POTTERY GRAVE GOODS FROM FUNERARY CONTEXTS AT THE ARGARIC SITE OF PEÑALOSA (JAÉN). A METHODOLOGICAL APPROACH

Abstract: The need for interdisciplinary studies is the basis of ambitious research (ARCHEM Project) that is carried out in the argaric settlement of Peñalosa (Baños de la Encina, Jaén), combining organic residues analysis and techno-typological studies of pottery found in funerary contexts. Manufacture and use of pottery could inform us about customs and traditions that remain hidden in time and in the archaeological record. Knowing the implications and decisions of potters as well as the functionality of those vessels deposited inside the graves can approach the idiosyncrasy of a society in the Bronze Age in the southeast of the Iberian Peninsula. The methodology used to identify patterns of functionality is highlighted by the combination of cutting-edge analysis techniques in both fields such as the application of different chromatographic techniques (GC-MS, UPLC-HRMS and GC-C-IRMS) that allow to identify the organic compounds in the ceramics and the application of analytical techniques from Earth Sciences (Stereomicroscopic, X-Ray Diffraction and Petrography), which allow us to characterize ceramic pastes and knowing the catchment of raw materials. This study highlights the Peñalosa site as a melting pot of new research and it brings us closer with the use of a complex methodology combined to the societies 4000 years ago.
Keywords: *Organic residues, Ceramic technology, Funerary context, Argar culture, Bronze Age.*

Alejandra GARCÍA-GARCÍA
University of Granada, Faculty of Philosophy and Letters, Department of Prehistory and Archaeology.
alejandragarcia@ugr.es

Laura VICO-TRIGUERO

Eva ALARCÓN-GARCÍA

Juan Jesús PADILLA-FERNÁNDEZ

Adrián MORA-GONZÁLEZ

Auxilio MORENO-ONORATO

Francisco CONTRERAS-CORTÉS

Eloísa MANZANO-MORENO
University of Granada, Faculty of Sciences, Department Analytical Chemistry

Antonio Samuel CANTARERO-MALAGÓN

Francisco MARTÍN-PEINADO
University of Granada. Faculty of Sciences, Department of Edaphology and Agricultural Chemistry

DOI: 10.14795/j.v7i3.536
ISSN 2360 – 266X
ISSN–L 2360 – 266X

1. INTRODUCTION

Pottery has always been considered as products/objects for use[1] that are endowed with an enormous capacity to explain and interpret past societies[2]. Pottery vessels are products of human technology and, therefore, transmitters of the knowledge, thoughts, traditions, innovations and social and cultural relations of the societies that produced them[3]. They were designed as objects with multiple uses and functions[4] and therefore contain cultural, political, ideological and economic codes[5] that should be analysed and not taken for granted.

Attempting to approach the functionality and use of pottery has been one of the constant challenges faced by archaeology and archaeometry. It

[1] COLOMER 2005.
[2] GARCÍA-ROSSELLÓ 2008.
[3] STARK *et alii* 2000.
[4] COLOMER 2005.
[5] DOBRES 2000.

has traditionally been conceived of as something that was omnipresent but independent of the archaeological record. Its study has been segmented and fragmented and rarely have comprehensive studies been undertaken that cover the entirety of the manufacturing processes, techniques and functions from an archaeometric point of view[6].

We can learn about the lives of a group of people by looking at their day-to-day existence and understanding the relations they established and materialised in certain spaces. The specific remains of their material culture are excellent sources of information in this respect. To recognise the functions and uses of pottery vessels, this study proposes a methodological combination of technological studies and organic residue analyses. The techniques used are stereomicroscopy and X-Ray Diffraction (XRD), Gas Chromatography-Mass Spectrometry (GC-MS), Ultra-Performance Liquid Chromatography-High Resolution Mass Spectrometry (UPLC-HRMS) and Gas Chromatography-Combustion-Isotope Ratio Mass Spectrometry (GC-C-IRMS).

Our archaeological record will be the Bronze Age settlement of Peñalosa (Baños de la Encina, Jaén)[7]. The archaeological methodology used to reveal the archaeological record –microspatial excavation together with a very high degree of conservation of contexts and material remains– makes this an ideal archaeological site for this study. Our sample consists of nine vessels that were excavated as grave goods or actual funerary structures (*pithoi*). It is a highly significant sample given that in the Argaric culture we see a series of relations that were acted out in the domestic spaces through funerary rituals. For example, in Argaric settlements we find that life and death share the same sphere, the domestic space. The tombs are found below the habitat floors or simply disguised between domestic structures. Pottery always played an important role in this ritual and the Argaric culture stands out in the general, national and international bibliography for its particular funerary practices and its standardised pottery productions[8]. Many questions have been raised regarding homogeneity and standardisation in the Argaric funerary world and these need to be discussed[9].

The objectives of this study are, therefore, (1) to define the functionality and use of the pottery vessels from different methodological perspectives; (2) to determine the possible dietary patterns of these Argaric populations based on the residues that remain trapped in the pottery matrixes; and (3) to establish a correlation (if any) between the typology of the vessels, the technological processes they were subjected to and the organic compounds identified.

2. ARCHAEOLOGICAL CONTEXT OF PEÑALOSA

Peñalosa is a Bronze Age settlement in the heart of the eastern Sierra Morena Mountains in the municipality of Baños de la Encina (Jaén, Spain) (Fig. 1).

Built on a plateaued hill, its economy was based mainly on agriculture, stockbreeding and, above all, mining and metallurgy. Its pottery in both domestic and funerary contexts followed standardised models. Finally, its funerary ritual consisted of burials inside the domestic spaces. It is these characteristics that allow us to attribute the site to the Argaric culture, which is defined chronologically between 2200 and 1500 BC and geographically in the southeastern Iberian Peninsula.

The archaeological record of this Bronze Age settlement has now been systematically studied for more than 30 years[10]. This means that we currently have exhaustive knowledge of the settlement's internal structure and the areas in which the different maintenance activities were carried out in both the domestic and funerary contexts. Peñalosa has three artificial terraces and an upper area known as the acropolis. There are currently 16 structurally defined dwellings in the whole settlement. For this study, we selected the pottery grave goods from the tombs in Dwellings III and IV situated on the settlement's lower terrace; their excellent conservation allowed the combined application of technological and organic residue analyses.

3. MATERIALS AND METHODS

3.1. Archaeological samples

Nine pottery vessels were studied. They all came from grave goods associated with four tombs discovered in two dwellings on the lower terrace. Their spatial distribution was as follows (Fig. 2):

1. Dwelling III. Two funerary structures corresponding to Tombs 9 and 15. The grave goods in Tomb 9 contained a parabolic bowl (20128), a semispherical bowl (20130) and a globular pot (20129), while those from Tomb 15 consisted of two bowls, one of which was parabolic (20367) and the other carinated (20369). They were analysed along with the funerary structure itself, which was a flat pot (*pithoi*) (20149).
2. Dwelling IV. Two funerary structures belonging to Tombs 6 and 16. In the case of Tomb 6, the studied pottery grave goods consisted of a globular pot (14584) and a parabolic cup (14601); from Tomb 16 an ovoid pot was analysed (14546).

In total, we have four pottery vessels that can be typologically associated with food processing or cooking and five with consumption practices (Fig. 3). They were all found in funerary contexts associated with a specific space in the settlement of Peñalosa –the lower terrace– and attributed to the same chrono-cultural period.

3.2. Technological analysis

The technological analysis consisted of characterising the surfaces and pastes of the funerary ware from Peñalosa.

[6] ADMIRAAL *et alii* 2020.

[7] CONTRERAS *et alii* 1992; 1995; 2000; CONTRERAS/CÁMARA 2002; ALARCÓN 2010; MANZANO *et alii*. 2015; 2016.

[8] SIRET/SIRET 1890; CUADRADO 1947; CONTRERAS 1986; CONTRERAS *et alii* 87-88; ARANDA/ESQUIVEL 2006; ARANDA *et alii* 2015.

[9] LULL 1983; ARANDA 2004; 2010; MOLINA 2015:362.

[10] ARNANZ 1991; MORALES/SANZ 1994; PEÑA 1995; 1999; RODRÍGUEZ/CONTRERAS 1991; CEREIJO 1993; MORENO *et alii* 1995; LIZCANO 1995; LIZCANO *et alii* 1996; MORALES 1996; CÁMARA *et alii* 1996; CÁMARA 1998; CONTRERAS *et alii* 1992; 1995; 2000; CONTRERAS/CÁMARA 2002; SÁNCHEZ/MORENO 2003; 2005; GARCÍA-SOLANO 2004; SÁNCHEZ 2004; JARAMILLO 2005; ALARCÓN 2010; ARBOLEDAS/CONTRERAS 2010; ARBOLEDAS *et alii* 2012; SÁNCHEZ/ALARCÓN 2012; MORA 2017; GARCÍA-GARCÍA 2018

The methodology involved various levels. The first was a macroscopic examination of the vessel surfaces with the aim of defining the treatments that had been applied to them, as well as the type of modelling and firing used in their manufacture. These could be seen thanks to the surface marks and colourations that had remained impressed as they were being made[11]. The second was an analysis with a binocular microscope. This analysis had a double function: to characterize the technological evidence and create technological groups (TG). TG are groups of ceramic samples which have common physical features identified by stereomicroscopy[12], and this facilitates the subsequent representative sampling of each group with regard to other analytical techniques. Finally, it has been apply X-ray diffraction to define the mineralogical composition of the pottery pastes[13]. These techniques allow certain aspects of the pottery production sequence to be reconstructed: the provenance of the raw material and its alteration, the intensity of the kneading and the type of modelling, and the drying and firing phase[14]. These help us establish the degree of pottery-making expertise in this cultural group.

3.2.1 Analytical techniques

We used the following instruments: a Leica L80 stereo microscope with up to 7.5X-60x magnification, a coupled Leica EC3 camera, and a Leica Achro 0.5x lens. The images were captured using the Leica Application Suite software. This analysis made it possible to characterise the pottery pastes and to form technological groups (TG) that allowed technical differences to be established between the vessels. The pastes were described according to the reference tables of other studies[15].

For the XRD analysis, the samples were reduced to a powder (10 microns), the size needed for their laboratory analysis[16]. All the samples were analysed in a BRUKER D8 ADVANCE diffractometer with Cu radiation (sealed tube) and a LINXEYE detector. The measurement parameters were 2s per scan with a magnification of 0.0393766, a limit of 2 theta at the start and 3 at the stop in 70.0108 at a power of 40 Kw and 40 mA. The data was obtained using the DIFRAL software plus XRD Commander. The peaks of the resulting diffractograms were read using the XPowder 12 software Version 2014.04.37. The readings identified the diverse crystalline phases of the minerals that made up the pottery paste with the assistance of the Difdata database, comparing the results to those provided by the RRUFF Project database, which includes a semiquantitative characterisation of the results. In those cases the RIR (Reference Intensity Ratios) method[17] was applied to identify the mineral phases of the samples.

[11] SCHIFFER *et alii* 1994; GARCÍA-ROSELLÓ/CALVO 2006, 2013; FERNÁNDEZ *et alii* 2015; LONGO/NASTRI 2017; GÁMIZ *et alii* 2013; GÁMIZ 2018; VICO *et alii* 2018.

[12] GAMIZ *et alii*. 2013; GAMIZ 2018:314; DRUC/CHAVEZ 2014.

[13] HOLAKOOEI *et alii* 2014.

[14] DRUC/CHAVEZ 2014; VECSTAUDŽA *et alii* 2013; ALBERO 2014; GÁMIZ *et alii* 2013; GÁMIZ 2018; VICO *et alii* 2018.

[15] CASTRO 1989; GÁMIZ *et alii* 2013.

[16] NAVARRO 2008.

[17] CHUNG 1974; MARTÍN 2004.

3.3. Organic residue analysis

3.3.1. Sampling

The sample taken from each of the nine ceramics studied were analysed in accordance with the Dunne Good Practice Guide[18]. The sample pottery powder was scraped from the interior surface of the vessels with a diamond-tipped electric drill. It was then blended in an agate mortar and conserved at -4 °C until the analysis was carried out. Approximately one gram of the sample was used for each test (Table 1).

Table 1: Weights of the samples analysed.

Sample	mg
14546	0.983
14584	1.077
14601	0.927
20128	1.021
20129	1.056
20130	0.986
20149	1.028
20367	1.028
20369	1.040

3.3.2. Chemicals and Reagents

Dichloromethane and methanol (Analytical Grade) purchased from Fluka (St. Louis, MO, USA) were used as extraction solvents. Hexane (Analytical Grade) and m-trifluoromethylphenyl trimethylammonium hydroxide were respectively selected as the solvent and the reagent for the derivatization process prior to gas chromatography analysis. Both were purchased from Sigma-Aldrich (St. Louis, MO, USA). In order to prevent degradation and obtain high levels of reproducibility, m-trifluoromethylphenyl trimethylammonium hydroxide was stored at -4°C in the freezer. Furthermore, LC-MS grade Formic Acid and Methanol (Sigma- Aldrich) were used in High Resolution Mass Spectrometry assays. Analytical grade standards of fatty acids (C10, C12, C13, C14, C15, C16, C16:1, C18, C18:1, C18:2, C19, C20, C22, C26, C30) and Cinnamic acid, Azelaic acid, Suberic acid, Adipic acid, Tartaric acid, Syringic acid, Cholesterol and β-Sitosterol were purchased from Sigma- Aldrich (St. Louis, MO, USA). Fatty acid C13 was used as an Internal Standard. Individual standard solutions of compounds (1000 mg·mL-1) were prepared in metanol and stored at 20°C. These solutions were prepared fresh monthly. Working standard mixtures were prepared by diluting the individual stock solution in methanol. They were stored at 4°C and prepared fresh weekly. All solutions were stored in dark glass bottles to prevent photodegradation.

3.3.3. Sample treatment

A modified GC–MS extraction procedure[19] was performed using 15 mL dichloromethane: methanol (2:1 v/v) as solvent. For the extraction of lipids, fatty acids and other compounds, the ceramic powder was sonicated twice for 15 min (5133 JP Selecta, Barcelona, Spain) and centrifugated at

[18] DUNNE 2017.

[19] EVERSHED *et alii* 1990.

4000 rpm for 10 min. The two extracted liquids were dried in a nitrogen atmosphere at 60°C. Prior to injection into the chromatograph a derivatization reaction was performed with 500 µL of hexane and 20 µL of 3-trifluoromethylphenyl trimethylammonium hydroxide dissolved in 5% methanol as derivatization reagent. This derivatization is based on a procedure used for characterization of drying oils in paintings that was developed by our research team and successfully tested in previous studies[20]. Prior to the GC–MS analysis, a measured amount of internal standard (C13 n-alkane) was added to each sample. Finally, 2 µL of the derivatized samples were injected into the chromatograph. The UPLC-HRMS extraction procedure was similar to the GC procedure but so as to analyze polar compounds, a mixture of methanol: water (70/30 v/v) with 0.1% of HCl was used as solvent. Finally, 10 µL of the samples was injected into the liquid chromatograph and analysed by HRMS.

3.3.4. Analytical techniques

The organic composition of residues in pottery was obtained by GC–MS, GC-C-IRMS, and UPLC-HRMS.

GC–MS analyses were carried out on an Agilent 6890 N gas chromatograph system (Agilent Technologies, Palo Alto, CA, USA) coupled to an Agilent 5973 N mass spectrometer (Agilent Technologies, Palo Alto, CA, USA). The GC was fitted with an automatic injector (model 7683) and automatic sample tray (model 7683). An HP-5MS capillary column (30m×0.25mm×0.25 µm particle size) was used. Samples (2 µL) were injected using splitless injector at 250 °C. The oven was initially held at 70 °C for 2 min, ramped at 12 °C min–1 to 250 °C, and finally increased to 290 °C at 20 °C min–1 and held for 8 min. The mass spectrometer was operated with an ionization potential of 70 eV and mass spectra were collected by scanning over the range m/z 50–520 uma. Instrumental parameters were established as described previously[21]. Peak assignments were determined on the basis of the analysis of available standard compounds and comparing their mass spectra with those from the Wiley Mass Spectral Library.

A Thermo Delta V Advantage coupled to a Thermo Trace GC Ultra Gas Chromatograph was used for **IRMS** detection (ThermoFisher Scientific, Waltham, MA). A Conflo IV system was the interface and the reactor temperature (Cu–Ni–Pt) was established at 1000 °C. The mass spectrometer source pressure was 1.9×10–06 mbar. The GC was fitted with an HP-1 column (30m×0.25mm ID×0.25 µm). The carrier gas was helium and the GC oven was optimized at 70 °C for 2 min, ramped at 12 °C min–1 to 250 °C, and finally increased to 290 °C at 20 °C min–1 and held for 8 min. Carbon isotope ratios are presented in the standard delta notation relative to the Pee Dee Belemnite (PDB) standard. The results were shown as $\delta^{13}C$ (%)=[(Rsample–Rstandard) / Rstandard], where R is $^{13}C/^{12}C$ in per mil. Accurate and reproducible were calculated using triplicate injections for each sample. CO_2 gas of a known isotopic composition was used as working reference standard. Desviations were estimated for the methyl group added during the methylation process: $\delta^{13}C_{sample}=(29\times\delta^{13}C_{measured}-\delta^{13}C_{meoh})/28$, where $\delta^{13}C_{meoh}$. 1σError, based on repeated analysis of an external fatty acid methyl ester (FAME) standard through the sample runs, was 0.2‰.

UPLC–HRMS analysis was performed on a Waters Acquity UPLC™ HClass system (Waters, Manchester, UK), consisting of an ACQUITY UPLC™ binary solvent manager and an ACQUITY UPLC™ sample manager. An ACQUITY UPLC HSS T3™ column (1.8 µm, 2.1mm×100 mm) (Waters, UK) was used for the separation of compounds. A Synap G2 quadrupole tandem time of flight (QTOF) mass spectrometer (Waters), equipped with an orthogonal Z-spray™ electrospray ionization (ESI) source was established for the analysis of molecular formulae. Chromatographic separation was performed with a gradient mobile phase consisting of 0.5% (v/v) aqueous acetic acid solution (solvent A) and acetonitrile (solvent B). The flow rate was 400 µL min^{-1}, the column was maintained at 40 °C, and the injection volume was 10 µL. Gradient was as follows: initial mobile phase 5% (B), which was linearly increased to 100% (B) within 15.0 min and maintaining for 1.0 min in order to preserve the column using 100% acetonitrile phase. Last, back to 5% in 0.1 min and held for 1.9 min to equilibrate the column. Total run time was 18.0 min. The QTOF mass spectrometer was established with ESI in positive and negative ion mode. The QTOF parameters were optimized so as to fulfill the required accuracy for the determination of mass molecular formulae. Regarding mass spectrometer parameters, were established as: capillary voltage, 2.8 kV; cone voltage, 25.0 V; source temperature, 100 °C; desolvation temperature, 500 °C; cone gas flow, 40 Lh^{-1}; desolvation gas flow, 800 Lh^{-1}. About cone and desolvation gas, Nitrogen with 99.995% of purity was selected. The working mass range was performed between 50.0 uma and 1200.0 uma in positive and negative mode.

4. RESULTS

4.1. Technological analyses

4.1.1. Macroscopic and stereomicroscopic analyses

The examination of the surfaces and pottery pastes yielded information on the technology used for the manufacture of the different vessels analysed.

On a macroscopic level, we observed the application of an optimum surface treatment (polishing) that resulted in uniformly smooth surfaces. The characteristic polishing of these pottery productions was applied once the clay had become leather-like. Only a flat pot with slightly incurving walls (20149) had been spatulated on both the interior and exterior surfaces. On a microscopic level, the pressure exerted during the polishing can be observed through the complete insertion of the temper into the matrix, as well as through the homogeneity of the surface layers, in contrast to the result with those techniques that exercise less pressure on the (spatulated) surface[22] (Fig. 4).

In terms of the type of modelling, due to the homogeneity of the surfaces as a result of the treatments applied, we were only able to determine the modelling

[20] MANZANO *et alii* 2011; 2015; 2016; 2019.

[21] MANZANO *et alii* 2015.

[22] ALBERO 2014, IONESCU *et alii* 2019.

Table 2: Technological groups and characteristics of funerary pottery from Peñalosa.

Sample	Shape	Compacity	Temper size	Temper (%)	Technological group
20129	Cooking pot	Compact	Medium	30%	1
20149	Cooking pot	Compact	Medium	30%	1
14546	Cooking pot	Compact	Medium	40%	1
14584	Cooking pot	Compact	Medium	40%	1
20128	Bowl	Compact	Small	40%	2
20130	Bowl	Compact	Small	30%	2
20367	Bowl	Compact	Small	40%	2
20369	Bowl	Compact	Small	30%	2
14601	Chalice	Compact	Small	30%	2

through coil pottery, consisting of the superposition of layers of clay[23], in the case of bowls, as these did not receive such an intense treatment as other forms. This allowed us to observe clearly the features related to that type of manufacture, which are particularly evident on the surfaces.

On the other hand, stereomicroscope analysis allowed us to establish two technological groups (Table 2) whose samples are grouped according to their similarity in the compactness of the paste and the size of the grains.

A common characteristic among all the vessels analysed is the compact pastes resulting from the insistent kneading of the clay, leading to a low presence of striations and pores[24]. Good compacting would have produced more resistant pastes due to the elimination of excess water, thus avoiding possible fractures during the drying, firing and subsequent use[25]. This aspect is also related to a homogenous distribution of the temper, the consequence of an insistent and prolonged kneading of the clay prior to its modelling.

All the samples contained a large amount of temper (30-40%) of variable sizes ranging from small to medium with angular shapes suggesting that they had been added. In all the matrixes we observed a predominant mineral of larger dimensions than the rest, which was identified by X-ray diffraction as quartz (Table 3).

On the other hand, as a common characteristic among the vessels studied, we deduced that the drying time of the pieces had been correct as the density of pores and striations can be considered as low (< 10%) and the absence of fractures indicates that the dehydration process was carried out slowly. Temper contention allows an optimum drying phase and provides greater resistance to firing and exposure to flame during the functional life of the piece[26].

Table 3: Semiquantitative percentages of X-ray diffraction analyses of funerary goods from Peñalosa.

Sample	Qz	Mc	Ab	Ilt-Ms	Amorphous
12130	85.8	0	6	5.4	2.8
14546	75	4.4	13.1	4.4	3.1
14584	82.8	2.8	8.8	2.6	2.9
14601	87.7	3.3	4.4	1.8	2.7
20128	92.2	0	2.9	2.9	2
20129	83.7	1.4	10.1	2.6	2.3
20149	76.3	6	13.3	3.6	1.9
20367	84.7	2.6	8.2	1.9	2.6
20369	84.6	4.5	5.4	3	2.5

The majority of the vessels (14546, 20149, 20128, 20129, 20130, 14584 and 20367) were fired in a reduced atmosphere. This can be seen from the dark colourations (black and brown) in their matrixes. However, three cases have pastes with colourations that attest mixed atmospheres (14601, 20367 and 20149), alternating beige and dark tones. This heterogeneity in the colourations is due to the use of combustion structures known as earth kilns or *horneras* that allow no control over the intake and exit of air, a type of kiln that has been documented in the prehistory of the Iberian Peninsula[27]. In them, those vessels that were closer to the heat focus would have received a dark colouring as a result of a reduction in oxygen, while those that were farther away from the heat would have been exposed to the intake and throughput of oxygen, which would have caused the twin colouration. This lack of control in the firing is also observed in the colour of the surfaces, where all the vessels present blotches of different tonalities.

4.1.2. X-ray diffraction

The sample analysed with XRD gave very homogenous results (Table 3). In all the cases we documented the remains of quartz as the main mineral and secondary plagioclases (albite) and phyllosilicates (illite-white mica). Also identified in the majority of the samples (77.77%) were alkaline feldspars (microcline), except in two cases (12130 and 20128), an absence that could be explained by the raw material being extracted from a different area to the rest of the samples. However, this type of mineralogy can be found in other granitic rocks or rocks of volcanic origin near Peñalosa (5 km), meaning that all the raw material came from the local area.

These results confirm the fact that the funerary ware from Peñalosa was fired at temperatures of between 500 °C and 800 °C. The absence of clay minerals, such as chlorite, which tends to disappear at temperatures above 500 °C[28], as well as the presence in all the samples of peaks of thermally modified phyllosilicates, indicates that the vessels were

[23] HERAS 1992; ROSELLÓ/CALVO 2013.
[24] DRUC/CHAVEZ 2014.
[25] SCHIFFER/SKIBO 1987.
[26] WEST 1992; SCHIFFER *et alii* 1994.
[27] GARCÍA-ROSELLÓ/CALVO 2006.
[28] LINARES *et alii* 1983; PISKAREVA *et alii* 2019.

Table 4: Peak assignment to compounds identified (% assignment greater than 90%), retention time (tR) and *m/z* selected.

	tR (min.)	m/z	Compound
1	**7.754**	172	Nonanoic acid
2	**7.966**	174	Hexanedioic acid, dimethyl ester
3	**9.435**	172	Decanoic acid
4	**9.496**	186	Nonanoic acid, 4-oxo
5	**9.602**	216	Propanoic acid, 2-methyl-, 3-hydroxi-2,4,4-trimethylpenthyl ester
6	**10.435**	194	1,2-Benzenedicarboxilic acid, dimethyl ester
7	**10.995**	226	Phenol, 2,4-bis(1,1-dimethylethyl)
8	**11.374**	216	Nonanedioic acid, dimethyl ester
9	**11.873**	222	1,2-Benzenedicarboxilic acid, diethyl ester
10	**11.919**	226	Hexadecane
11	**12.464**	226	Dihydro methyl jasmonate
12	**12.721**	258	Hexanedioic acid, bis(2-methylpropyl) ester
13	**13.115**	242	Tetradecanoic acid
14	**13.766**	256	Tetradecanoic acid, 12-methyl
15	**13.812**	254	Octadecane
16	**13.857**	282	Hexadecane, 2,6,10,14-tetramethyl
17	**14.024**	256	Pentadecanoic acid
18	**14.387**	278	1,2-Benzenedicarboxilic acid, bis(2-methylpropyl) ester
19	**14.690**	268	Nonadecane
20	**14.902**	270	Hexadecanoic acid
21	**14.993**	292	Methyl-3-(3,5-ditertbutil-4-hydroxiphenyl) propionate
22	**15.296**	278	1,2-Benzenedicarboxilic acid, dibutyl ester
23	**15.417**	284	Hexadecanoic acid, 14-methyl
24	**15.478**	284	Hexadecanoic acid, 15-methyl
25	**15.523**	282	Eicosane
26	**15.720**	284	Heptadecanoic acid
27	**16.023**	288	14-beta-h-pregna
28	**16.311**	296	9-Octadecenoic acid
29	**16.326**	296	Heneicosane
30	**16.538**	298	Octadecanoic acid
31	**16.720**	342	1-Propene-1,2,3-tricarboxilic acid, tributyl ester
32	**17.083**	310	Docosane
33	**17.250**	312	Nonadecanoic acid
34	**17.931**	326	Eicosanoic acid
35	**18.204**	324	Tricosane
36	**18.355**	338	Tetracosane
37	**18.507**	340	Heneicosanoic acid
38	**18.900**	352	Pentacosane
39	**19.052**	354	Docosanoic acid
40	**19.082**	390	1,2-Benzenedicarboxilic acid, bis(2-etilhexyl) ester
41	**19.431**	328	Dehydroabietic acid
42	**19.582**	368	Tricosanoic acid
43	**19.976**	380	Heptacosane
44	**20.173**	382	Tetracosanoic acid
45	**21.582**	410	Hexacosanoic acid
46	**23.567**	438	Octacosanoic acid
47	**24.734**	382	Cholesta-3,5-dien-7-one

14546: 7; 10; 13; 16; 17; 19;20;21;26;28;30;44;45;46.
14584: 8;13;15;16;18;19;20;25;29;30;32;34;36;38;40;43;44;45;47.
14601: 2;4;6;8;9;11;17;18;19;20;25;26;27;29;30;32;34;35;36;39;42;43;44;45.
20128: 3;5;12;18;19;20;25;27;29;30;32;36;40;41.
20129: 7;10;13;16;19;20;25;26;27;29;30;32;34;36;39;41;42;44.
20130: 7;13;15;17;18;20;25;29;30;31;32;36;39;42;43;44;45.
20149: 1;7;10;13;14;17;20;23;24;26;28;30;33;34;37;40; 41;42;44;45;46.
20367: 13;17;20;21;22;26;28;30;31;32;38;40.
20369: 9;13;15;18;19;20;21;22;25;27;29;30;32;36;40.

heated to more than 500 ºC. However, there are various factors that indicate that the temperatures did not exceed 800 ºC: the absence of neoformed phases related to high temperatures, the considerable presence of illite-muscovite whose dehydroxylation does not begin at temperatures lower than 850-900 ºC[29] and the low percentages of amorphous material in the samples, resulting from the destruction of material by high firing temperatures.

4.2. Organic residue analysis

All the samples analysed in this study are attributed to grave goods. The lipidic residues were extracted using the methodological protocol described above in "sample treatment" and were analysed using chromatographic techniques such as GC-MS, UPCL-HRMS and GC-C-IRMS. The combination of the results of all the analytical techniques used in this study was basically to provide us with a more accurate definition of the raw materials originally contained in those pottery vessels. In chromatograms of analysed samples we observed the presence of phthalates associated with plastics, such as 1, 2-Benzenedicarboxylic acid. These compounds were interpreted as contamination and did not influence the final discussion of the results. Figure 5 shows the chromatogram with compounds identified in the selected vessel 14546.

Palmitic acid (C16:0) and stearic acid (C18:0) were the predominant acids in all the samples. This was to be expected as they are fatty acids normally found in abundance in the lipidic extracts of archaeological pottery. Vegetable fats were found in seven of the pottery vessels studied (77.77% of the samples analyzed) (20149, 20367, 20128, 20129, 14546, 14584, 14601). Their presence was due to the identification of compounds in the chromatograms above 90% assignment (an attribution verified by the NIST Mass Spectral Library), such as short-chain fatty acids (C9:0), unsaturated fatty acids (C18:1), dicarboxylic acids (2C6), long-chain fatty acids (C20:0, C21:0, C22:0, C23:0, C24:0, C26:0, C28:0) and saturated hydrocarbons (C16H34, C18H38, C19H40, C20H42, C21H44, C22H46, C23H48, C24H50, C25H52, C27H56)[30]. Animal fats were clearly identified in three of the vessels (33.33% of the samples analyzed) (20149, 20128, 14584). Their presence can be explained by the identification of compounds such as short-chain fatty acids (C10:0), odd-chain fatty acids (C15:0, C17:0, C19:0), branched-chain fatty acids (C14:0br, C16:0br) and the presence of cholesterol and/or its derivatives (cholesta-3.5-dien-7-one). These compounds are associated in literature to animal fat[31]. The mixture of both types of fats (vegetable and animal) was identified in three of the nine pottery vessels studied (20149, 20128, 14584. Residues that cannot be defined specifically were also identified in the chromatogram, for example in vessels 20369 and 20130.

The presence of lignoceric acid (C24:0) and saturated hydrocarbon C27H56 has sometimes been associated with the presence of vegetable waxes and is probably related to beeswax[32]. These residues were identified in five vessels (55.55% of the samples analyzed) (14584, 14601, 20129, 20130, 20149). Dehydroabietic acid was identified in three of the samples studied (20128, 20129, 20149) (33.33%), all from House III. It is a residue linked to conifer resins[33]. Table 4 shows the peak assignment to compounds identified.

Lipid residue analysis is complemented by isotopic analysis from GC-C-IRMS. For this purpose, fatty acids C16:0 and C18:0, which were the most abundant in all the samples, were studied according to their carbon isotopic composition.

The isotopic composition of stearic acid ($\delta^{13}C_{18:0}$) versus the modern values for palmitic acid ($\delta^{13}C_{16:0}$) from European plants and animals[34] will be used as reference values and the comparison with the $\delta^{13}C$ and $\Delta^{13}C$ values of our samples. The isotopic values of the samples from House III and House IV are shown in table 5. In the ruminant fats cluster we find the residues from vessels 20149, 20369, 20128, 20129, 14546 and 14601. Samples 20367, 20130 and 14584 are in the area of the dividing line between the ruminant and non-ruminant clusters (including horse fat) based on the scientific literature[35], which could suggest the mixing of both types of fats in those vessels. There is a discrepancy in this area of the diagram, given that the vegetable fats that are less studied than the animal fats are also located in the area of non-ruminant animals, leading to confusion in the results obtained[36]. The isotopic values of these samples are shown in figure 6.

Table 5: Isotopic values of the samples analysed.

Sample	δ13C/ δ12C	
	δ13C16:0	δ13C18:0
20128	−22.23	−23.50
20129	−22.27	−23.64
20130	−21.30	−21.63
20149	−23.99	−25.12
20367	−24.38	−24.67
20369	−24.31	−25.96
14546	−23.79	−25.03
14584	−20.85	−21.39
14601	−21.34	−22.97

Finally, UPLC-HRMS technique has allowed identified molecular formulas tentatively linked to organic residues in two of the nine samples analysed (20129 and 14584). In globular pot 20129, the formula C19H32O7 (mass: 371.2023 in negative mode $[M-H]^-$) was identified and linked to 7a-Acetoxy-15-methoxy-10-O-methyldeacetyldihydrobotrydial associated with polyphenols of vegetable origin that were associated with fungi and mushrooms such as *Daldinia concentrica*, which has medicinal properties[37] (Fig.7). In pot 14584, the formula

[29] PEÑA-POZA 2011.
[30] COPLEY *et alii* 2005.
[31] DUDD/EVERSHED 1998; BABOT/APELLA 2003; SPANGENBERG *et alii* 2006; BAETEN *et alii* 2013; SALQUE *et alii* 2013.
[32] HERON *et alii* 1994; MAYYAS *et alii* 2012 a/b.
[33] MILLS/WHITE 1977.
[34] DUDD/EVERSHED 1998; KIMPE *et alii* 2004; SPANGENBERG *et alii* 2006.
[35] MILETO *et alii* 2017.
[36] STEELE *et alii* 2010.
[37] XIANG-DONG *et alii* 2008.

C19H32O7 (mass: 371.2014 in negative mode [M-H]$^-$) was identified as Byzantionoside B that can be linked to vegetable residues found in *Sclerochloa dura* with medicinal and anti-inflammatory properties[38]. Both compounds were identified using a highly accurate program (Mass Fragment) that allow us to analyze the structure using mayor fragments in the obtained mass spectra in negative mode ([M-H]-).

5. DISCUSSION

The results obtained based on the methodology used for the analysis of organic residues and the technological study allows us to make inferences about the manufacture and use of the Peñalosa funerary vessels.

The technological features identified show us a very homogeneous assemblage of pottery vessels, which can also be seen in the typology of their forms, the result of a production executed by the expert hands of people with a good knowledge of pottery making. The intensive surface treatment of the vessels (polishing), as well as certain technological aspects (good compacting of the pastes, the refinement of large inclusions, the marked orientation of the temper, etc.) imply that the makers were clearly focused on producing high-quality objects, both in aesthetic and functional terms. This fact contrasts with the results obtained in other studies that attested the presence of vessels of low technological quality in other Argaric tombs[39].

However, despite the apparent homogeneity of the assemblage studied here, we observe differences in both the type of manufacture and the organic residues absorbed by the pottery matrixes among those vessels used for preparing and consuming foodstuffs.

On a technological level we can determine that the grains contained in the pots was larger than that of the bowls. The sizes of these tempers are related to the purpose the vessels were destined to be used for, as they produced pastes that are more resistant. Thanks to these qualities, the potters obtained objects with greater mechanical, fire and heat resistance, making it less likely that the pastes would crack[40]. Likewise, there are fire exposure marks (soot, scratches and abrasions) on these vessels, which show that they were used for other purposes before burial placement[41] (Fig. 8).

We studied four pots linked to **food preparation**. The globular pot (20129) and the flat pot (20149) were documented in House III, while the globular pot (14584) and the ovoid pot (14546) came from House IV.

The globular pots (20129 and 14584) had polished surfaces and contained fat residues identified by GC-MS and UPLC-HRMS analysis that linked them to vegetable polyphenols. These vegetable fats were associated, in the case of 20129, with fungi and mushrooms that could have medicinal properties[42] and in pot 14584 with a kind of pasture native to Eurasia with anti-inflammatory and medicinal properties[43], that also contained acorn seeds. This is a very interesting fact, as it shows us the considerable knowledge the society had of its environment, although above all it tells us of knowledge acquired about the characteristics and the nutritional and medicinal properties of these raw materials. Animal fats are also present; in the case of 20129 we identified residues associated with ruminant fats and in 14584 compounds located in zone 0, specifically in the cluster corresponding to horse fat (Fig. 6). This is corroborated by the archaeological record of Peñalosa in which horse remains are common and zooarchaeological studies have identified cut marks, implying that horse meat was eaten[44]. Waxy material related to beeswax was also identified in both vessels[45], perhaps residues associated with their content. Conifer resins were found through the identification of dehydroabietic acid in vessel 20129[46]. This suggests two hypotheses. The first is that the resins were used to reinforce the surface treatment (polishing), thus making the vessel more impervious and protecting the content[47]. We could relate this to liquid foodstuffs and their preparation by, for example, boiling and cooking. The second hypothesis is that the resins would have formed part of the content of the pot as an ingredient, as they have traditionally been used for their medicinal and purgative properties[48].

The flat pot (20149) and the ovoid pot (14546) both had a spatulated surface treatment and inside contained fat residues from ruminant animals, as well as vegetable fats and waxes. These were confirmed by the identification of lignoceric acid (C24:0) and the location of the isotopic values in the ruminant cluster that, according to Steele[49], coincides with that of beeswax. The waxy materials would be related to the content as, with exposure to heat sources, the waxes would have made no sense as impermeability for the vessel walls[50]. These residues are complemented in the flat pot with compounds associated with the conifer resins that were linked to their impermeability[51]. This pot would have been excavated in its secondary use as a burial receptacle for an infant individual (*pithoi*). Thus, we deduce that these pieces were used for two different purposes: food preparation and funerary structures.

In relation to **consumption** practices, five vessels were analysed: two parabolic bowls (20128 and 20367), a semispherical bowl (20130) and a carinated bowl (20369), all from House III, and a cup (14601) from House IV.

In these types of vessels we observe smaller-sized grains. We attribute this to their use for food consumption, rather than its processing. This meant they did not have to be heat resistant, so the pastes of the bowls and cups were more refined and temper was added to reduce plasticity during the kneading and modelling phase.

Thus we observed, on the one hand, that the parabolic bowl (20128), the carinated bowl (20369) and the cup (14601) contained ruminant animal fats, with the addition

[38] BUKHARI *et alii* 2016.
[39] CONTRERAS 1986; ARANDA/ESQUIVEL 2006.
[40] SCHIFFER/SKIBO 1987; TITE 2008.
[41] RAFFERTY *et alii* 2015.
[42] XIANG-DONG *et alii* 2008.
[43] BUKHARI *et alii* 2016.
[44] CONTRERAS *et alii* 2000; SANZ/MORALES 2000; GREGG/SLATER 2010; MILETO *et alii* 2017; PÄÄKKÖNEN *et alii* 2018.
[45] HERON *et alii* 1994.
[46] MALAINEY 2011.
[47] SCHIFFER 1990; SILVA 2008; TITE 2008.
[48] CAMARDA *et alii* 2011; TERMENTZI *et alii* 2011; MOLINA 2015.
[49] STEELE 2008.
[50] MOLINA 2015.
[51] MILLS/WHITE, 1977; STARK 1991.

of vegetable fats in the case of the parabolic bowl and the cup[52]. In the semispherical bowl (20130) and the parabolic bowl (20367) the isotopic values place the animal fats on the border between the ruminant and non-ruminant animal fats, perhaps compatible with horse fat[53] as in the previous vessels and vegetable fats in the case of the parabolic bowl.

The presence of waxes and resins in those vessels is not abundant. The waxy materials were only found in two of the vessels, 20130 and 14601, while conifer resins were identified in parabolic bowl 20128. The application of those compounds (waxes and resins) in polished vessels suggests two hypotheses: either that they were used to improve the impermeability of the vessels or that they formed part of their content.

6. CONCLUSIONS

The comparison of the results obtained through different scientific disciplines related to technological studies and the analysis of organic residues provided information about the Argaric funerary rituals.

Based on the combination of these studies, we have been able to define the uses grave goods were put to before they were placed in tombs. The presence of contents and well-defined technological strategies in these vessels reveals their prior use before being deposited in tombs, in contrast to the hypotheses defended in other studies of nuclear zone as well as in its closest hinterland such as the province of Granada with Purullena and Monachil sites[54]. In these settlements pottery are made exclusively to be deposited in the graves, behaviour that seems not to occur in the mining-metallurgical area of Sierra Morena.

This justifies the application of this twin methodology for the study of pottery vessels as the basis of a new line of research that will give us a better understanding of the functionality of pottery grave goods in funerary contexts.

ACKNOWLEDGEMENTS

R & D Project HAR2015-66009-P, ARCHEM Project, funded by the Spanish Ministry of Economy and Competitiveness and Junta de Andalucía (HUM 274 and FQM 338) has supported this research. L. Vico acknowledges support from a PhD scholarship from the Spanish Ministry of Economy, Industry and Competitiviness. E. Alarcón acknowledges support from University of Granada to her Project "Cocinando nuevas perspectivas en Arqueología. Las prácticas alimenticias en la Prehistoria Reciente".

REFERENCES

ADMIRAAL *et alii* 2020
Admiraal, M./Lucquin, A./Drieu, L./Casale, S./Jordan, P.D./Craig, O.E., Leftovers: the presencia of manufacture derived aquatic lipids in alaskan pottery, *Archaeometry* 62 (2), 346-361.

ALARCÓN 2010
Alarcón, E., *Continuidad y cambio social. Las actividades de mantenimiento en el poblado argárico de Peñalosa (Baños de la Encina, Jaén)*, Doctoral Thesis. Universidad de Granada.

ALBERO 2014
Albero, D., *Materiality, techniques and society in pottery prodution* (Berlin: De Gruyter Open).

ALBERO/ARANDA 2014
Albero, D./Aranda, G., Elección tecnológica y expresión social: Análisis arqueométrico de cerámicas funerarias argáricas del Cerro de San Cristóbal (Ogíjares, Granada), *Complutum* 25,109-27.

ARANDA 2004
Aranda, G., Craft specialization in pottery production during the Bronze Age in south eastern Iberia, *Journal of Iberian Archaeology* 6, 157-79.

ARANDA 2010
Aranda, G., Entre la tradición y la innovación: el proceso de especialización en la producción cerámica argárica, *Menga* 1, 77-95.

ARANDA/ESQUIVEL 2006
Aranda, G./Esquivel, J.A., Ritual Funerario y Comensalidad en las Sociedades de la Edad del Bronce del Sureste Peninsular: La Cultura de El Argar, *Trabajos de Prehistoria* 63 (2), 117-133.

ARANDA/MONTÓN/ SÁNCHEZ 2015
Aranda G./Montón S./Sánchez M., *The Archaeology of Bronze Age Iberia. Argaric Societies* (New York: Routledge).

ARBOLEDAS/CONTRERAS 2010
Arboledas, L./Contreras, F., Mina del polígono o Contraminas (Baños de la Encina, Jaén). Evidencias de la explotación de mineral de cobre en la antigüedad. *Cuadernos de Prehistoria y Arqueología de la Universidad de Granada* 20, 355-379.

ARBOLEDAS/ROMÁN/PADILLA 2012
Arboledas, L./Román, J./Padilla, J.J., Peñalosa en época romana. Más allá de un poblado argárico del Alto Guadalquivir (Baños de la Encina, Jaén), *Antiquitas* 24, 133-151.

ARNANZ 1991
Arnanz, A., Materiales carpológicos del yacimiento de Peñalosa (Baños de la Encina, Jaén), *Trabajos de Prehistoria* 48, 405-420.

BABOT/APELLA 2003
Babot, M.P./Apella, M. C., Maize and bone: residues of grinding in northwestern Argentina, *Archaeometry* 45 (1), 121–132.

BAETEN *et alii* 2013
Baeten, J./Jervis, B./De Vos D./Waelkens M., Molecular evidence for the mixing of meat, fish and vegetables in anglo-saxon coarseware from hamwic, UK, *Archaeometry* 55(6), 1150–1174.

BUKHARI *et alii* 2016
Bukhari, S.M./Jullumstrø, A./Maja, H./Isaksen, S.M./Sæther, M./Krane, T.H./Villa, S./Schmid, R./Brunsvik, A./Fuglseth, E./Zlatković, B./Johansen, B./Simić, N., A novel compound isolated from Sclerochloa dura has anti-inflammatory effects, *Journal Serbian Chemical Society* 81 (1), 1-12.

CÁMARA/CONTRERAS/LIZCANO 1996
Cámara, J.A./Contreras, F./Lizcano, R., Enterramientos y diferenciación social II. La problemática del Alto Guadalquivir durante la Edad del Bronce. *Trabajos de Prehistoria* 53 (1), 91-108.

CÁMARA *et alii* 2005
Cámara, J.A./Milá, S./Arana, R./Contreras, F., Estudio arqueométrico de diversos materiales cerámicos procedentes de Peñalosa (Baños de la Encina, Jaén), *Anuario arqueológico de Andalucía* 2002, II, 37-50.

CÁMARA 1998
Cámara, J.A., *Bases teóricas y metodológicas para el estudio del ritual funerario utilizado durante la Prehistoria Reciente en el*

[52] COPLEY *et alii* 2005; MILETO *et alii* 2017
[53] SANZ/MORALES 2000; MILETO *et alii* 2017
[54] CÁMARA *et alii* 2005; ALBERO/ARANDA 2014

sur de la Península Ibérica, Doctoral thesis, Universidad de Granada.

CAMARDA/DI STEFANO/ PITONZO 2011
Camarda, L./Di Stefano, V./Pitonzo, R., Natural resins: Chemical constituents and medicinal uses. Researchgate:https://www.researchgate.net/publication/287289098_Natural_resins_Chemical_constituents_and_medicinal_uses. Accesed 10 Abril 2020.

CASTRO 1989
Castro, A., *Petrografía Básica. Texturas, clasificación y nomenclatura de rocas* (Madrid: Paraninfo).

CEREIJO 1993
Cereijo, M.A., Las rapaces nocturnas como acumuladores potenciales de restos faunísticos en yacimientos arqueológicos: los micromamíferos de Peñalosa, *Archaeofauna* 2, 219-230.

CHUNG 1974
Chung, F., Quantitative interpretation of X-ray diffraction patterns: Matrix flushing method of quantitative multicomponent analisys. *Journal Applied Crystallography* 7, 519-525.

COLOMER 2005
Colomer, L., Cerámica prehistórica y trabajo femenino en el Argar: una aproximación desde el estudio de la tecnología cerámica. In Sánchez Romero M (ed.), *Arqueología y Género*, 177-217.

CONTRERAS 1986
Contreras, F., *Aplicación de métodos y análisis estadísticos a los complejos cerámicos de la Cuesta del Negro (Purullena, Granada),* Doctoral thesis, Universidad de Granada.

CONTRERAS 2000
Contreras, F. (ed.), *Proyecto Peñalosa. Análisis histórico de las comunidades de la Edad del Bronce del Piedemonte meridional de Sierra Morena y Depresión Linares-Bailén* [Arqueología Monografías 10] (Sevilla: Consejería de Cultura).

CONTRERAS/CÁMARA 2002
Contreras, F./Cámara, J.A., Peñalosa, la Edad del Bronce en Baños de la Encina, *ARQUEO, la aventura de la Arqueología* 6, 66-73.

CONTRERAS *et alii* 1987-1988
Contreras, F./Capel, J./De la Torre, F./Molina, F./Esquivel, J.A., Los ajuares cerámicos de la necrópolis argárica de la Cuesta del Negro (Purullena, Granada): Avance al estudio analítico y estadístico, *Cuadernos de Prehistoria y Arqueología de la Universidad de Granada* 12-13, 135-155.

CONTRERAS *et alii* 1992
Contreras, F./Cámara, J.A./Moya, S./Sánchez, R., Primer avance metodológico al estudio de la cultura material del poblado de Peñalosa (Baños de la Encina, Jaén), *Anuario Arqueológico de Andalucía* 1990, II, 281-290.

CONTRERAS *et alii* 1995
Contreras, F./Cámara, J.A./Lizcano, R./Péres, C./Robledo, B./Trancho, G., Enterramientos y diferenciación social I. El registro funerario del yacimiento de la edad del Bronce de Peñalosa (Baños de la Encina, Jaén), *Trabajos de Prehistoria* 52 (1), 87-108.

COPLEY *et alii* 2005
Copley, M.S./Bland, H.A./Rose, P./Horton, M./Evershed, R.P., Gas chromatographic, mass spectrometric and stable carbon isotopic investigations of organic residues of plant oils and animal fats employed as illuminant in archaeological lamps from Egypt, *Analyst* 130, 860-871.

CUADRADO 1947
Cuadrado, E., Útiles y armas de El Argar. Ensayo de tipología, *I Congreso Nacional de Arqueología/V Congreso Arqueológica Del Sudeste, Cartagena*, 103-225.

DOBRES 2000
Dobres, M.A., *Technology and social agency* (London: Blackwell).

DRUC/CHAVEZ 2014
Druc, I.C./Chavez, L., *Pastas cerámicas en lupa digital. Componentes, textura y tecnología* (Wisconsin: Deep Universtiy Press).

DUDD/EVERSHED 1998
Dudd, S.N./Evershed, R.P., Direct demonstration of milk as an Element of Archaeological Economies, *Science* 282, 1478-1481.

DUNNE 2017
Dunne, J., *Organic Residue Analysis and Archaeology Guidance for Good Practice*. Historic England.

EVERSHED/HERON/GOAD 1990
Evershed, R.P./Heron, C./Goad, J., Analysis of organic residues of archaeological origin by high –temperature gas chromatography and gas chromatography- mass spectrometry, *Analyst* 115 (10), 1339- 1342.

FERNÁNDEZ/ BENITEZ DE LUGO/PALOMARES 2015
Fernández, S./Benítez de Lugo, L./Palomares, N., La cerámica del yacimiento arqueológico de Castillejo del Bonete (Terrinches, Ciudad Real). Estudio morfológico y tecnológico, *Complutum* 26 (1), 133-152.

GÁMIZ/DORADO/CABADAS 2013
Gámiz, J./Dorado, A./Cabadas, H., Análisis de cerámica prehistórica con estereomicroscopía: una guía revisada sobre la descripción de las fases de producción, *Cuadernos de Prehistoria de la Universidad de Granada* 23, 365- 385.

GÁMIZ 2018
Gámiz, J., *La cerámica neolítica de Los Castillejos (Montefrío, Granada). Estudio tipológico, decorativo y tecnológico*. Doctoral thesis Universidad de Granada.

GARCÍA-GARCÍA 2018
García-García, A., *La alimentación en la Cultura del Argar. Análisis de residuos orgánicos en las cerámicas de Peñalosa (Baños de la Encina, Jaén).* Doctoral Thesis. Universidad de Granada.

GARCÍA-ROSELLÓ 2008
García-Roselló, J., Reflexiones en torno a las vinculaciones entre la cultura material y los procesos sociales, *Mayurga* 32, 213-250.

GARCÍA-ROSELLÓ/ CALVO 2006
García-Roselló, J./Calvo, M., Análisis de las evidencias macroscópicas de cocción en la cerámica prehistórica una propuesta para su estudio, *Mayurqa* 31, 83-112.

GARCÍA-ROSELLÓ/CALVO 2013
García-Roselló, J./Calvo, M., *Making pots. El modelado de la cerámica y su potencial interpretativo*. [British Archaeological Reports International Series 2540] (Oxford: Archaeopress).

GARCÍA-SOLANO 2004
García-Solano, J.A., *Análisis de los patrones de asentamiento en la cuenca del río Guadiel durante la Edad del Bronce desde la perspectiva del paisaje*. Master thesis. Universidad de Granada.

GREGG/SLATER 2010
Gregg, M.W./Slater, G.F., A new method for extraction, isolation and transesterification of free fatty acids from archaeological pottery, *Archaeometry* 52 (5), 833–854.

HERAS 1992
Heras, C.M., Glosario terminológico para el estudio de las cerámicas arqueológicas, *Revista Española de Antropología Americana* 22, 9-34.

HERON *et alii* 1994
Heron, C./Nemcek, N./Bonfield, K.M./Dixon, D./Ottaway, B.S., The Chemistry of Neolithic Beeswax,

Naturwissenschaften 81(6), 266–269.

HOLAKOOEI *et alii* 2014
Holakooei, P./Tessari, U./Verde, M./Vaccaro, C., A new look at XRD patterns of archaeological ceramic bodies, *Journal of Thermal Analysis and Calorimetry* 118, 165-176.

IONESCU *et alii* 2019
Ionescu, C./Fischer, C./Hoeck, V./Lütge, A., Discrimination of ceramic surface finishing by vertical scanning interferometry, *Archaeometry* 61, 31-42.

JARAMILLO 2005
Jaramillo, A., *Recursos y materias primas en la Edad del Bronce del Alto Guadalquivir, Medioambiente y el Registro Arqueológico en la Cuenca del Rumblar*. Doctoral thesis. Universidad de Granada.

KIMPE *et alii* 2004
Kimpe, K./Drybooms, C./Schrevens, E./Jacobs, P.A./Degeest, R./Waelkens, M., Assessing the relationship between form and use of different kinds of pottery from the archaeological site Sagalassos (southwest Turkey) with lipid analysis, *Journal of Archaeological Sciences* 31 (11), 1503–1510.

LINARES/HUERTAS/CAPEL 1983
Linares, J./Huertas, F./Capel, J., La arcilla como material cerámico: características y comportamiento, *Cuadernos de Prehistoria de la Universidad de Granada* 8, 479-490.

LIZCANO 1995
Lizcano, R., *Las comunidades del Neolítico Final en el Alto Guadalquivir*, Doctoral thesis, Universidad de Granada.

LIZCANO *et alii* 1996
Lizcano, R./Pérez, C./Nocete, F./Cámara, J.A./Contreras, F./Casado O./Moya S., La organización del territorio en el Alto Guadalquivir entre el IV y III milenios (3300-2800 a.C.), *Rubricatum* 1, 305-312.

LONGO/NASTRI 2017
Longo, A./Nastri, J., Análisis exploratorio de la variabilidad de la ceráamcia de superficie en el sitio de El Carmen 2 (valle de Santa María, Tucumán), *Arqueología* 24 (2), 87-108.

LULL 1983
Lull, V., *La Cultura del Argar. Un modelo para el estudio de las formaciones económico-sociales prehistóricas* (Madrid: Akal).

MALAINEY 2011
Malainey, M.E., *A Consumer's Guide to Archaeological Science* (New York: Springer).

MANZANO *et alii* 2011
Manzano, E./Rodríguez, L.R./Navas, N./Checa, R./Romero, L.F./Capitán, L.F., Study of the GC–MS determination of the palmitic-stearic acid ratio for the characterization of drying oil in painting: La Encarnación by Alonso Cano as a case study, *Talanta* 84, 1148–1154.

MANZANO *et alii* 2015
Manzano, E./García, A./Alarcón, E./Cantarero, S./Contreras, F./Vílchez, J., An integrated multianalytical approach to the reconstruction of daily activities at the Bronze Age settlement in Peñalosa (Jaén, Spain), *Microchemical Journal* 122, 127-136.

MANZANO *et alii* 2016
Manzano, E./Cantarero, S./García, A./Adroher, A./Vilchez, J.L., A multi-analytical approach applied to the archaeological residues in Iberian glasses. Earliest evidences on the consumption of fermented beverages in votive rituals. *Microchemical Journal* 129, 286-292.

MANZANO *et alii* 2019
Manzano, E./García, A./Cantarero, S./García D./Morgado, A./Vílchez, J.L., Molecular and isotopic analyses on prehistoric pottery from the Virués-Martínez cave (Granada, Spain), *Journal of Archaeological Sciences: Reports* 27, 101929.

MARTÍN 2004
Martín, J.D., *Using XPowder: A software package for Powder X-Ray diffraction analysis*.

MAYYAS *et alii* 2012a
Mayyas, A.S./Al-Qudah, M.A./Douglas, K.A./Al-Ajlouny, F.K., Beeswax Preserved in Archaeological Ceramics: Function and Use, *Annals of the Faculty of Arts, in Shams University* 40, 343-371.

MAYYAS *et alii* 2012b
Mayyas, A./Stern, B./Gillmore, G./Coningham R./Nashli, H.F., Beeswax preserved in a Late Chalcolithic bevelled-rim bowl from the tehran plain, Iran, *Journal of the British institute of Persian studies* 50, 13–25.

MILETO *et alii* 2017
Mileto, S./Kaiser, E./Rassamakin, Y./Evershed, R.P., New insights into the subsistence economy of the Eneolithic Dereivka culture of the Ukrainian North-Pontic region through lipid residues analysis of pottery vessels, *Journal of Archaeological Sciences: Reports* 13, 67–74.

MILLS/WHITE 1977
Mills, J.S./White, R., Natural Resins of Art and Archaeology Their Sources, Chemistry, and Identification, *Studies in Conservation* 22, 12-31.

MOLINA 2015
Molina, E., *La producción cerámica en el sudeste de la península ibérica durante el III y II milenio ane (2200-1550 cal ane): integración del análisis de residuos orgánicos en la caracterización funcional de los recipientes argáricos*. Doctoral thesis. Barcelona: Universidad Autónoma de Barcelona.

MORA 2017
Mora, A., *Irrigación o secano en el Mediterráneo occidental (III-I Milenio A.N.E.): Un estudio isotópico*. Doctoral thesis. Universidad de Granada.

MORALES/SANZ 1994
Morales, A./Sanz J.L., Arqueo-acarología: potencialidades y limitaciones de una prácticamente inédita subdisciplina arqueozoólogica, *Pyrenae* 25, 17-29.

MORALES 1996
Morales, A., Algunas consideraciones teóricas en torno a la fauna como indicadora de espacios agrarios en la Prehistoria, *Trabajos de Prehistoria* 53 (2), 5-17.

MORENO/MOLINA/CONTRERAS 1995
Moreno, A./Molina, F./Contreras, F., La investigación arqueometalúrgica de la Prehistoria Reciente en el Sureste de la Península Ibérica. In: Vaquerizo D (ed.), *Minería y metalurgia en la España Prerromana y romana*, Córdoba, 13-54.

NAVARRO 2008
Navarro, J.V., Aplicaciones de la Difracción de Rayos X al estudio de los bienes culturales. In: Del Egido M, Prous S (eds.) *La Ciencia y el Arte, Instituto del Patrimonio Histórico Español* (Madrid: Ministerio de Cultura. Gobierno de España), 134-139.

PÄÄKKÖNEN *et alii* 2018
Pääkkönen, M./Bläuer, A./Olsen, B./Evershed, R.P./Asplund, H., Contrasting patterns of prehistoric human diet and subsistence in northernmost Europe, *Scientific Reports* 8, 1148.

PISKAREVA/YANSKAYA/KISELEVA 2019
Piskareva, M.R./Yanskaya, A./Kiseleva, D., Determination of the firing temperature and redox atmosphere of the pottery from the Bronze Age settlement Kamenny Ambar by x-ray diffraction phase analysis, *AIP Conference Proceedings* 2174.

PEÑA 1995
Peña, L., Avance preliminar sobre los restos vegetales del

yacimiento de la Edad del Bronce de Peñalosa (Baños de la Encina, Jaén), *Trabalhos de Antropologia e Etnologia*, 35 (1), 1159-168.

PEÑA 1999
Peña, L., *Prehistoric Agriculture in Southern Spain during the Neolithic and the Bronze Age. The application of ethnographic models* [British Archaeological Reports. International Series 818], Oxford: Archaeopress).

PEÑA-POZA 2011
Peña-Poza, J., Estudio arqueométrico de cerámicas procedentes del yacimiento de Sratosiedle (Gubin, Polonia), *Strat Critic* 5 (3), 82-90.

RAFFERTY/MACCAIN/SMITH 2015
Rafferty, J./MacCain, R./Smith, J., Cooking pots as burial urns, *Midcontinental Journal of Archaeology* 40 (1), 48-72.

RODRÍGUEZ/CONTRERAS 1991
Rodríguez, Mª.O./Contreras, F., *Contrastación antracológica entre dos complejos estructurales del yacimiento del Bronce de Peñalosa (Baños de la Encina, Jaén)*. Arqueología Medioambiental a través de los macrorrestos vegetales, Madrid 1-8 noviembre de 1991.

SALQUE *et alii* 2013
Salque, M./Bogucki, P.I./Pyzel, J./Sobkowiak-Tabaka, I./Grygiel, R./Szmyt, M./Evershed R.P., Earliest evidence for cheese making in the sixth millennium BC in northern Europe, *Nature* 493 (7433), 522-525.

SÁNCHEZ 2004
Sánchez, Romero M., Propuesta para el análisis de género en las sociedades argáricas: Las mujeres en el yacimiento de Peñalosa (Baños de la Encina, Jaén). In: Hernández, L., Hernández, M. (eds.), *La Edad del Bronce en tierras valencianas y zonas limítrofes. Villena* (Villena-Alicante: Ayuntamiento de Villena, Instituto de Estudios "Juan Gil-Albert"), 525-529.

SÁNCHEZ/MORENO 2003
Sánchez, M./Moreno, A., Metallurgical production and women in Bronze Age societies: the Peñalosa site (Baños de la Encina, Jaén). In: *Archaeometallurgy in Europe* (Milán: Associazione Italiana di Metallurgia), 415-422.

SÁNCHEZ/MORENO 2005
Sánchez, M./Moreno, A., Mujeres y producción metalurgia en la prehistoria: el caso de Peñalosa (Baños de la Encina, Jaén). In: Sánchez Romero M. (ed.), *Arqueología y género* (Granada: Universidad de Granada), 261-282.

SÁNCHEZ/ALARCÓN 2012
Sánchez M./Alarcón E., Lo Que los Niños nos cuentan: Individuos infantiles Durante La Edad del Bronce en el sur de la Península Ibérica. In: Justel Vicente, D. (ed.), *Niños en la antigüedad. Estudios sobre la infancia en el Mediterráneo antiguo* (Zaragoza: Prensas de la Universidad de Zaragoza), 57-97.

SANZ/MORALES 2000
Sanz, J. L./Morales, A., Los restos faunísticos de Peñalosa, In: Contreras, C. (ed.), *Proyecto Peñalosa. Análisis histórico de las comunidades de la Edad del Bronce del Piedemonte meridional de Sierra Morena y Depresión Linares- Bailén* [Arqueología Monografías, 10] (Sevilla: Consejería de Cultura), 223-236.

SCHIFFER/SKIBO 1987
Schiffer, M.B./Skibo, J.M., Theory and experiment in the study of technological change, *Current Anthropology* 28, 595-622.

SCHIFFER 1990
Schiffer, M.B., The influence of surface treatment on heating effectiveness of ceramic vessels. *Journal of Archaeological Sciences* 17, 373–81.

SCHIFFER *et alii* 1994
Schiffer, M.B./Skibo, J.M./Boelke, T.C./Neupert, M.A./Aronson, M., New perspectives on experimental archaeology: surface treatments and thermal response of the clay cooking pot, *American Antiquity* 59 (2), 197-217.

SILVA 2008
Silva, F.A., Ceramic technology of the Asurini do Xingu, Brazil: an ethnoarchaeological study of artifact variability, *Journal of Archaeological Method and Theory* 15, 217-265.

SIRET/SIRET 1890
Siret, H./Siret, L., *Las primeras edades del metal en el Sudeste de España. Resultados obtenidos en las excavaciones hechas por los autores de 1881 a 1887* (Barcelona).

SPANGENBERG/JACOMET/SCHIBLER 2006
Spangenberg, J.E./Jacomet, S./Schibler, J., Chemical analyses of organic residues in archaeological pottery from Arbon Bleiche 3, Switzerland – evidence forndairying in the late Neolithic, *Journal of Archaeological Sciences* 33 (1), 1–13.

STARK 1991
Stark, M.T., Ceramic production and community specialization: A Kalinga ethnoarchaeological study, *World Archaeology* 23:1, 64-78.

STARK/BISHOP/MIKSA 2000
Stark, M.T./Bishop, R.L./Miksa, E., Ceramic technology and social boundaries: cultural practices in Kalinga clay selection and use, *Journal of Archaeological Method and Theory* 7 (4), 295-331.

STEELE 2008
Steele, V.J., *Organic residue analysis of Red Lustrous Wheelmade Wade vessels traded across the Eastern Mediterranean during the Late Bronze Age*. Doctoral Thesis. Universidad de Bradford.

STEELE/STERN/STOTT 2010
Steele, V.J./Stern, B./Stott, A.W., Olive oil or lard ?: Distinguishing plant oils from animal fats in the archeological record of the eastern Mediterranean using gas chromatography / combustion / isotope ratio mass spectrometry, *Rapid Communication Mass Spectrometry* 24, 3478–3484.

TERMENTZI/FOKIALAKIS/SKALTSOUNIS 2011
Termentzi, A./Fokialakis, N./Skaltsounis, A.L., Natural resins and bioactive natural products there of as potential antimicrobial agents, *Current Pharmaceutical Design*, 17 (00), 1-24.

TTE 2008
Tite, M.S., Ceramic production, provenance and use: A review, *Archaeometry* 50 (2), 216-231.

VICO *et alii* 2018
Vico, L./Molina, F./Cámara, J./Gámiz, J., El estudio tecno-tipológico de las cerámicas del Cobre Reciente de Los Castillejos (Montefrío, Granada), *SPAL* 27 (2), 29-53.

VECSTAUDŽA *et alii* 2013
Vecstaudža, J./Jakovļevs, D./Bērziņa-Cimdiņa, L./Stikāne, V., XRD and SEM studies of archaological stove tile ceramics of Turaida Castle, *Material Science and Applied Chemistry,* 40-45.

WEST 1992
West, S.M., *Temper, Thermal Shock, and Cooking Pots: A Study of Tempering Materials and Their Physical Significance in Prehistoric and Traditional Cooking Pottery*. Unpublished Master's thesis, Department of Materials Science and Engineering, University of Arizona, Tucson.

XIANG-DONG *et alii* 2008
Xiang-dong, Q./Hong-Jun, S./Ze-Jun, D./Ji-Kai, L., Six New Induced Sesquiterpenes from the Cultures of Ascomycete Daldinia concentrica, *The Journal of Antibiotics* 61, 556-562.

Fig. 1 Peñalosa location (Jaen, Spain).

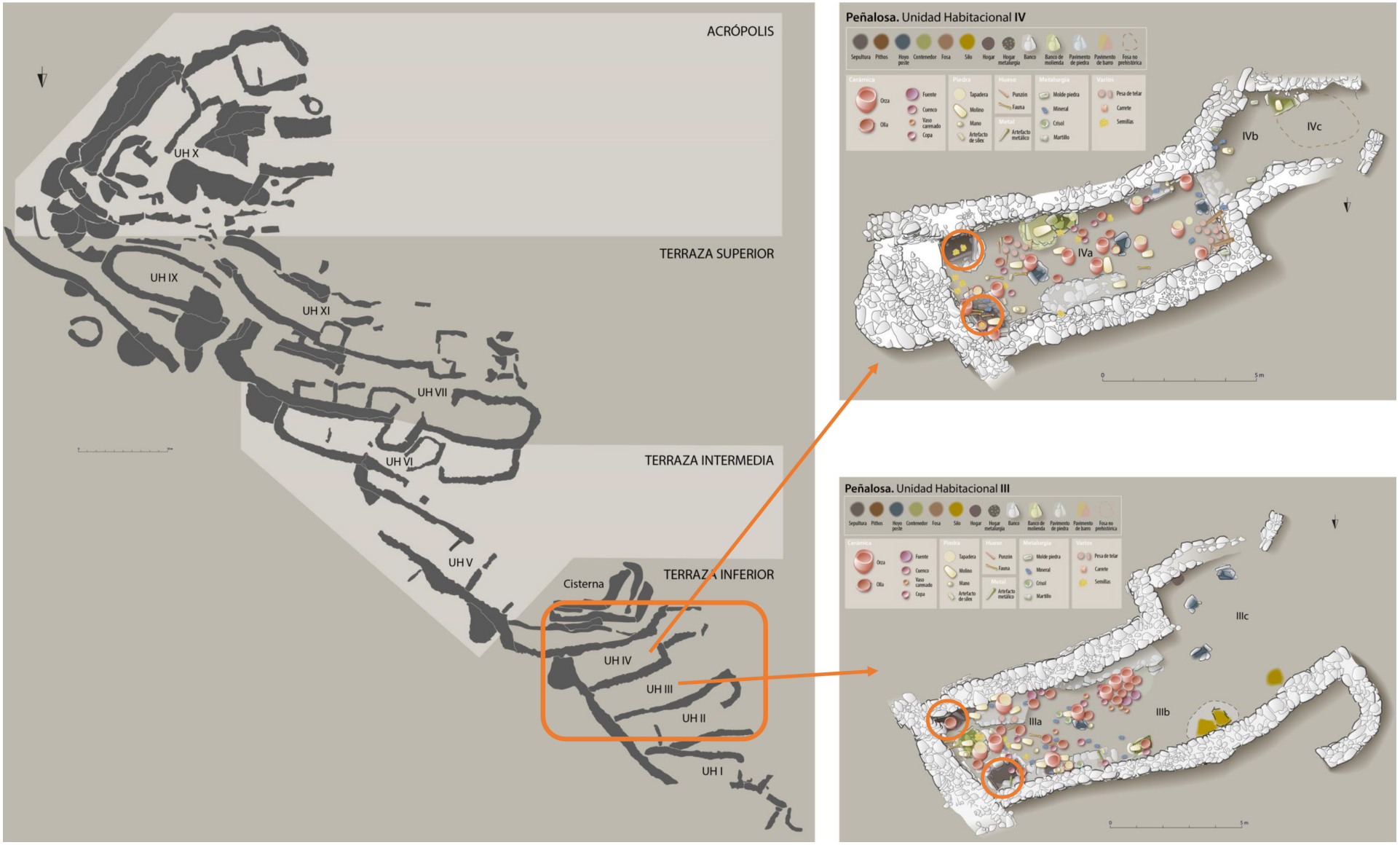

Fig. 2. Dwelling III and IV from Peñalosa (Spain) and their graves location.

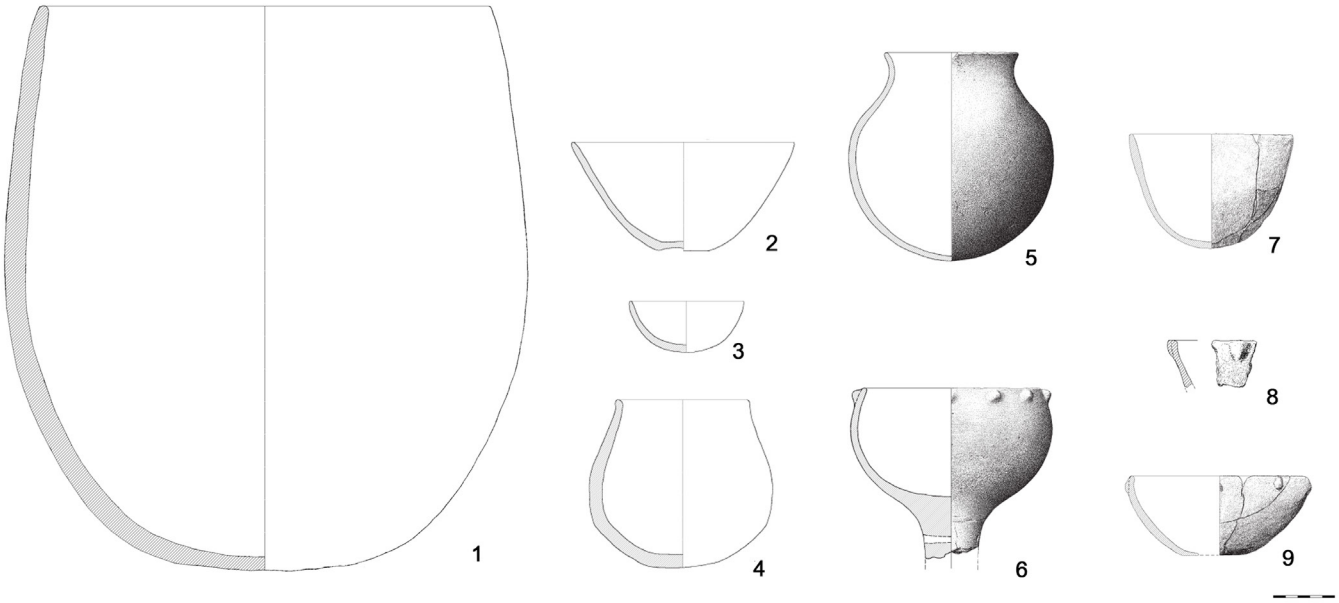

Fig 3 The nine pottery vessels studied (*14564, 14584, 14601, 20128, 20129, 20130, 20149, 20367* and *20369*) .

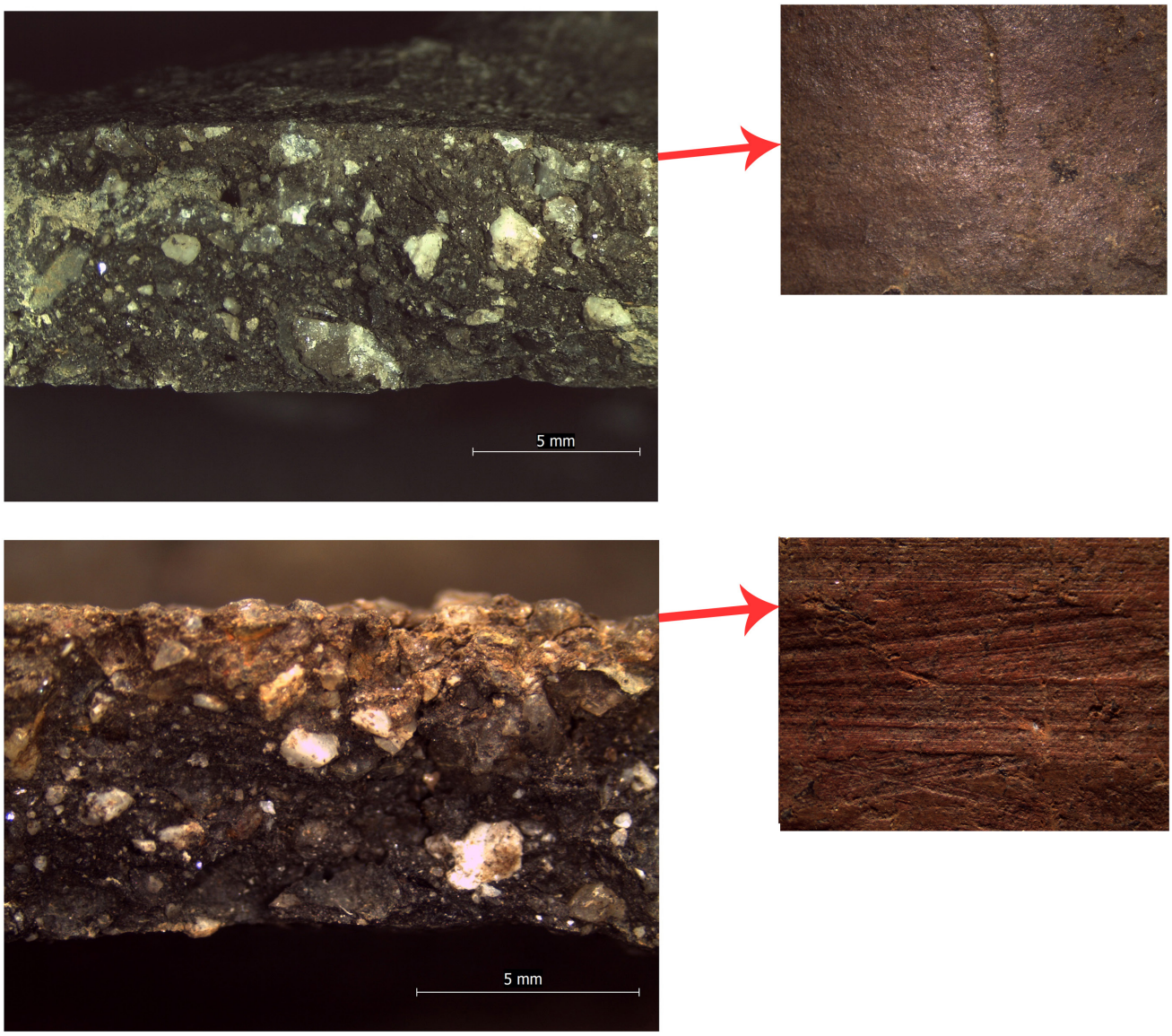

Fig. 4. Examples of pottery surfaces bearing horizontal traces of spatula (below) and polishing (above)

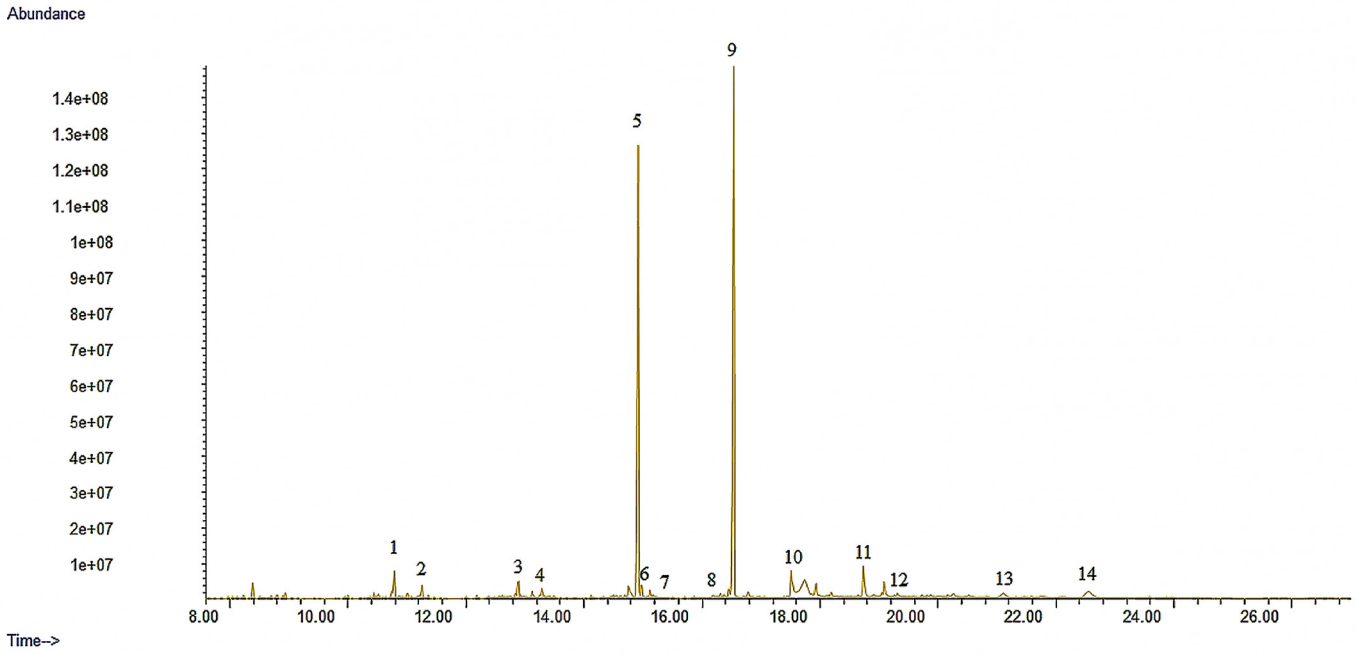

Fig. 5. Chromatogram for the organic residues extracted from vessel *14546.*Compounds identified: 1: Phenol, 2, 4.bis (1,1-dimethylethyl); 2: Hexadecane; 3: Tetradecanoic acid; 4: Pentadecanoic acid; 5: Hexadecanoic acid; 6: Methyl-3-(3,5-diterbutyl-4-hydroxyphenyl) propionate; 7: Heptadecanoic acid; 8: 9-octadecenoic acid; 9: Octadecanoic acid; 10: Hexadecane, 2,6,10,14-tetramethyl; 11: Nonadecane; 12: Tetracosanoic acid; 13: Hexacosanoic acid; 14: Octacosanoic acid.

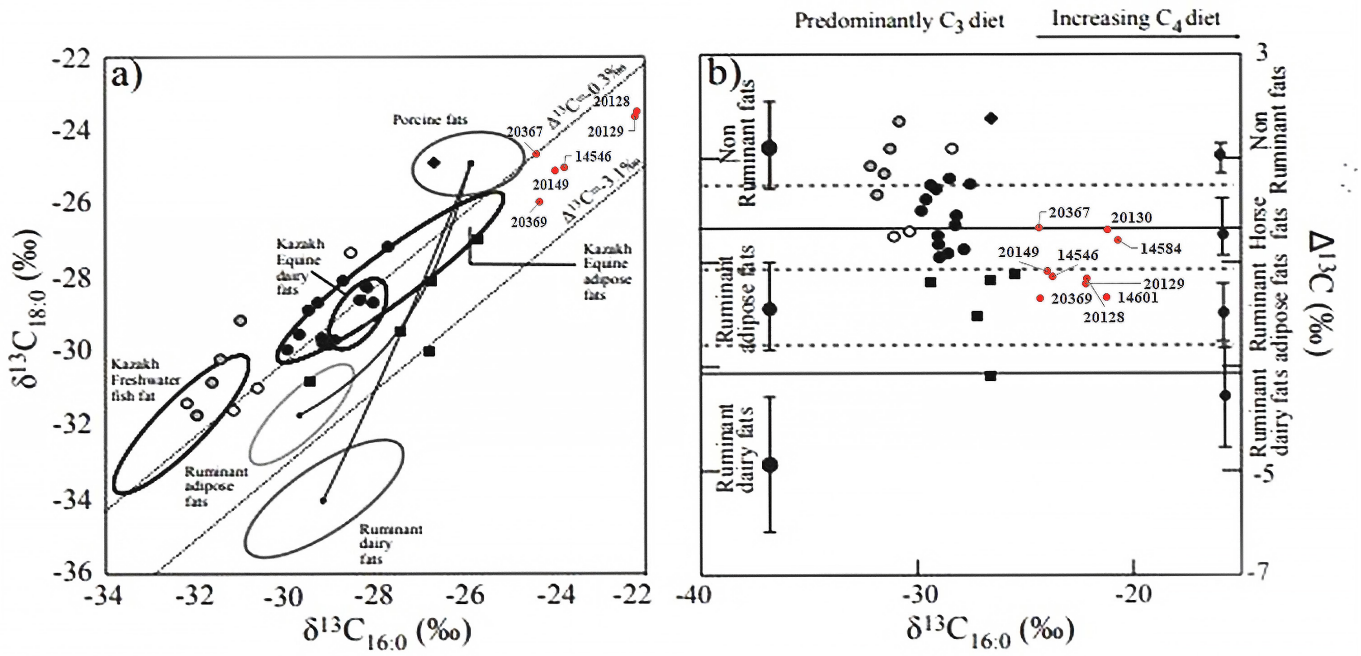

Fig. 6. Scatterplots of (a) $\delta^{13}C$ values of C16:0 fatty acid against the C18:0 fatty acid extracted from modern reference fats as reference samples (Mileto et al. 2017) and (b) $\delta^{13}C$ values of C16:0 against the $\Delta^{13}C$ values (δ13C18:0–δ13C16:0) in which the nine analyzed samples of Peñalosa are inserted.

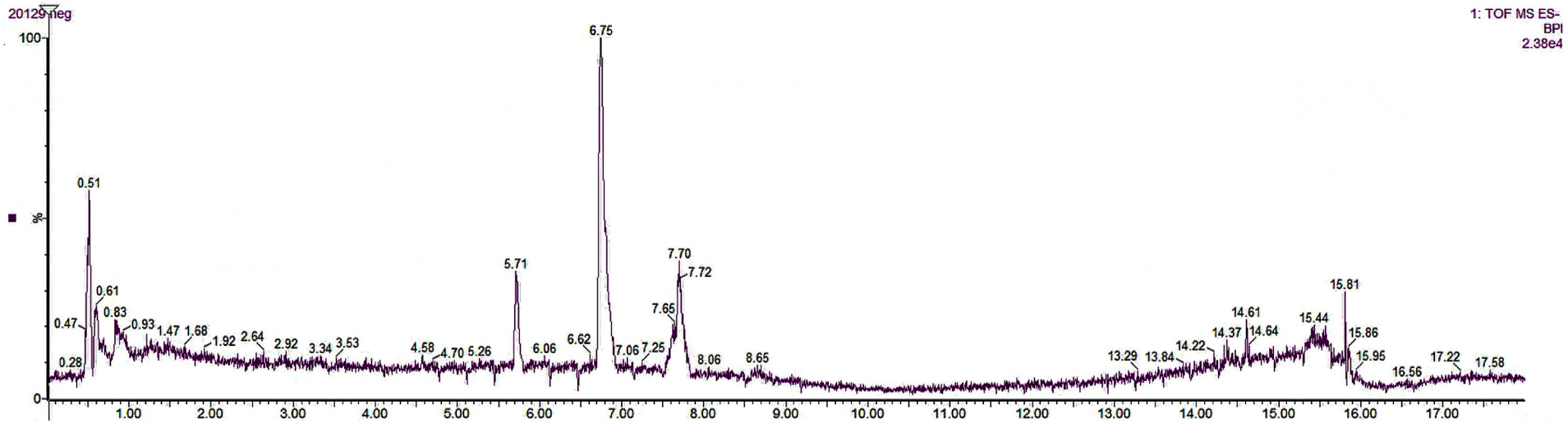

Fig. 7. Sample *20129* obtained by UPLC-HRMS.

Fig. 8. Carbonization marks and roughness at the base of *20129* due to the use of this vessel to heat food.