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.

1. INTRODUCTION

Pottery has always been considered as products/objects for use¹ that are endowed with an enormous capacity to explain and interpret past societies². 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 them³. They were designed as objects with multiple uses and functions⁴ and therefore contain cultural, political, ideological and economic codes⁵ 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

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

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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 (\textit{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 was 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 (\textit{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.

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\(^6\) ADMIRAAL \textit{et alii} 2020.


\(^8\) SIRET/SIRET 1899; CUADRADO 1947; CONTRERAS 1986; CONTRERAS \textit{et alii} 87-88; ARANDA/ESQUIVEL 2006; ARANDA \textit{et alii} 2015.


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\(^\text{11}\). 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\(^\text{12}\), 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\(^\text{13}\). 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\(^\text{14}\). 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\(^\text{15}\).

For the XRD analysis, the samples were reduced to a powder (10 microns), the size needed for their laboratory analysis\(^\text{16}\). All the samples were analysed in a BRUKER D8 ADVANCE diffractometer with Cu radiation (sealed tube) and a LIXEYE 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\(^\text{17}\) was applied to identify the mineral phases of the samples.

### 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\(^\text{18}\). 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>
<thead>
<tr>
<th>Sample</th>
<th>mg</th>
</tr>
</thead>
<tbody>
<tr>
<td>14546</td>
<td>0.983</td>
</tr>
<tr>
<td>14584</td>
<td>1.077</td>
</tr>
<tr>
<td>14601</td>
<td>0.927</td>
</tr>
<tr>
<td>20128</td>
<td>1.021</td>
</tr>
<tr>
<td>20129</td>
<td>1.056</td>
</tr>
<tr>
<td>20130</td>
<td>0.986</td>
</tr>
<tr>
<td>20149</td>
<td>1.028</td>
</tr>
<tr>
<td>20367</td>
<td>1.028</td>
</tr>
<tr>
<td>20369</td>
<td>1.040</td>
</tr>
</tbody>
</table>

#### 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-trifluoromethylphenyltrimethylammonium 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-trifluoromethylphenyltrimethylammonium 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, 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 methanol 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\(^\text{19}\) 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

\(^\text{13}\) HOLAKOÖEL et alii 2014. 
\(^\text{15}\) CASTRO 1989; GÁMIZ et alii 2013. 
\(^\text{16}\) NAVARRO 2008. 
\(^\text{17}\) CHUNG 1974; MARTÍN 2004. 
\(^\text{18}\) DUNNE 2017. 
\(^\text{19}\) EVERSHEDE 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 studies20. Prior to the GC–IRMS 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 previously21. 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 1.9×10−6 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 δ13C (%)=[(Rsample–Rstandard) / Rstandard] × 1000, where R is 13C/12C in per mil. Accurate and reproducible were calculated using triplicate injections for each sample. CO2 gas of a known isotopic composition was used as working reference standard. Desviations were estimated

$$\delta^{13}C_{\text{sample}} = \frac{(29 \times \delta^{13}C_{\text{measured}} - \delta^{13}C_{\text{sample}})}{28},$$

for the methyl group added during the methylation process: Error, 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 maintained 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) surface22 (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

21 MANZANO et alii 2015.
Table 2: Technological groups and characteristics of funerary pottery from Peñalosa.

<table>
<thead>
<tr>
<th>Sample</th>
<th>Shape</th>
<th>Compact</th>
<th>Temper size</th>
<th>Temper (%)</th>
<th>Technological group</th>
</tr>
</thead>
<tbody>
<tr>
<td>20129</td>
<td>Cooking pot</td>
<td>Compact</td>
<td>Medium</td>
<td>30%</td>
<td>1</td>
</tr>
<tr>
<td>20149</td>
<td>Cooking pot</td>
<td>Compact</td>
<td>Medium</td>
<td>30%</td>
<td>1</td>
</tr>
<tr>
<td>14546</td>
<td>Cooking pot</td>
<td>Compact</td>
<td>Medium</td>
<td>40%</td>
<td>1</td>
</tr>
<tr>
<td>14584</td>
<td>Cooking pot</td>
<td>Compact</td>
<td>Medium</td>
<td>40%</td>
<td>1</td>
</tr>
<tr>
<td>20128</td>
<td>Bowl</td>
<td>Compact</td>
<td>Small</td>
<td>40%</td>
<td>2</td>
</tr>
<tr>
<td>20130</td>
<td>Bowl</td>
<td>Compact</td>
<td>Small</td>
<td>30%</td>
<td>2</td>
</tr>
<tr>
<td>20367</td>
<td>Bowl</td>
<td>Compact</td>
<td>Small</td>
<td>40%</td>
<td>2</td>
</tr>
<tr>
<td>20369</td>
<td>Bowl</td>
<td>Compact</td>
<td>Small</td>
<td>30%</td>
<td>2</td>
</tr>
<tr>
<td>14601</td>
<td>Chalice</td>
<td>Compact</td>
<td>Small</td>
<td>30%</td>
<td>2</td>
</tr>
</tbody>
</table>

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

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

24 DRUC/CHAVEZ 2014.

through coil pottery, consisting of the superposition of layers of clay, 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. 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. 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 matrices we observed a predominant mineral of larger dimensions than the rest, which was identified by X-ray diffraction as quartz (Table 3). In all the cases we documented the 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. 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, as well as the presence in all the samples of peaks of thermally modified phyllosilicates, indicates that the vessels were...
Table 4: Peak assignment to compounds identified (% assignment greater than 90%), retention time (tR) and m/z selected.

<table>
<thead>
<tr>
<th>tR (min.)</th>
<th>m/z</th>
<th>Compound</th>
</tr>
</thead>
<tbody>
<tr>
<td>1</td>
<td>7.754</td>
<td>172 Nonanoic acid</td>
</tr>
<tr>
<td>2</td>
<td>7.966</td>
<td>174 Hexanediolic acid, dimethyl ester</td>
</tr>
<tr>
<td>3</td>
<td>9.435</td>
<td>172 Decanoic acid</td>
</tr>
<tr>
<td>4</td>
<td>9.496</td>
<td>186 Nonanoic acid, 4-oxo</td>
</tr>
<tr>
<td>5</td>
<td>9.602</td>
<td>216 Propanoic acid, 2-methyl-, 3-hydroxy-2,4,4-trimethylpentyl ester</td>
</tr>
<tr>
<td>6</td>
<td>10.435</td>
<td>194 1,2-Benzenedicarboxilic acid, dimethyl ester</td>
</tr>
<tr>
<td>7</td>
<td>10.995</td>
<td>226 Phenol, 2,4-bis[1,1-dimethylethyl]</td>
</tr>
<tr>
<td>8</td>
<td>11.374</td>
<td>216 Nonanedioic acid, dimethyl ester</td>
</tr>
<tr>
<td>9</td>
<td>11.872</td>
<td>222 1,2-Benzenedicarboxilic acid, diethyl ester</td>
</tr>
<tr>
<td>10</td>
<td>11.919</td>
<td>226 Hexadecane</td>
</tr>
<tr>
<td>11</td>
<td>12.464</td>
<td>226 Dihydro methyl jasmonate</td>
</tr>
<tr>
<td>12</td>
<td>12.721</td>
<td>258 Hexanediolic acid, bis[2-methylpropyl] ester</td>
</tr>
<tr>
<td>13</td>
<td>13.115</td>
<td>242 Tetradecanoic acid</td>
</tr>
<tr>
<td>14</td>
<td>13.766</td>
<td>256 Tetradecanoic acid, 12-methyl</td>
</tr>
<tr>
<td>15</td>
<td>13.812</td>
<td>254 Octadecane</td>
</tr>
<tr>
<td>16</td>
<td>13.857</td>
<td>282 Hexadecane, 2,6,10,14-tetramethyl</td>
</tr>
<tr>
<td>17</td>
<td>14.024</td>
<td>256 Pentadecanoic acid</td>
</tr>
<tr>
<td>18</td>
<td>14.387</td>
<td>278 1,2-Benzenedicarboxilic acid, bis[2-methylpropyl] ester</td>
</tr>
<tr>
<td>19</td>
<td>14.690</td>
<td>268 Nonadecane</td>
</tr>
<tr>
<td>20</td>
<td>14.902</td>
<td>270 Hexadecanoic acid</td>
</tr>
<tr>
<td>21</td>
<td>14.993</td>
<td>292 Methyl-3-(3,5-ditertbutil-4-hydroxiphenyl) propionate</td>
</tr>
<tr>
<td>22</td>
<td>15.296</td>
<td>278 1,2-Benzenedicarboxilic acid, dibutyl ester</td>
</tr>
<tr>
<td>23</td>
<td>15.417</td>
<td>284 Hexadecanoic acid, 14-methyl</td>
</tr>
<tr>
<td>24</td>
<td>15.478</td>
<td>284 Hexadecanoic acid, 15-methyl</td>
</tr>
<tr>
<td>25</td>
<td>15.523</td>
<td>282 Eicosane</td>
</tr>
<tr>
<td>26</td>
<td>15.720</td>
<td>284 Heptadecanoic acid</td>
</tr>
<tr>
<td>27</td>
<td>16.023</td>
<td>288 14-beta-h-pregna</td>
</tr>
<tr>
<td>28</td>
<td>16.311</td>
<td>296 9-Octadecanoic acid</td>
</tr>
<tr>
<td>29</td>
<td>16.326</td>
<td>296 Heneicosane</td>
</tr>
<tr>
<td>30</td>
<td>16.538</td>
<td>298 Octadecanoic acid</td>
</tr>
<tr>
<td>31</td>
<td>16.720</td>
<td>342 1-Propene-1,2,3-tricarboxilic acid, tributyl ester</td>
</tr>
<tr>
<td>32</td>
<td>17.083</td>
<td>310 Docosane</td>
</tr>
<tr>
<td>33</td>
<td>17.250</td>
<td>312 Nonadecanoic acid</td>
</tr>
<tr>
<td>34</td>
<td>17.931</td>
<td>326 Eicosanoic acid</td>
</tr>
<tr>
<td>35</td>
<td>18.204</td>
<td>324 Tricosane</td>
</tr>
<tr>
<td>36</td>
<td>18.355</td>
<td>338 Tetracosane</td>
</tr>
<tr>
<td>37</td>
<td>18.507</td>
<td>340 Heneicosanoic acid</td>
</tr>
<tr>
<td>38</td>
<td>18.900</td>
<td>352 Pentacosane</td>
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<td>19.052</td>
<td>354 Docosanoic acid</td>
</tr>
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<td>40</td>
<td>19.082</td>
<td>390 1,2-Benzenedicarboxilic acid, bis[2-etilhexyl] ester</td>
</tr>
<tr>
<td>41</td>
<td>19.431</td>
<td>328 Dehydroabietic acid</td>
</tr>
<tr>
<td>42</td>
<td>19.582</td>
<td>368 Tricosanoic acid</td>
</tr>
<tr>
<td>43</td>
<td>19.976</td>
<td>380 Heptacosane</td>
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<tr>
<td>44</td>
<td>20.173</td>
<td>382 Tetracosanoic acid</td>
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<tr>
<td>45</td>
<td>21.582</td>
<td>410 Hexacosanoic acid</td>
</tr>
<tr>
<td>46</td>
<td>23.567</td>
<td>438 Octacosanoic acid</td>
</tr>
<tr>
<td>47</td>
<td>24.734</td>
<td>382 Cholesta-3,5-dien-7-one</td>
</tr>
</tbody>
</table>
heated to more than 500 °C. However, there are various factors that indicate that the temperatures did not exceed 800 °C: 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 °C\(^2\) 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 (C26), 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)\(^3\). Animal fats were clearly identified in three of the nine pottery vessels studied according to their carbon isotopic composition.

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\(^4\), 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\(^5\). The isotopic values of these samples are shown in figure 6.

**Table 5: Isotopic values of the samples analysed**

<table>
<thead>
<tr>
<th>Sample</th>
<th>δ13C16:0</th>
<th>δ13C18:0</th>
</tr>
</thead>
<tbody>
<tr>
<td>20128</td>
<td>-22.23</td>
<td>-23.50</td>
</tr>
<tr>
<td>20129</td>
<td>-22.27</td>
<td>-23.64</td>
</tr>
<tr>
<td>20130</td>
<td>-21.30</td>
<td>-21.63</td>
</tr>
<tr>
<td>20149</td>
<td>-23.99</td>
<td>-25.12</td>
</tr>
<tr>
<td>20367</td>
<td>-24.38</td>
<td>-24.67</td>
</tr>
<tr>
<td>20369</td>
<td>-24.31</td>
<td>-25.96</td>
</tr>
<tr>
<td>14546</td>
<td>-23.79</td>
<td>-25.03</td>
</tr>
<tr>
<td>14584</td>
<td>-20.85</td>
<td>-21.39</td>
</tr>
<tr>
<td>14601</td>
<td>-21.34</td>
<td>-22.97</td>
</tr>
</tbody>
</table>

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-methyldeacetylidyhydrobrotlydial associated with polyphenols of vegetable origin that were associated with fungi and mushrooms such as *Daldinia concentrica*, which has medicinal properties\(^5\) (Fig.7). In pot 14584, the formula...
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 properties38. Both compounds were identified using a highly accurate program (Mass Fragment) that allow us to analyze the structure using major 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 tombs39.

However, we observe 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 for which the vessels were destined to be used for, as they produced pastes that are more resistant. Thanks to these qualities, the pots obtained objects with greater mechanical, fire and heat resistance, making it less likely that the pastes would crack40. Likewise, there are fire exposure marks (soot, scratches and abrasions) on these vessels, which show that they were used for other purposes before burial placement41 (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 properties42 and in pot 14584 with a kind of pasture native to Eurasia with anti-inflammatory and medicinal properties43, 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 eaten44. Waxy material related to beeswax was also identified in both vessels45, perhaps residues associated with their content. Conifer resins were found through the identification of dehydroabietic acid in vessel 2012946. 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 content47. 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 properties48.

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 Steele49, 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 walls50. These residues are complemented in the flat pot with compounds associated with the conifer resins that were linked to their impermeability51. 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 of...
of vegetable fats in the case of the parabolic bowl and the cup. 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 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 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

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Fig. 1. Petralosa 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: Heptacosanoic acid; 14: Octacosanoic acid.

Fig. 6. Scatterplots of (a) δ¹³C values of C16:0 fatty acid against the C18:0 fatty acid extracted from modern reference fats as reference samples (Mileo et al. 2017) and (b) δ¹³C values of C16:0 against the Δ¹³C 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.