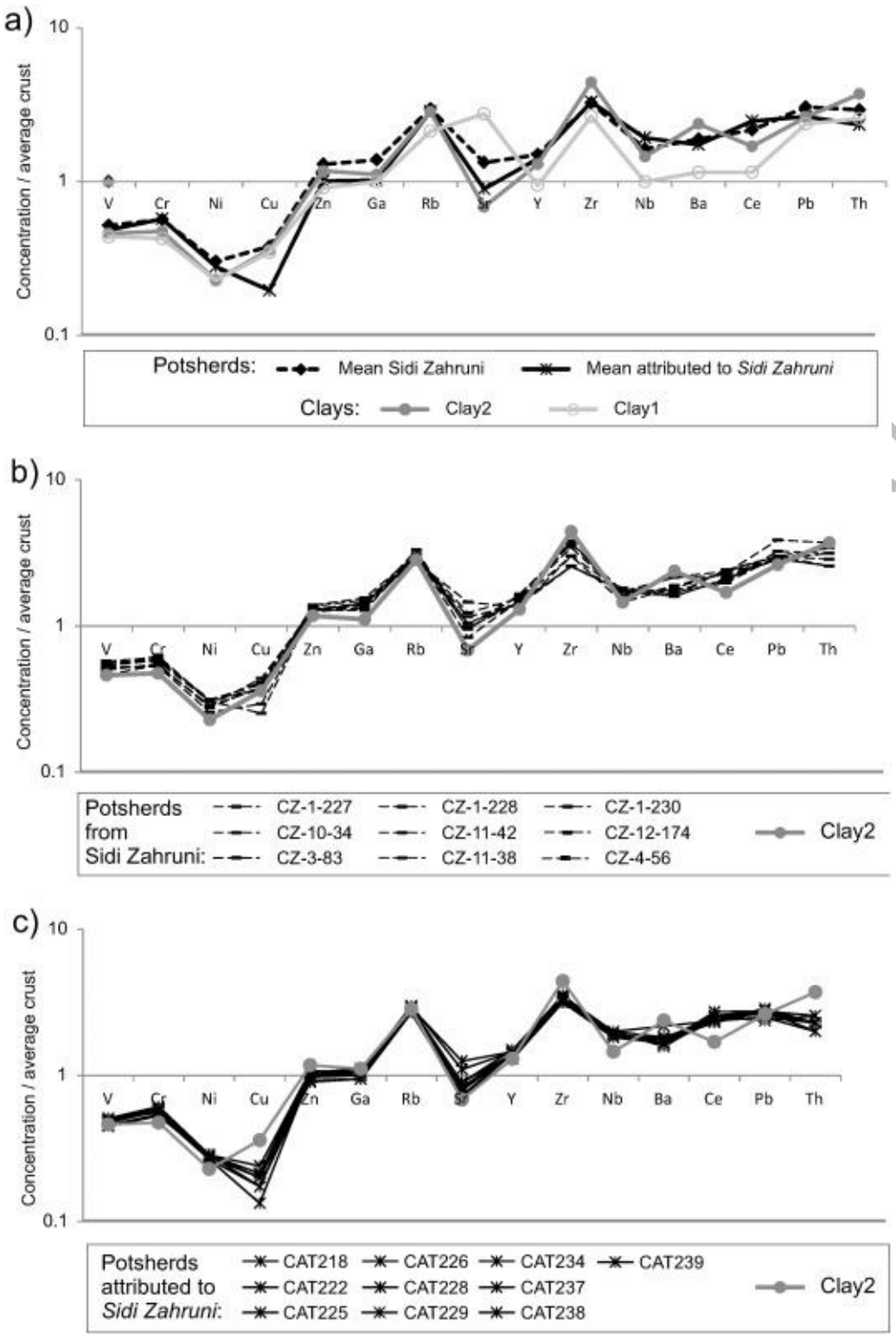

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Fig. 12. Normalised elemental distribution of some trace and rare earth elements of: a) clay materials collected near Sidi Zahruni and average values of potsherds analysed in present work and attributed to *Sidi Zahruni* (italic) from Fantuzzi et al. (2015a); b) some selected potsherds from Sidi Zahruni here analysed and Clay2; c) the samples attributed to *Sidi Zahruni* from Fantuzzi et al. (2015a) and the Clay2.

Therefore, it can be established a new and consistent reference group of Keay 25.2 amphorae, together with those found in Catalonia by Fantuzzi et al. (2015a) according to their chemical composition, in view of available mineralogical and petrographic information, although some technological changes did occur.

6. Conclusions

This archaeometric study of Keay 25.2 amphorae from Sidi Zahruni (north-eastern Tunisia) based on petrographic and mineralogical compositions provided significant information to describe the nature of this ceramic class. Petrographically, all samples were very similar, with small differences in the textural features of the inclusions revealed by image analysis. The different proportions between matrix and inclusions, especially quartz, are responsible for the small geochemical differences among the samples. Also from a mineralogical viewpoint, the amphorae from Sidi Zahruni are homogeneous, indicating standardised production technology in terms of source clay, paste preparation and firing conditions. Geochemical comparisons with a group of African amphorae found in Tarragona (Spain) and attributed to the Sidi Zahruni workshop indicates that different variants of amphorae produced at Sidi Zahruni are similar, especially in terms of trace element patterns, indicating that the same clay source was used for these productions. In more detail, the amphorae produced at Sidi Zahruni were made from the clay outcropping in the nearby Upper Miocene deposits of the Chabat Al Qola. The availability of large quantities of raw materials would have been an obvious factor in locating a flourishing ceramic industry in the Nabeul area. The different geochemical composition of the amphorae produced in other workshops in the area indicates that the potters followed their own production strategies and used different base clays.

A new and consistent reference group for the Keay 25.2 amphorae from Sidi Zahruni is now established, and is also representative of the various fabrics found at the site. This reference group will be useful for further provenance studies on Roman amphorae, to identify possible exportation from Sidi Zahruni to other sites, especially in Hispania, where the Keay 25.2 type of amphora has often been found, and to better define how this type spread and where it was imitated. Such data will aid interpretation of the socio-economic relations between various regions, trade routes and the technological evolution of societies.

Acknowledgements

The authors would like to thank the Institute of Patrimony of Tunis (INP) for providing access to the archaeological site of Sidi Zahruni. They are also very grateful to G. Walton, who revised the English text, and two anonymous referees and Faiza Bergaya, whose comments improved the manuscript. This research is part of ongoing collaboration between the Unité de Recherche: Pétrologie crystalline et sédimentaire (Université Tunis El Manar), the Department of Geosciences of the University of Padova (Italy) and the Geology Department of the Universitat Autònoma de Barcelona (Spain). It benefited by funding from the University of Padova (Erasmus Mundus Al-IDRISI II scholarship and International cooperation), from the Spanish Ministerio de Economía y Competitividad (project

CGL2013-42167) and from technical and human resources of the Faculty of Sciences of the University of Tunis.

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