**Archaeometric analysis of Late Roman amphorae from *Africa* in the ancient city of *Iluro* (Mataró, Catalonia, Spain)**

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ABSTRACT

In this paper, the results of the archaeometric study of an assemblage of Late Roman amphorae, found in several contexts from the ancient city of *Iluro* (Mataró, Catalonia, Spain) and with a presumable origin in Roman *Africa*, is presented. A total of 57 samples have been analysed, by means of Optical Microscopy (thin-section analysis), X-Ray Fluorescence and X-Ray Diffraction, in order to obtain an integrated petrographic, chemical and mineralogical characterisation and, from this data, to shed light on their provenance. The results indicate the presence of a large number of fabrics, most of them with a Tunisian provenance; a fabric probably from Algeria was also found, as well as a few chemical-petrographic loners that should be related to a provenance out of *Africa*. Concerning the Tunisian fabrics, the comparison with data from production centres allows for a more precise provenance hypothesis for many of them. These results are useful for the study of the trade networks of *Iluro* in Late Antiquity, since they provide new evidence on the diversity of transport amphorae that were arriving to this urban centre in *Hispania Tarraconensis*, showing a more complex reality than initially suggested by the archaeological evidence.

Keywords: amphora, Late Antiquity, petrography, XRF, provenance, Catalonia

## INTRODUCTION

The transport amphorae from the north of Africa, in particular from Tunisia, have been widely reported in many studies of Late Antique archaeological contexts in the western Mediterranean, including the current Catalan territory (*e.g.* Keay 1984; Járrega 1993, 2013; Aquilué and Burés 1999; Remolà 2000; Cela and Revilla 2004). They are usually the most frequent amphorae in these contexts, in association with the transport of oil, wine, *garum* or other foodstuffs (see discussion in Bonifay 2004; Bonifay and Garnier 2007).

A large assemblage of Late Roman amphorae was uncovered in a series of excavations at the archaeological site of *Iluro* ―currently Mataró (Catalonia, Spain)― conducted during the last decades

(Fig. 1) (Cerdà *et al*. 1997; Cela and Revilla 2004; Revilla 2011). This was a secondary urban centre in *Hispania Tarraconensis*, the pottery materials revealing, in any case, an important commercial activity in the area, with the arrival of amphorae and their contents, fine wares, cooking wares and other products from various Mediterranean regions. More than 600 Late Roman amphorae were documented in these excavations, the imports from the Roman dioecesis of *Africa* being the most represented with almost half of the total (Cerdà *et al*. 1997; Cela and Revilla 2004).

In the present paper, a large sample of presumably African amphorae uncovered at the excavations of *Iluro* is archaeometrically analysed, in order to examine the diversity of chemical-petrographic groups and, through this evidence, to provide information on the different production centres where these amphorae could have been produced, as well as on some aspects of their production technology. Some other archaeometric analyses of African amphorae in the current Catalan area have been carried out in the past, as part of broader studies along with other Late Roman amphorae. In some of these studies, the internal variability of the African amphorae was not explored, the analysis being focused on the differentiation of the major production areas in the Mediterranean (*Africa*, *Baetica*, *Lusitania*, etc.) (*e.g.* Remolà *et al*. 1993, 1996; Uscatescu and García 2005). However, recent studies on amphorae found in *Tarraco* (Fantuzzi *et al*. 2015a) and *Emporiae* (Fantuzzi *et al*. 2015b) revealed an important diversity of products from *Africa* (mainly from Tunisia), coming from different production centres and zones. On this basis, an archaeometric characterisation of African amphorae from the *Iluro* contexts is presented in this paper, aiming to provide new evidence on these materials and, through the provenance approach, to achieve a better understanding of the trade activity of this urban centre in Late Antiquity.

The macroscopic examination of these African amphorae found in *Iluro* reveals a significant variability of fabrics. Several workshops have been archaeologically identified in the Tunisian and Tripolitanian regions in the last decades and many of them have also been studied archaeometrically (in particular through petrographic analysis), which enables improved comparative studies with data from consumption centres. From a chemical point of view, a very limited number of studies are available (Liddy 1985; Taylor 1993; Taylor *et al*. 1997; Sherrif *et al*. 2002; Madrid and Buxeda 2010) and a comparison with their results is somewhat problematic in most cases. However, with regard to the petrographic research, an increasing number of studies on Tunisian amphora workshops can be found (Peacock and Tomber 1991; Bonifay *et al*. 2002, 2010; Sherriff *et al*. 2002; Capelli 2005a, 2005b, 2007; Ghalia *et al*. 2005; Capelli *et al*. 2006; Capelli and Bonifay 2007, 2014). The comparison with reference groups from production centres is essential in the case of Tunisian amphorae, since the different workshops share broad similarities in their fabrics, thus it is necessary to take into account the compositional, textural and technical particularities in each case (Capelli and Bonifay 2007, 2014). Also the archaeological information must be considered and integrated with the archaeometric results; a number of archaeological studies on Tunisian workshops have been published so far (Peacock *et al*. 1989, 1990; Stirling and Ben Lazreg 2001; Bonifay 2004; Bonifay *et al*. 2005; Mrabet and Ben Moussa 2007; Nacef 2007, 2010; Dore 2011). In addition, some archaeometric studies on consumption centres (Gandolfi *et al*. 2010; Bonifay *et al*. 2011; Lemaître *et al*. 2011; Fantuzzi *et al*. 2015a, 2015b) have provided further evidence on the characterisation and provenance of Tunisian amphorae.

## MATERIALS

A total of 57 amphorae possibly related —according to the archaeological study— to an African provenance have been sampled for archaeometric analysis. A variety of Late Roman amphora types is included in this sampling (Table 1). The majority of them might be associated, from a first macroscopic examination, with various Tunisian amphorae. Five samples correspond to types Africana IIA, Africana IIC and Africana IID, dated from the 3rd century to the first half of the 4th century. Thirteen samples are related to typical fourth-century or early fifth-century Tunisian types, such as Keay 25.1, Keay 25.3, *Spatheion* 1, Keay 27 and Keay 24, while four samples belong to other types (Keay 35B, Keay 36, *Spatheion* 2A) more characteristic of the 5th century. The most represented amphorae in the analysed assemblage correspond to late fifth-century and especially sixth-century types (23 samples), in particular Keay 62 (variants A and, to a lesser degree, B, D and K), Keay 55 (mainly variant A), and Albenga 11-12/Keay 62Q, as well as a single individual of type Keay 57. Another six samples could not be related to any typology, although their macroscopic characteristics indicate their possible Tunisian provenance.

In addition to the presumably Tunisian amphorae, we have included into the analysis four samples that were ascribed to Mauretanian types Keay 1A and Keay 1B (3rd-4th centuries) according to their form and macroscopic aspect, as well as two possible Tripolitanian amphorae of type Tripolitanian III/Keay 11 (mainly dated to the 3rd century), as suggested by the archaeological study (Cerdà *et al*. 1997; Cela and Revilla 2004).

## METHODS

An integrated archaeometric approach was followed through chemical, mineralogical and petrographic analyses of the samples.

The chemical analysis was carried out by means of X-Ray Fluorescence (XRF) and it was performed using a Panalytical-Axios PW 4400/40 spectrometer (see for analytical routine Fantuzzi *et al*. 2015a). This allowed for the identification of 29 major, minor and trace elements, including $Fe_2O_3$, $Al_2O_3$, MnO, $P_2O_5$, $TiO_2$, MgO, CaO, $Na_2O$, $K_2O$, $SiO_2$, Ba, Rb, Th, Nb, Pb, Zr, Y, Sr, Ce, Ga, V, Zn, Cu, Ni, Cr, Mo, Sn, Co and W. However some of these elements were discarded from the analysis: Mo and Sn due to their low counting statistics, while Co and W due to a possible contamination from the tungsten carbide cell of the mill used in the preparation of the samples. Chemical data was examined through different statistical procedures —Cluster Analysis and Principal Component Analysis (PCA)— using the software S-PLUS 2000, after an additive log-ratio (alr) transformation of the values obtained by XRF (Aitchison 1986; Buxeda 1999).

The mineralogical and petrographic description of thin sections through Optical Microscopy (OM) was conducted using an Olympus BX41 microscope, working between x20 and x200 magnification, equipped with a DP70 Olympus digital camera. For the description of the ceramic fabrics a modified version of the system proposed by Whitbread (1989, 1995) was followed (see Quinn 2013).

The mineralogical composition was examined also through X-Ray Diffraction (XRD) using a PANalytical X'Pert PRO MPD alpha 1 diffractometer, working with the Cu–Kα radiation (l = 1.5406 Å). Spectra were taken from 5 to 80° 2θ, using a step-size of 0.026°2θ and a step-time of 47.5 s. Crystalline phases were determined through the software HighScore Plus by PANalytical, including the Joint Committee of Powder Diffraction Standards (JCPDS) data bank. This is essential to detect cryptocristalline minerals not observable under the polarising microscope and that can include crystalline phases formed during firing and secondary minerals formed by alteration or contamination processes due to use or to the deposition on the archaeological deposit. On the basis of the phases observed in the diffractograms an estimation of the equivalent firing temperature (EFT) (Roberts 1963) was carried out (*e.g.* Maggetti 1982; Cultrone *et al*. 2001; Buxeda and Cau 2004; Maggetti *et al*. 2011).

All the amphora samples were analysed through these three techniques, except sample ILU033 that was subjected to OM only, since the small sample size did not allow for XRF and XRD preparation.

## RESULTS

### *Chemical results*

The normalised chemical concentrations of 25 elements ($Fe_2O_3$, $Al_2O_3$, MnO, $P_2O_5$, $TiO_2$, MgO, CaO, $Na_2O$, $K_2O$, $SiO_2$, Ba, Rb, Th, Nb, Pb, Zr, Y, Sr, Ce, Ga, V, Zn, Cu, Ni and Cr) for 56 amphora samples are presented in Table 2. The chemical data show that the majority of the samples present a calcareous composition (CaO >5%), but usually not a highly calcareous one, since there is not any sample with CaO over 20% and only six samples present a CaO percentage over 15%. The content of $SiO_2$ tends to be high (usually 60-70%), while $Al_2O_3$, $Na_2O$, $K_2O$ and MgO concentrations are relatively low in most cases (below 16%, 1%, 3% and 3%, respectively, with few exceptions), and $Fe_2O_3$ ranges in general between 4.5-6.5%. Similar patterns have been recorded in other African amphorae assemblages (*e.g.* Madrid and Buxeda 2010; Fantuzzi *et al*. 2015a, 2015b) and related to quartz-rich, non-micaceous fabrics, with a ferruginous matrix and a variable content of calcareous inclusions.

A detailed examination of the chemical variability of this data set can be made through calculation of the Compositional Variation Matrix (CVM) (Aitchison 1986, 1992; Buxeda *et al*. 1995; Buxeda 1999; Buxeda and Kilikoglou 2003). This yields a total variation (*vt*) of 1.80, which is indicative of a polygenic population, since *vt* values for a monogenic sample should be much lower (usually <0.30), as demonstrated by previous studies (see Buxeda and Kilikoglou 2003). According to the CVM, most of this variation is associated with the following components: CaO ($\tau_{.CaO}$ = 12.94), Pb ($\tau_{.Pb}$ = 5.92), Cu ($\tau_{.Cu}$ = 5.78), $Na_2O$ ($\tau_{.Na2O}$ = 5.69), $P_2O_5$ ($\tau_{.P2O5}$ = 5.13), Sr ($\tau_{.Sr}$ = 4.93), Ba ($\tau_{.Ba}$ = 4.09), MnO ($\tau_{.MnO}$ = 4.03), MgO ($\tau_{.MgO}$ = 3.56) and Zr ($\tau_{.Zr}$ = 3.14).

For the statistical treatment of the chemical results, the raw compositional data were used after an additive log-ratio transformation (alr) using Ga as divisor, since it is the element with the lowest variability within the data set according to the CVM. A cluster analysis (using the centroid agglomerative method and the squared Euclidean distance) was carried out on the subcomposition $Fe_2O_3$, $Al_2O_3$, MnO, $TiO_2$, MgO, CaO, $K_2O$, $SiO_2$, Ba, Rb, Th, Nb, Zr, Y, Sr, Ce, V, Zn, Cu, Ni and Cr. $P_2O_5$, $Na_2O$ and Pb,

that are related to high $\tau_{.i}$ values in the CVM, were not included in this statistical analysis due to possible contamination problems related to these elements. The resulting cluster tree is shown in Fig. 2, where some chemical groups can be differentiated (CG1 to CG6); the mean chemical composition for these groups is given in Table 3.

Sample ILU005 behaves as a chemical loner, consistent with its particular composition (Table 2). Besides this loner, a first general division of the data set can be observed between the low calcareous and the calcareous samples (Fig. 2). There are six samples (ILU042, 043, 044, 075, 086, 087) with a very low calcareous content (CaO <3.2%) that share, also, a general compositional similarity, so they can be included in a same chemical group (CG1: Table 3). Another four samples (ILU046, 059, 060, 064) fall at the other end of the dendrogram, showing a slightly higher content of Cu and some other particularities in each case (Table 2). Samples ILU059 and ILU060 present a close chemical relationship and form a separate chemical group (CG6: Fig. 2, Table 3), while ILU046 and ILU064 are clear chemical loners. Some other samples (ILU054, 061, 066, 069, 074) can also be considered as loners, as suggested by the cluster tree (Fig. 2) and their particular chemical compositions (Table 2). These loners are related —in a varying degree— to higher concentrations of $Al_2O_3$ (ILU046, 054), MgO (ILU046, 069), $K_2O$ (ILU046, 061, 066), Rb (ILU046, 061), Sr (ILU054), Zn (ILU064) and Cr (ILU054 and, to a lesser extent, ILU064 and 069), in addition to some other particularities.

The remaining samples of the assemblage belong to the same general cluster in Fig. 2, where four main chemical groups (CG2 to CG5) as well as a few loners (ILU008, 022, 076, 082, 083) can be observed. A Principal Component Analysis (PCA) of this large cluster enables a better examination of the chemical separation of these groups. The plot of the first two principal components, PC1 and PC2 (both together accounting for 58.4% of the total variance), is shown in Fig. 3a; both PC1 and PC2 are dominated by CaO, Ba, Sr, Cu and MnO, while MgO is also important in PC2. This explains the negative PC1 and PC2 scores for samples with relatively low CaO and Sr concentrations (CG5 and loner ILU008), while the more calcareous samples (CG2, CG3 and some loners) show positive PC1 values, with a certain variation in PC2 according to their differences in the other elements aforementioned.

In addition to this variation concerning the calcareous composition, some other differences can be found between the chemical groups CG2 to CG5. This can be better observed in a second PCA excluding CaO, Sr and Ba from the analysis; Cu and MnO can also be discarded, since their variation does not seem to be relevant for the definition of the chemical groups, as suggested by the examination of the chemical data (Table 2). In this new PCA (Fig. 3b), PC1 is largely dominated by MgO and $K_2O$, while PC2 is mainly related to variations in Zn and Zr; both PCs explain 61.3% of the total variation. A clear separation of CG2 (ILU017 and ILU031) can be observed in this PCA, due to its high Zn and low Zr concentrations (Table 3).

The PCAs in Fig. 3 suggest that the chemical group CG3, defined from the cluster analysis in Fig. 2, actually present some internal variability. A close compositional relationship can be found especially between samples ILU016 and ILU050 (subgroup CG3.1); in Fig. 3b, they separate from the remaining samples in CG3 (ILU039, 041, 067) due to their lower MgO and $K_2O$ percentages (Table 2). In addition, a possible relation between ILU039 and ILU067 can also be observed, however not as evident as in CG3.1.

The chemical group CG4, on the other hand, comprises a large number of samples (n=24) (Fig. 2). The calculation of the CVM for this group yields a *vt* value of 0.51, which should be interpreted as indicative of a polygenic population (Buxeda and Kilikoglou 2003), although it is possible that the internal subgroups might be related in a broad sense. From the cluster analysis in Fig. 2 two main subgroups (CG4.1 and CG4.2) can be distinguished. The main differences between them are related to the lower content of MgO and $K_2O$ in CG4.1 (samples ILU010, 014, 051, 062), as clearly seen in Fig. 3b and Table 3. The subgroup CG4.2 includes a total of 16 samples; the CVM for this subgroup provides a *vt* value of 0.35, although the exclusion of $P_2O_5$, $Na_2O$ and Pb from the analysis yields a *vt* of 0.19, suggesting that this large subgroup may represent a monogenic population.

*Petrographic and mineralogical results*

The petrographic analysis of the 57 amphora samples through OM allowed us to differentiate a number of fabric groups and individual fabrics. In addition, the XRD results provided information on the mineral phases for each sample, being particularly important for the identification of secondary and firing minerals (Table 4).

The vast majority of the samples (51) can be included in a same general petrographic group, PG-1 ('quartz and calcite fabric group'), in which the non-plastic inclusion composition consists mainly of monocrystalline quartz (usually rounded in the coarser grains) and variable amounts of calcareous inclusions (microfossils and limestone fragments), as well as —in some cases— quartz-sandstone fragments and iron oxides; occasionally, a trace presence of metamorphic or volcanic inclusions can also be found. The clay matrix is generally iron-rich, although in some samples a mixing with a calcareous clay is observed. Based on the relative frequency of these components and on textural characteristics (i.e. packing, grain-size distribution, frequency and roundness of inclusions, porosity), a total of nine fabrics (PG-1.1 to PG-1.9), each represented by more than one sample, can be distinguished (Fig. 4):

- PG-1.1 ('fine- and coarse-grained quartz': ILU036, 037, 038; possibly ILU034): the fine fraction of quartz is moderately abundant and dominant over the coarse fraction, which is composed of common quartz grains in the range 0.20-0.30 mm (Fig. 4a). Few inclusions of micritic calcite and microfossils are visible, however the calcareous inclusion were affected by the firing process, with a high grade of alteration; the EFT can be estimated between 850-950ºC (Table 4), most probably over 900ºC as indicated by the intense peaks of pyroxene, plagioclase and hematite. Few voids (mesovughs and mesovesicles) are visible.

- PG-1.2 ('quartz-sandstone and abundant fine quartz': samples ILU019, 042, 043, 044, 073, 086, 087; possibly also ILU089, 090): characterised by a predominant fine fraction of angular quartz (mode 0.05-0.10 mm) and a less frequent coarse fraction dominated by fragments of quartz-sandstone with a calcareous cement (<3.5 mm, in many cases >1.0 mm) (Fig. 4b). The presence of quartz in the coarse fraction (usually fine or medium sand) is variable, from rare or very scarce (ILU043, 073, 086, 087) to slightly more common (ILU019, 042, 044), however always subordinated to the other components. Voids are not abundant (meso-sized vughs and vesicles). XRD reveals for these samples an EFT between 850-950ºC, although with some mineralogical differences related to variations in their CaO

content (Table 4). Some low calcareous samples (ILU042, 043, 044, 087) show partially decomposed illite-muscovite as well as intense peaks of hematite, while in a few samples with more CaO (though not very calcareous) small peaks of gehlenite and pyroxene can be observed (ILU019, 073). The samples ILU089 and ILU090 might be related to this fabric, although they show a higher frequency of fine calcareous inclusions, highly altered by the firing process; the estimated firing temperature is higher in ILU089, where the intense peaks of pyroxene and plagioclase (and an advanced decomposition of illite-muscovite) suggest an EFT over 900ºC (Table 4).

- PG-1.3 ('dominant fine quartz': ILU035, 040, 045, 085): fabric with a dominant fine fraction of quartz (silt to very fine sand), in addition to common coarse grains (0.20-0.30 mm) which are subordinated to the fine fraction (Fig. 4c). Compared with PG-1.1, the fine quartz is much more abundant and the coarse quartz slightly less frequent. Few calcareous inclusions (fine micritic calcite and rare microfossils, as well as some coarse limestone fragments) are also observed, more than in PG-1.2, while quartz-sandstone fragments are rare. The EFT can be estimated between 850-950ºC (Table 4).

- PG-1.4 ('coarse-grained quartz, few fine fraction': ILU007, 011): a dominant, moderately sorted coarse fraction of quartz (mode 0.15-0.25 mm, but common up to 0.60 mm) is observed, while the fine fraction (mainly quartz) is not very abundant (Fig. 4d). The bimodal distribution suggests the presence of an added temper. Some scarce iron oxides and polycrystalline quartz (usually <0.25 mm) can be found in the coarse fraction. Porosity is higher than in previous fabrics, with meso- and macro-sized vughs, vesicles and elongated voids with a parallel orientation to the surface. The EFT is variable, from ≤800/850ºC in ILU011 to 850-950ºC in ILU007 (Table 4).

- PG-1.5 ('coarse-grained quartz, very scarce fine fraction': ILU033, 054): fabric characterised by a predominant, well-sorted coarse fraction (fine/medium sand of quartz), in contrast with an almost absent silt fraction of quartz (Fig. 4e). Some coarse Fe-rich nodules and calcareous inclusions (micritic calcite, rare microfossils) can be found, especially in ILU054, in which a scatter of abundant fine iron oxides is observed throughout the clay matrix; these are absent in ILU033, a difference possibly related to the firing conditions. Few voids are present (meso-sized vughs and vesicles).

- PG-1.6 ('coarse- and fine-grained quartz with coarse iron nodules': ILU018, 075; probably also ILU006, 012): a clear dominance of quartz in both the fine (<0.10 mm) and the coarse fraction (mode <0.30 mm) is observed, along with frequent coarse iron nodules (usually >0.50 mm) (Fig. 4f); few quartz-sandstone fragments can also be found. Samples ILU006 and ILU012 show a slightly coarser texture and less frequent (but still important) iron nodules. Common to frequent voids (meso- and macro-sized vughs and vesicles mainly) are observed. For all these samples the EFT can be estimated between 850-950ºC, except for ILU018 with a higher EFT (≥950/1000ºC: Table 4).

- PG-1.7 ('coarse- and fine-grained quartz, with quartz-sandstone and large calcareous clay streaks': ILU010, 051, 062; probably also ILU014, 016, 050): the inclusions in this fabric comprise abundant fine and coarse quartz (mode <0.35 mm), occasional large fragments of quartz-sandstone with a Fe-rich cement and a characteristic presence of very large streaks of a calcareous clay (with a variable frequency in each sample) that can reach up to several millimeters long (Fig. 4g). In some cases, less evident streaks of a reddish clay can also be found, indicating a clay mixing. Voids are frequent, mainly as meso- and macro-sized vughs and vesicles, in addition to abundant fine elongated voids in ILU010 and

051. The three samples in this fabric show an estimated EFT of 850-950ºC (Table 4). Another three samples are probably related to this fabric, with textural and compositional similarities, however with some particularities in each case: ILU050, with a yellowish clay matrix, possibly more calcareous but also affected by a higher firing temperature (Table 4); ILU014, again with a high EFT (Table 4) and a slightly overfired matrix, shows a lesser abundance of fine quartz; ILU016 has a less abundant fine fraction (like ILU014), in addition to a lighter-coloured clay matrix that suggests a higher calcareous content (large streaks of two different clays are observed, as in the main fabric).

- PG-1.8 ('fine microfossils, calcite and quartz, with coarse limestone and clay pellets': ILU017, 031): in this fabric, the coarse fraction (>0.20 mm) includes large (usually >1 mm) limestone fragments as well as pellets of a yellow-red clay (also some large clay streaks in ILU017). Calcareous microfossils and quartz are also present in the coarse fraction, normally <0.40/0.50 mm. However, a clearly predominant, well-sorted fine fraction is observed, with abundant quartz, calcite and microfossils (mode 0.10-0.15 mm, the fraction under 0.10 mm being very scarce) (Fig. 4h). The two samples in this fabric show some differences in the frequency of voids and the colour of the clay matrix, however their petrographic relationship is evident. In both samples the matrix is optically active, in agreement with the low EFT estimated through XRD (≤800/850ºC) (Table 4).

- PG-1.9 ('limestone, microfossils and quartz, with accessory basic igneous inclusions': ILU056, 065): this fabric presents an abundant, well-sorted coarse fraction of limestone fragments, calcareous microfossils (including shell fragments, foraminifera and some echinoids) and rounded quartz, as well as an accessory —but quite distinctive— presence of coarse basic igneous inclusions (clinopyroxenes and rare plagioclase —both with zoning in some crystals— along with occasional fragments of basalt) (Fig. 4i). A clear bimodal distribution is seen, with a scarce fine fraction of quartz and calcareous inclusions. In sample ILU056 the mode of the coarse fraction is slightly higher than in ILU065, and the fine fraction scarcer, both representing textural variants of the same fabric. Voids are abundant (vughs, vesicles and elongated voids). The matrix displays no optical activity; in ILU065 it shows a dark, not oxidised core. The EFT can be estimated around 850-950ºC in both samples (Table 4).

In addition to these fabrics, a large number of petrographic loners (ILU008, 009, 013, 015, 022, 039, 041, 049, 064, 067, 068, 069, 074, 076, 082, 083) within the 'quartz and calcite fabric group' (PG-1) is present. The mineral phases seen through XRD indicate, for these samples, the presence of calcareous fabrics with EFTs of ≤800/850ºC or 850-950ºC (Table 4). The only exception is ILU069, with peaks of spinel and a total decomposition of phyllosilicates, suggesting high firing temperature (≥950/1000ºC) and a low calcareous composition.

Only six samples of the analysed assemblage are not included in PG-1 (Fig. 5). Two of them (ILU059, 060) form a same petrographic group (PG-2) with two different but related fabrics (PG-2.1 and PG-2.2: Fig. 5a-b), each with an abundant, moderately-sorted coarse fraction (>0.20 mm), representing an added temper. It is composed of acidic metamorphic rock fragments (quartzite, quartz-mica schist and gneiss) and monocrystalline quartz (with rounded grains), in addition to a subordinated presence of alkali feldspars, calcareous inclusions, polycrystalline quartz, plagioclase (zoned in some cases) and fine-grained sedimentary rock fragments, as well as, more rarely, arenites, clinopyroxene and basaltic rock fragments. The fabric PG-2.2 (ILU060) is characterised by a more frequent coarse fraction than PG-2.1

(ILU059). The fine fraction (silt and fine/very fine sand), quite abundant as well, comprises quartz, some micas (biotite and muscovite) and calcareous microfossils (more visible in ILU059 that is lower fired). The clay matrix shows a lighter colour in the core, and darker surfaces; it is optically inactive, in consistency with an EFT between 850-950ºC as inferred from XRD results (Table 4).

The petrographic group PG-3 includes two samples (ILU046, 061) with broad compositional similarities (acidic plutonic and metamorphic rocks, with feldspars, quartz and calcareous inclusions), however with a clearly different fabric in each case. The fabric PG-3.1 (ILU046: Fig. 5c) contains an abundant coarse fraction (>0.25 mm) with fragments of granite (and some granodiorite) and metamorphic rocks (metagranite, meta-argillites and schist), as well as alkali feldspar, quartz and a minor presence of sedimentary rock fragments. The fine fraction is scarce and consists of calcite, quartz and few micas (biotite and muscovite), calcareous microfossils and alkali feldspars. It is a high-fired fabric, with an EFT over 950/1000ºC (Table 4). Conversely, in the fabric PG-3.2 (ILU061: Fig. 5d), the granitic rock fragments in the coarse fraction are clearly subordinated to a predominance of metamorphic rock fragments (quartzite, quartzmicaschist, metagranite), alkali feldspar, polycrystalline quartz (grading into quartzite) and monocrystalline quartz; the scarce fine fraction comprises, instead, micas (biotite and muscovite), quartz and calcareous microfossils. XRD indicates for this fabric an EFT between 850-950ºC (Table 4).

Finally, samples ILU005 and ILU066 represent petrographic loners, each with a particular fabric. The fabric PG-4 (ILU005: Fig. 5e) shows an abundant coarse fraction (>0.20 mm) of metamorphic rock fragments (schist, quartzite, phyllite), in addition to some quartz (mono- and polycrystalline) and garnet inclusions. The fine fraction is also abundant (especially <0.10 mm), comprising quartz and altered micas (possibly muscovite). It is a porous fabric, with many mesovoids and some macrovoids (vughs and vesicles). XRD suggests high firing temperatures (≥950/1000ºC), with spinel and small peaks of mullite as firing phases, while phyllosilicates are totally decomposed. The fabric PG-5 (ILU066), on the other hand, shows a fabric with fine-grained inclusions (<0.20 mm but especially <0.05/0.10 mm) of quartz, mica (biotite and muscovite) and calcareous particles (Fig. 5f); coarse calcareous inclusions and some calcareous clay pellets can also be found, but very occasionally. It is a fabric with few voids; the mineral phases observed through XRD suggest an EFT between 850-950ºC (Table 4).

## DISCUSSION

The integration of the chemical, petrographic and mineralogical evidence allows for a better characterisation of the analysed amphorae. Combined with the archaeological information, these results provide new evidence on the diversity of chemical-petrographic groups, their possible provenance and some aspects of their technology of production.

The large ‘quartz and calcite fabric group’ (PG-1), that includes 51 samples (Table 5; Fig. 6-7), can be considered equivalent to the ‘Tunisian Fabric’ or ‘Aeolian Group’ defined by Capelli (2005a, 2005b), for which a general provenance in northern Africa (especially in Tunisia) is proposed. Both the petrographic and the chemical evidence suggest the existence of a large variability within this group. A more specific provenance hypothesis for these samples relies on the comparison with archaeometric

reference groups of Tunisian production centres. In addition to the increasing number of publications on this topic in the last years (see Introduction), we also had the opportunity to compare the thin sections of African amphorae from *Iluro* with the fabric reference collection for Tunisian workshops of the University of Genova. This comparison enabled a better definition of the provenance hypothesis for many of the analysed African samples from *Iluro* (Table 5; Fig. 8). In some cases, the integration of the archaeometric results and the archaeological information allowed for a typological reclassification of the samples (ILU009 and ILU049). This integrated archaeometric and archaeological approach is particularly important in the case of provenance studies on Tunisian amphorae, as various studies have shown (Capelli and Bonifay, 2007, 2014). For some of the main African fabrics identified in *Iluro*, a clear association with archaeometrically known production centres was possible, while for others a more precise provenance hypothesis is not possible so far, as summarized below.

- PG-1.1: this fabric, observed in samples of type Keay 62 (Fig. 6), is equivalent to the typical fabric of Henchir ech Chekaf workshop in central-eastern Tunisia (Capelli, 2005a, 2005b, 2007), where the production of this amphora type has been widely documented (Peacock *et al.* 1989; Bonifay 2004: 33-35; Nacef 2007, 2010). The chemical and petrographic results suggest the use of a calcareous clay paste with no clear evidence of an added temper; the workshop is located near Pliocene clayey deposits that could have been used as a source of raw materials (Capelli and Bonifay 2014). The samples in this fabric share a similar chemical composition, not only between them but also with many samples in other fabrics that form all together the chemical subgroup CG4.2 (Fig. 2; Table 5), which ―as discussed in the chemical results― might be considered a monogenic population according to its total variation value.

- PG-1.2: this fabric is characteristic of the northeastern Tunisian workshop of Sidi Zahruni, in the zone B of Nabeul area (Bonifay 2004; Capelli 2005a, 2005b; Ghalia *et al.* 2005; Bonifay *et al.* 2010; Gandolfi *et al.* 2010). The seven samples in PG-1.2 are related to the amphora types Keay 55, Keay 35B and Keay 62A (Fig. 6), which are all archaeologically documented in Sidi Zahruni. Five of the samples belong to the chemical group CG1, except for ILU019 and ILU073 that show slightly higher CaO percentages and ―in ILU019― lower concentrations of Ba, Zr and Sr (Table 2). In general, the archaeometric results indicate the use of an iron-rich, low calcareous or border calcareous clay paste, with abundant fine quartz; the large quartz-sandstone fragments are not necessarily representing an added temper from a different source, since the clayey deposits that are available near the workshop contains intercalations of these rocks, according to Ghalia *et al*. (2005). Two samples (ILU089, 090), on the other hand, resemble much PG-1.2 in thin section, but show strong compositional similarities to PG-1.1 since they are included in the chemical subgroup CG4.2; both samples are Keay 62Q amphorae (Fig. 6), for which a possible origin in the Nabeul area has been proposed (Bonifay 2004), although similar forms (*e.g.* Albenga 11-12) may have been produced in the Sahel area, in the Roman province of *Byzacena* (Gandolfi *et al*. 2010; Bonifay *et al*. 2011; Capelli and Bonifay 2014). In our case, the fabric characteristics might suggest a provenance in Nabeul, but the chemical similarity to the products from Henchir ech Chekaf does not allow us to exclude an hypothetical provenance in the Sahel; further investigation on the Keay 62Q production centres (unknown so far) is still needed in order to verify these hypotheses.

- PG-1.3: it presents partial petrographic similarities with PG-1.2, a typical fabric from Nabeul zone B; this fabric similarity has also been suggested on the basis of macroscopic observations (M.

Bonifay, pers. comm.). However, the chemical evidence indicates a quite different composition, closer to the fabric PG-1.1 (from Henchir ech Chekaf), since the four samples (ILU035, 040, 045, 085) in PG-1.3 are also included in the chemical subgroup CG4.2, with a very low total variation (*vt*) value. The typological evidence favours the latter hypothesis, since all the samples belong to type Keay 62A (Fig. 6), with variants that are more characteristic of the Sahel area (see Bonifay 2004). However, taking into account the fabric particularities, we cannot exclude a production in Nabeul using raw materials that are similar in chemical composition to those of Henchir ech Chekaf, with a possible chemical overlapping area (Picon 1984; Buxeda *et al*. 1995) between different production centres. It seems preferable to wait for further archaeometric and archaeological evidence from production centres before drawing major conclusions on the provenance of this fabric.

- PG-1.4: poorly represented fabric (ILU007, 011), with no petrographic relation to any of the workshops known in Tunisia so far. It is found in a Keay 25.1 amphora —a type produced in both northern and central-eastern Tunisia (Bonifay 2004; Bonifay *et al*. 2005)— and an indeterminate rim (Fig. 6). Their chemical composition (CG4.2) is similar to samples from the Sahel area (PG-1.1) and others of less clear provenance (see PG-1.3 and samples ILU089, 090 in PG-1.2).

A few petrographic loners within PG-1 (ILU009, 013, 015, 068: Fig. 7), related to Keay 25 amphorae mainly (Table 5), are also included in the chemical subgroup CG4.2. For the moment, the existing reference data from production centres do not allow a more precise provenance for these samples. The amphora ILU013 bears a stamp *PGT* on the neck and a fabric that has been suggested to come from Nabeul (M. Bonifay, pers. comm.), however the chemical and petrograhic composition in this study does not provide conclusive evidence, apart from a general Tunisian provenance.

- PG-1.5: fabric observed in the two analysed amphorae of type Keay 24 (Table 5; Fig. 6). The same fabric has been reported in other samples of the same type and —based on archaeological and archaeometric evidence— a possible provenance in northwestern Tunisia or Algeria has been recently proposed (C. Capelli, pers. comm.), although no workshops have been located so far. The chemical composition in ILU054 (ILU033 was not analysed chemically) is quite different from the other African samples analysed in this work, especially for its higher content of $Al_2O_3$, Sr and Cr, and lower of Zr.

- PG-1.6: for the moment it is not possible to find a clear similarity between PG-1.6 and any known fabric from Tunisian production centres. However, the chemical results suggest a certain compositional similarity with PG-1.2 (Nabeul zone B), since samples of both fabrics form the chemical groups CG1 and CG5 (Fig. 2); the latter is differentiated by a CaO content of 4.0-6.6% (in contrast with 1.4-3.2% in CG1) and lower concentrations of Rb, Sr and Zr (Table 3). CG5 comprises samples of earlier amphora types (from the 3rd/4th to the 5th centuries) than those included in CG1 (late 5th/6th century); this may indicate a possible change in the source of raw materials for these fabrics from the late 5th or early 6th century, however a higher number of samples will be needed to assess this hypothesis. The amphora types included in PG-1.6 (Africana IIA, Keay 25.2, Keay 35B and Keay 62K: Fig. 6) would be consistent with a possible provenance in the zone B of Nabeul, as suggested by the chemical results, since it was the main production area of Keay 25.2 and Keay 35B amphorae (Bonifay 2004; Ghalia *et al.* 2005; Bonifay *et al.* 2010). An increased sample size and further archaeometric evidence from production centres could provide a more solid ground for this attribution.

- PG-1.7: the samples included in this fabric belong to amphora types Keay 27, Keay 36, *Spatheion* 1 (or Keay 25?) and Keay 7/25.1 (Table 5; Fig. 6-7). The same petrographic fabric has been documented in other consumption centres (Bonifay *et al.* 2011; Fantuzzi *et al*. 2015a, 2015b), particularly in Keay 27 and 36 amphorae. Even if no workshops have been identified so far, the archaeological evidence points to a probable provenance in northwestern Tunisia (west to Carthage) for this fabric (see Bonifay 2004: 22; Bonifay *et al.* 2011). The chemical results show a homogeneous composition for the main fabric (CG4.1), while other samples probably related to this fabric (ILU016, 050) form a separate chemical subgroup (CG3.1) due to their higher concentrations of CaO, Ba and (in ILU016) Sr. However, a chemical relation with the samples in CG4.1 can be observed after the exclusion of these elements from the analysis (Fig. 3b). This evidence would indicate the use of a more calcareous clay paste (or a higher amount of calcareous clay, considering that a mixing with an iron-rich clay is observed in the fabric) in these samples, as suggested also by the petrographic analysis.

- PG-1.8: the samples in this fabric (ILU017, 031: Table 5; Fig. 7) show also a similar chemical composition (CG2). Their calcareous content (CaO 13.6-14.1%) can be related to the abundant calcareous inclusions that are seen in thin section. It is not possible for the moment to associate this fabric with any specific Tunisian workshop or zone.

- PG-1.9: this fabric, found in two samples of the type Africana IIA (ILU056, 065: Fig. 7), is quite characteristic of the *Sullecthum*/Salakta workshops (Capelli 2005a, 2005b; Capelli *et al*. 2006; Capelli and Bonifay 2014), where the production of Africana IIA amphorae is well attested (Bonifay 2004). Both samples present some chemical differences between them (particularly in $Na_2O$, Ba, Sr, Cr and, to a lesser degree, CaO), as well as textural particularities in thin section. In any case, their provenance in *Sullecthum* is unquestionable, where some textural variability in the fabrics has been reported by Capelli *et al*. (2006).

A number of petrographic loners within the ‘quartz and calcite fabric group’ (Fig. 7) have also shown a particular chemical composition in each case, some of them being included in the chemical groups CG3 or CG4 (but with no clear relation to other samples in these groups) while others behaving as chemical loners in the statystical analysis (Table 5; Fig. 2). For many of them, the comparison with reference groups from Tunisian workshops does not allow a specific provenance hypothesis. However, on the basis of fabric characteristics, the sample ILU022 can be related to a provenance in the Moknine workshop, in central-eastern Tunisia (Bonifay 2004; Gandolfi *et al*. 2010; Capelli and Bonifay 2014; Fantuzzi *et al*. 2015a, 2015b), while the fabric in ILU074 is quite similar to the fabrics from the zone A in Nabeul (Bonifay *et al*. 2010; Capelli and Bonifay 2014); in these two samples, the high CaO percentages (Table 2) can be associated with the abundant calcareous inclusions (limestone and microfossils) observed in thin section. The fabric in sample ILU067, an Africana IID amphora, is similar to the fabrics from *Leptiminus* (see Sherriff *et al*. 2002; Bonifay 2004; Capelli 2005a; Capelli and Bonifay 2014), which might be consistent with the typological evidence from this production centre (*e.g.* Stirling and Ben Lazreg 2001: fig. 3.13 nº 7; Bonifay 2004: fig. 16 n. 6). On the other hand, two samples that were classified initially as Tripolitana III in the archaeological analysis (ILU009, 069) show fabrics that suggest a provenance in Tunisia rather than in Tripolitania (see for example Peacock 1984; Bonifay 2004;

Bonifay *et al*. 2010; Capelli and Bonifay 2014); from this evidence, the rim ILU009 could be related to type Keay 25.1 rather than to Tripolitana III (Table 5).

Of the few samples that are not included in the 'quartz and calcite fabric group', only ILU059 and ILU060 (Keay 1B amphorae) may be related to an African provenance (Table 5; Fig. 7). They share a similar chemical composition (CG6) and petrographic fabric (PG-2). This latter is similar to the fabric usually found in samples of the same amphora type (Peacock and Williams 1986: 172; Capelli and Bonifay 2007; Lemaître *et al.* 2011) and for which a probable provenance in Algeria has been proposed, though not excluding other possibilities (see Capelli and Bonifay 2007). For the moment, the scarcity of published archaeometric and archaeological information on Mauretanian or Numidian amphora workshops does not allow further conclusions.

Four samples, on the other hand, can be associated with a non-African provenance according to their petrographic characteristics (PG-3 to PG-5). Based on this evidence, the initial typological classification for these samples must be revised (Table 5). They all represent chemical and petrographic loners in this study:

- ILU066: its chemical and petrographic composition (PG-5) can be associated with amphorae from Eivissa (see for comparison Buxeda and Cau 1997, 2004; Buxeda *et al.* 1998, 2005); on this basis, and bearing in mind the presence of many residual materials in the Late Antique context where it was found, this rim (Fig. 7) may be reinterpreted as a PE-25 amphora.

- ILU005: the fabric (PG-4) suggests a southern Hispanic provenance, most probably in the coast of *Baetica* between Granada and Málaga, near some outcrops of the Alpujárride, Maláguide and/or Nevado-Filábride complexes (IGME 1980; Junta de Andalucía 1998). This amphora rim (Fig. 7) can be reinterpreted as a Beltrán IIB amphora (residual in the archaeological context).

- ILU046: the fabric (PG-3.1) and chemical composition —with high percentages of MgO (4.3%) and $K_2O$ (5.7%)— are quite similar to some products from the northeastern Iberian Peninsula (eastern *Tarraconensis*) with an earlier chronology (see Martínez 2014), this being the most probable provenance hypothesis.

- ILU061: the general characteristics of the fabric (PG-3.2) might be compatible with different zones from the eastern *Tarraconensis* or the *Baetican* area (*e.g.* Guadalquivir/Genil valleys), not excluding other possibilities.

For ILU046 and ILU061, the initial typology (Table 1) proposed from the archaeological study (Cela and Revilla 2004) must be revised; for the moment, we consider them as indeterminate rims (Table 5), since it is not possible to find a clear relation to any known amphora type so far (from the Late Antique period or earlier).

## CONCLUSIONS

The analysis of 57 Late Roman amphorae that had been related to an African provenance in the initial archaeological study (Cerdà *et al*. 1997; Cela and Revilla 2004) reveals, on one hand, a clearly more diversified and complex reality for these amphorae, with a wide diversity of chemical-petrographic compositions. On the other hand, it indicates a non-African provenance for a few samples, suggesting the

need for extreme caution in interpreting the provenance region for an amphora fabric or form on a macroscopic basis, especially when working with highly fragmented materials.

The vast majority of the samples in this study were included in the 'quartz and calcite fabric group' (PG-1), that corresponds, in a broad sense, to Tunisian products. This comprises a variety of fabrics, some of them being clearly better represented than others in the analysed assemblage. Many of these amphorae, related to various types over a large time span (3rd/4th centuries to 6th century), may be associated with a provenance in Nabeul (northeastern Tunisia), especially in the zone B (Sidi Zahruni and possibly other workshops). Other analysed amphorae, dated to the 4th-5th centuries, can be related to a probable provenance in northwestern Tunisia, not excluding a possible Algerian origin in some cases.

Apart from these northern Tunisian (*Zeugitana*) imports, other fabrics observed in this study must be associated with a provenance in central Tunisia (*Byzacena*). Few of them correspond to amphorae from the 3rd or early 4th century, coming from *Sullecthum* and, probably, *Leptiminus*. Other central Tunisian imports, however, are related to sixth-century amphorae, in particular to type Keay 62, with a provenance in the workshops of Henchir ech Chekaf and, more rarely, Moknine. For another fabric related to some Keay 62A amphorae it is not possible, for the moment, to determine a specific provenance, since it shows similarities with products from both the Sahel and the Nabeul areas.

The large number of fabrics and loners within the 'quartz and calcite fabric group' offers an overview of the diversity of Tunisian transport amphorae that were arriving to *Iluro* in Late Antiquity. The analysis reveals that similar amphora types could have been produced in more than one production centre (*e.g.* Keay 62, Keay 55, Keay 25.1, Keay 25.2, Africana IIA). For some of the fabrics, a precise provenance attribution has been possible through comparison with available reference materials from production centres, while for others only a general African provenance (Tunisian mainly) can be proposed. The analysis has shown a strong chemical similarity between a typical fabric from Henchir ech Chekaf workshop (PG-1.1) and other fabrics of uncertain provenance (PG-1.3, PG-1.4, a few samples similar to PG-1.2, and four petrographic loners), including some that are actually more similar (in thin section and to the naked eye) to fabrics from the Nabeul area; they all form a same chemical subgroup (CG4.2) with a low total variation (*vt*) value that is usually indicative of a monogenic population. A better understanding of these chemical similarities between such a variety of fabrics is an aspect that requires further investigation in the future, if possible incorporating new archaeometric evidence from the production centres.

A more or less similar diversity of Tunisian fabrics has been identified recently in other Late Antique consumption centres in the northeastern Iberian Peninsula, such as *Tarraco* (Fantuzzi *et al*. 2015a) and *Emporiae* (Fantuzzi *et al*. 2015b). Many of the Tunisian fabrics found in these urban centres can also be observed in the assemblage from *Iluro* analysed in this paper, indicating that the trade networks enabled the arrival of similar amphora products to all these centres. In the case of *Iluro*, this was a secondary centre where no port has been documented so far (Cerdà *et al.* 1997; Bonamusa 2007), so the supplying with imported products could have taken place through a secondary network, probably connected to the port of *Barcino* (Cela and Revilla 2004). This restriction did not preclude, anyway, the arrival of highly diversified products to *Iluro* in Late Antiquity, as has been suggested by the

archaeological evidence (*e.g.* Cerdà *et al.* 1997; Cela and Revilla 2004; Revilla 2011) and, in this study, by the archaeometric evidence concerning the variability of transport amphorae imported from *Africa*.

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