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ANALYSIS OF ORGANIC RESIDUES IN THE CONTEXT OF THE MIDDLE BRONZE AGE IN THE NORTHWESTERN IBERIAN PENINSULA: THE CASE OF THE COVA DA MOURA FUNERARY MONUMENT (VIANA DO CASTELO, NW PORTUGAL)

ANÁLISE DE RESÍDUOS ORGÂNICOS NO CONTEXTO DA IDADE DO BRONZE MÉDIO DO NOROESTE DA PENÍNSULA IBÉRICA: O CASO DO MONUMENTO FUNERÁRIO DA COVA DA MOURA (VIANA DO CASTELO, NOROESTE DE PORTUGAL)

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Abstract This paper presents and discusses the results of chromatographic analyses conducted on the contents of a ceramic pot recovered from *Cova da Moura*, a coastal funerary context located on the western slope of the Serra de Santa Luzia in Carreço, within the municipality and district of Viana do Castelo, Northwest Portugal.

The site was excavated in 1931 by Abel Viana, who uncovered a monument beneath a tumulus that concealed a cluster of granite outcrops with numerous cavities (*tafoni*). Depositional activities in these cavities revealed several artifacts, including fragments of various ceramic containers, metal and glass objects, knapped and polished stone tools, animal remains, a diverse assemblage of plant-derived charcoal, and at least one anthropomorphic stele. The materials exhibit a wide chronological range, with the earliest occupation dating from the Late Middle Bronze Age (circa 15th-14th century BCE) and the most recent from the Late Iron Age.

Among these finds was a fragmented ceramic pot containing organic residues at its internal base, dated from the regional Bronze Age. Chromatographic analysis of these residues identified traces of animal meat, possibly from monogastric species, and plant oils, both of which appear to have been exposed to high temperatures.

This study represents the first application of organic chemical analysis to a ceramic vessel from a plausible Middle Bronze Age funerary context in Northwestern Portugal. The findings suggest the presence of commensal rituals or the deposition of prepared meat offerings as part of funerary practices.

Keywords: Commensality ceremonies, Chromatographic analysis, Funerary context, Middle Bronze Age.

Resumo Este trabalho tem como objetivo apresentar e discutir os resultados das análises cromatográficas efetuadas aos conteúdos de um vaso depositado num contexto funerário costeiro, no sítio da Cova da Moura, situado na vertente oeste da Serra de Santa Luzia, em Carreço, concelho e distrito de Viana do Castelo, Noroeste de Portugal.

O sítio arqueológico foi explorado, em 1931, por Abel Viana, tendo revelado um monumento sob *tumulus*, que tapou um aglomerado de afloramentos graníticos com inúmeras cavidades (*tafoni*), onde se terão efetuado deposições. Nelas foi recuperado um conjunto de fragmentos de diversos recipientes cerâmicos, e outros materiais metálicos e vítreos, além de peças líticas talhadas e polidas, restos de animais, uma grande variedade de carvões de origem vegetal e, pelo menos, uma estela de perfil antropomórfico. A cronologia dos materiais encontrados tem uma larga diacronia, sendo a ocupação mais antiga dos finais da Idade do Bronze Médio – entre o início do século XV ao início do século XIV a.C. – e a mais tardia da Idade do Ferro Recente.

Neste contexto foram encontrados restos de um pote, datado da Idade do Bronze regional, que se encontrava fragmentado e continha vestígios orgânicos no seu interior (no fundo). Estes vestígios foram estudados através de análises cromatográficas, o que revelou resíduos de carne de animais, possivelmente monogástricos, e óleos de plantas, tendo estes conteúdos sido exposto a temperaturas elevadas.

Trata-se do primeiro vaso, indiscutivelmente proveniente de um contexto funerário da Idade do Bronze Médio do Noroeste de Portugal, a ser analisado por cromatografia gasosa, tendo o seu estudo evidenciado ritos de comensalidade ou deposições de preparados de carne no contexto das práticas funerárias.

Palavras-chave: Cerimónias de comensalidade, Análises cromatográficas, Contexto funerário, Idade do Bronze Médio.

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1. Introduction

Chromatographic studies of organic residues preserved in archaeological artifacts have become an indispensable tool for revealing little-known aspects of the daily and symbolic lives of ancient societies (Evershed *et al*., 1990; Polla & Springer, 2022; Rowe, 2024; Shoda *et al*., 2018). By identifying chemical markers, these analyses provide insights into dietary practices, such as the preparation and consumption of food, as well as on ceremonial rituals and the use of substances for medicinal or symbolic purposes (Becher *et al*., 2024; Miller *et al*., 2020; Whelton *et al*., 2021).

The use of this analytical approach for prehistoric vessels in the Iberian Peninsula remains relatively insipient, especially regarding ceramic vessels tied to Bronze Age funerary rituals in Northwestern Portugal. To bridge the knowledge gaps in this domain, a research initiative was launched utilizing gas chromatography-mass spectrometry (GC-MS) to analyze and characterize organic residues preserved in archaeological materials from this time period.

This paper aims to present the results of chromatographic analyses conducted on the contents of a ceramic container deposited in the funerary monument of Cova da Moura, in Northwest Portugal. Within the set of fragmentary ceramic remains associated with monument, most typically small in size, this specimen was the only one that exhibited organic residues capable of being studied through gas chromatography; in turn, of providing some information about its contents and, consequently, about Bronze Age funeral rites.

2. Archaeological Context

The ceramic vessel analyzed in this study was discovered during archaeological excavations conducted by Abel Viana in 1931, at the site then referred to as Cova da Moura (Viana, 1932a, 1932b, 1932c, 1932d, 1932e, 1932f, 1932g, 1932h, 1932i, 1943, 1955). The site is in the parish of Carreço, within the municipality and district of Viana do Castelo, Northwest of Portugal. It lies on a platform at an average altitude of 186 meters a.s.l., halfway up to the western slope of the Santa Luzia Mountain overlooking the Atlantic coastal platform (Oliveira, 2024: 854-913) (Fig. 1).

The geological substrate of the site consists of fine-grained porphyroid granite, which is abundantly exposed, particularly to the north-northeast and south-southwest area of the monument. Less than 1 kilometer to the northeast, there is an occurrence of Andalusian schist (Teixeira & Medeiros, 1972) (Fig. 1).

The monument consists of a large artificial mound measuring approximately 45 meters in length (north-south), 28 meters in width (east-west), and up to 11 meters in maximum height. It was constructed by depositing sediments over various granite outcrops that feature natural shelters and *tafoni* (small natural cavities), and was covered with a lithic armor, which remains particularly prominent on the western side (Viana, 1943; 1955: 487-488; Bettencourt, 2013: 165; Oliveira, 2024: 861-867).

There were two walls inside the monument, at least in its northern half. The western wall, constructed from unworked granite blocks, was double-layered and designed to contain the sediment on that side. However, it left exposed the entrance to a granite cavity, which Viana (1955: 486) describes as being "(...) very irregular, decreasing in height from the entrance to the bottom and from the North side to the south, but with enough space to fit a dozen crouching people" (Fig. 2).

On the opposite side, there was a wall measuring over 4 meters in length, which was heavily damaged during the excavation. This wall was constructed from granite blocks approximately 30 to 40 centimeters in length, arranged without fitting and without the use of mortar. Its original height could not be determined.

These walls appear to have been constructed in at least two distinct phases (Viana, 1955: 487-488), due to the differing construction techniques employed, although the phases may not have been far apart in time. Notably, the walls do not extend to the top of the monument, raising several questions. Could these walls have delimited a corridor that may have been covered, or might the space have remained open during a certain period of use, serving solely as a circulation passage for depositing materials in the side cavity or central ground-level cavities? Were the sediments visible on the walls contemporary with the walls' construction, or were they deposited later, during the monument's closure?

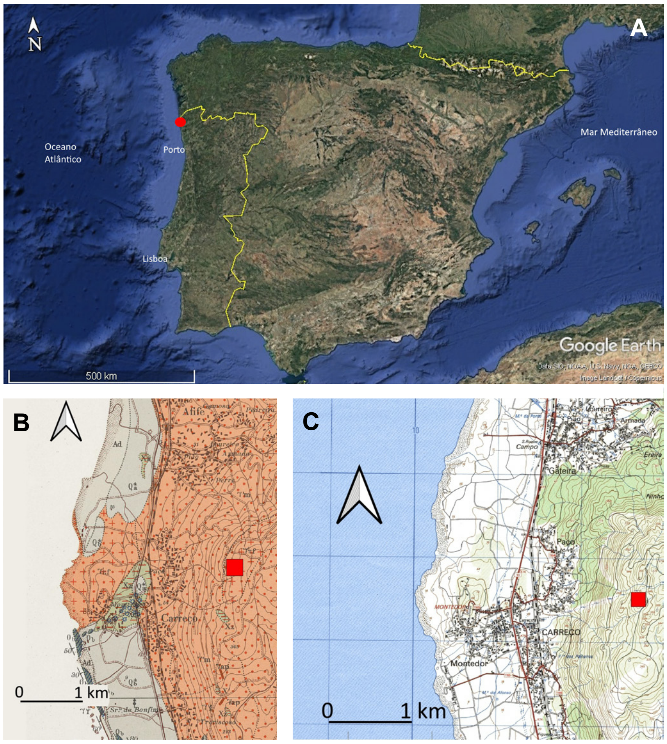


Fig. 1. A: Location of Cova da Moura at Iberian Peninsula (source: Google Earth). B: Location of Cova da Moura on the Geological Map of Portugal, scale 1:50000, sheet 5A (Viana do Castelo), 1970, Serviços Geológicos de Portugal. The Yπf symbol corresponds to fine-grained porphyroid granite; the Xz symbol corresponds to Andalusian schists. C: Location of Cova da Moura on the Military Map of Portugal, series M888, sheet no. 27 (Vila Praia de Âncora), scale 1:25000, CIGeoE.

Fig. 1. A: Localização da Cova da Moura na Península Ibérica (fonte: Google Earth). B: Localização da Cova da Moura na Carta Geológica de Portugal, escala 1:50000, folha 5A (Viana do Castelo), 1970, Serviços Geológicos de Portugal. O símbolo Yπf corresponde a granito porfiroide de grão fino; o símbolo Xz corresponde a xistos andaluzíticos. C: Localização da Cova da Moura na Carta Militar de Portugal, série M888, folha n.º 27 (Vila Praia de Âncora), escala 1:25000, CIGeoE.

According to Viana, the shelters and small cavities within the structure may have been used for cremation deposits, sometimes accompanied by lithic, ceramic, or metallic artifacts, as well as eight mammalian bones and teeth that were also recovered.

Fig 2. A: Aspect of the western profile of the interior of the Cova da Moura monument, where it is possible to see a sediment containment wall and the entrance to a natural cavity (Viana, 1955: 499, Est. 1, adapted). B: West side wall as it is today (Oliveira, 2024: 867, Figure 7.9). C and D: Photographs of the interior of the cavity as it is today (Oliveira, 2024: 870, Figure 7.11).

Fig. 2. A: Aspeto do perfil oeste do interior do monumento da Cova da Moura, onde é possível observar um muro de contenção de sedimentos e a entrada para uma cavidade natural (Viana, 1955: 499, Est. 1, adaptado). B: Muro do lado oeste na sua forma atual (Oliveira, 2024: 867, Figura 7.9). C e D: Fotografias do interior da cavidade tal como se encontra atualmente (Oliveira, 2024: 870, Figura 7.11).

He noted that "remains of ashes" were deposited in the central part of the monument, within the shelters, and beneath other excavated layers. He further elaborated: "The fragments of ceramic vessels that were discovered suggest that part of the ashes were enclosed in funerary urns, or more likely in simple pieces of pottery (...) It is to be believed that these containers were placed under the slabs, where they were, without any arrangement that would favor their preservation, as a result of which everything was crushed under the formidable weight of the roof" (Viana, 1955: 489).

The pot under analysis was found near the center of the monument, close to the entrance of a granite cavity. In reference to this context, Viana noted: "Only in the layer discovered in the center, in which the copper bead and snare were found, was there a larger number of shards, especially the one that corresponds to the bottom of a vessel; but equally small and scarce are the fragments that can be attributed to the bowl, and the rim only a very limited portion" (Viana, 1955: 489) (original in Portuguese).

Although Bettencourt suggested that this monument was constructed during the Late Bronze Age, based on the discovery of a Rocannes-type sickle (Bettencourt, 2013: 165), a recent review of archaeological materials stored at the Geological Museum of Portugal in Lisbon (Oliveira, 2024: 873-904), alongside with radiocarbon dating obtained from organic matter found in the ceramic container studied in this paper (see below), indicates an initial construction phase at the end of the Middle Bronze Age. Nevertheless, later reconstructions cannot be ruled out, as the site was used over an extended diachronic period, including the Late Bronze Age and the Early and Late Iron Ages, spanning the 1st millennium BCE (Oliveira, 2024: 905).

3. The Ceramic Container

The pot under study is a handmade piece composed of a sandy paste with large-sized temper inclusions, including prominent quartz grains, and with mica particles visible on the exterior, likely originating from the clay's natural composition. The texture is coarse, while the finish is smooth. In formal terms, the rim is slightly open, the lip is semi-flat, and the base is a simple flat bottom. Based on the drawing (Fig. 3), the pot exhibits a very smooth S-shaped profile, with a rim diameter of 37.8 cm (Oliveira, 2024: 874-875). Considering these characteristics, the pot has been attributed to a Bronze Age chronology and typologically classified as a form 2 pot (Fig. 3), according to Bettencourt's table of forms for this period (Bettencourt, 1999: 1039-1054).

Evidence of fire exposure is present on the exterior of the base and walls of the pot. Inside, particularly at the bottom, a significant concentration of organic debris was observed. These residues were sampled for analysis by chromatography.

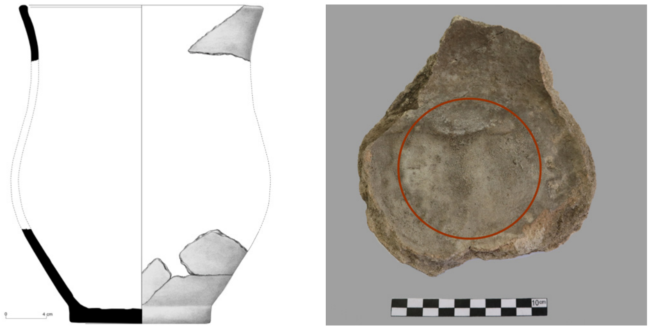


Fig. 3. Pot of form 2 and traces of organic contents inside its bottom (Oliveira, 2024: 877, Figs. 7.14 and 7.15, adapted).

Fig. 3. Pote da forma 2 e vestígios de conteúdo orgânico no seu fundo (Oliveira, 2024: 877, Figs. 7.14 e 7.15, adaptadas).

Radiocarbon dating was conducted at the Vilnius Radiocarbon Laboratory of the Center for Physical Sciences and Technology in Lithuania on a sample of the organic residue from the pot. The date was calibrated online using OxCal v4.4.4 software (Bronk Ramsey, 2021) and IntCal20 calibration curve (Reimer *et al.*, 2020). The radiocarbon date of 3261±31 BP places the pot within a time span from the middle of the 15th century to the end of the 14th century BC, corresponding to the conclusion of the regional Middle Bronze Age (Table 1).

Table 1. Radiocarbon Age

Quadro 1. Datação por radiocarbono

Lab. Ref.	Context	^{14}Cya	Cal. 1σ BCE	Cal. 2σ BCE
FTMC-FU30-4	Organic matter at the bottom of the container	3174 ± 29 BP	1492-1481 (7.5%) **1451-1398 (60.8%)**	**1499-1383 (85.7%)** 1341-1313 (9.8%)

4. Organic Residue Analysis

The ceramic was handled with nitrile gloves and stored in aluminum foil to minimize the risk of contamination. Powdered samples were collected from the inside bottom of the vessel, where organic residues had adhered, and analyzed using gas chromatography-mass spectrometry (GC-MS). The analysis, conducted at the HERCULES Laboratory of the University of Évora, successfully identified the organic residues present. Special care was taken on the detection of contaminants, being the chromatograms examined for common organic contaminants, such as squalene, a compound from skin oils indicating ungloved handling of the ceramic material, personal cosmetic products typically linked to ungloved handling, and plasticizers.

4.1. Sample preparation and analysis

The samples were collected using a clean scalpel with sterilized blades and finely ground with an agate mortar and pestle. To this, 7 mL of a chloroform: methanol mixture (2:1 v/v) was added. The mixtures were vortexed, sonicated for 20 minutes, and centrifuged at 2500 rpm for 15 minutes. The supernatants were carefully transferred using clean glass Pasteur pipettes, and the extraction process was repeated. The supernatants from each sample were combined and dried at 40° C under a gentle stream of nitrogen. The dried extracts were then suspended in *n*-hexane and derivatized with *N,O*-bis (trimethylsilyl) trifluoroacetamide containing 1% trimethylchlorosilane (BSTFA + 1% TMCS) in a microwave oven (700 W, 30 seconds). After excess derivatizing agent was removed under a gentle stream of nitrogen, the extracts were dissolved in *n*-hexane and analyzed by GC-MS.

The chromatographic analyses were performed with a Shimadzu GC2010 gas chromatograph coupled to a GCMS-QP2010 Plus Mass Spectrometer device equipped with a column Zebron ZB-5HT. The identification of compounds was based on the analysis of fragmentation patterns and the comparison of resulting spectra with spectra from the commercial libraries Wiley 8 and Nist17.

4.2. Results

The GC-MS analysis of the total lipids extract (chromatogram at Fig. 4) shows the presence of degraded lipids, clearly presenting traces of triacylglycerol degradation products (Evershed, 1993, 2008; Roffet-Salque *et al.*, 2017; Romanus *et al.*, 2007; Rosiak *et al.*, 2020), as the intense peaks of monoacylglycerols (2- and 1-monostearin, 2- and 1-monopalmitin, the most intense detected peak), and diacylglycerols (1,2- and 1,3-dipalmitin, 1,2- and 1,3-distearin, and 1,2- and 1,3 $C_{18:0}$/$C_{16:0}$ diacylglycerols) (Fig. 5a).

The chromatogram reveals a homologous series of alkanoic acids ranging from C_{14} to C_{34}, with intense peaks of palmitic ($C_{16:0}$) and stearic ($C_{18:0}$) acids (Fig. 5b). The palmitic-to-stearic acid ratio is a key tool in lipid analysis for determining the origin of organic residues, such as differentiating between dairy, adipose, or aquatic sources (Kimpe *et al.*, 2004; Romanus *et al.*, 2007). Ratios below 1 reflect a higher proportion of stearic acid, which is typically associated with animal fats. As the value obtained was approximately 0.73, it suggests a strong predominance of animal fats over vegetable oils (Romanus *et al.*, 2007). This conclusion is further supported by the presence of cholesterol (Fig. 4), a sterol of animal origin.

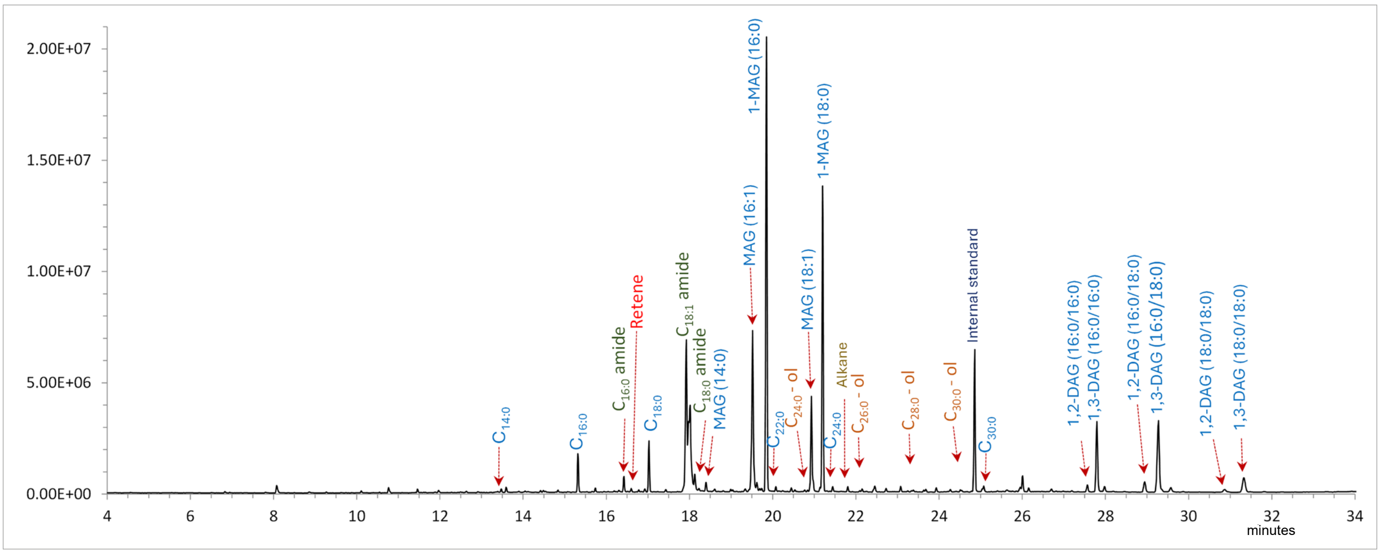


Fig. 4. Gas chromatogram obtained from the total lipids extract. $C_{n:x}$ - fatty acids with carbon length *n* and number of unsaturation *x*; ol - alcohols; MAG: monoacylglycerols; DAG: diacylglycerols. Compounds marked in blue are degradation products of triacylglycerols from fats; in green, compounds resulting from the heating and breakdown of nitrogen-rich meat proteins; and in red, a biomarker for coniferous tree resin, produced by the thermal degradation of abietic acid.

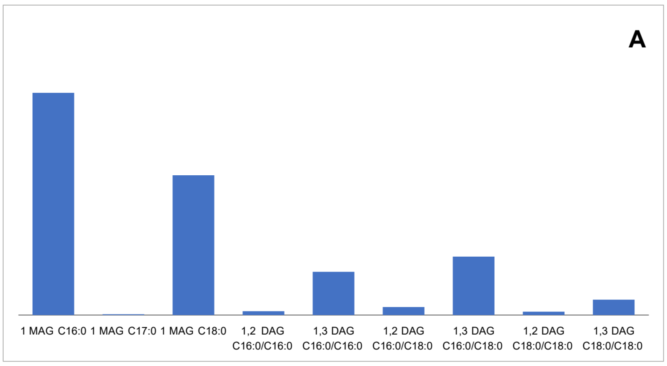
Fig. 4. Cromatograma gasoso do extrato lipídico total. $C_{n:x}$ - ácidos gordos com *n* átomos de carbono e um grau de insaturação *x*; ol - álcoois; MAG: monoacilgliceróis; DAG: diacilgliceróis. A azul representa-se produtos de degradação dos triacilgliceróis oriundos de gorduras; a verde, os compostos formados pelo aquecimento e decomposição de proteínas de carnes ricas em azoto; e a vermelho, identifica-se um biomarcador de resina de árvores coníferas, produzido pela degradação térmica do ácido abiético.

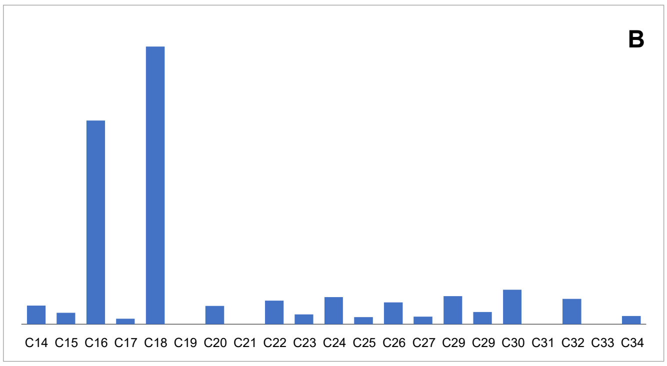
The possible detection of several isomers of $C_{18:1}$ fatty acid resulting from biohydrogenation processes in the rumen is a distinctive feature of ruminant fats. Additionally, ruminant fats typically exhibit diverse isomers of odd-chain fatty acids such as C_{15} and C_{17} (Dudd *et al.*, 1999; Regert, 2011). In contrast to ruminant fats, monogastric animal fats exhibit a simpler profile of fatty acids, as they generally lack the diversity of $C_{18:1}$ isomers and odd-chain fatty acids like C_{15} and C_{17}, as non-ruminants do not undergo ruminal biohydrogenation. Instead, their fatty acid composition more directly reflects their diet, with minimal microbial alteration in the gut. The absence of positional and geometrical isomers of $C_{18:1}$ and the scarcity of odd-chain fatty acids in non-ruminant fats are key indicators distinguishing them from ruminant fats, highlighting metabolic and digestive differences between these groups (Regert, 2011). Figure 4 shows that a single isomer of $C_{18:1}$ acid was detected, along with both linear $C_{15:0}$ and $C_{17:0}$ carbon acids. The absence of positional isomers indicates that the fat is derived from non-ruminant animals (Evershed *et al.*, 2002; Mukherjee *et al.*, 2008; Regert, 2011).

As noted by Malainey and Eerkens (Eerkens, 2005; Malainey, 1997), the ratio $(C_{15:0} + C_{17:0})/(C_{12:0} + C_{14:0} + C_{16:0} + C_{18:0})$ serves as a marker to differentiate fats from monogastric and ruminant animals. When this ratio exceeds 0.04, the organic residue may originate from ruminant fats. In this case, the measured value was 0.034, aligning with the presence of fats derived from monogastric animals.

Besides the strong animal contribution, the sample also shows traces of vegetable fats (Fig. 5c), as the presence of long-chain alkanes and alcohols with an even carbon number, from $C_{22:0}$ to $C_{30:0}$ (Evershed, 2008).

Amides are chemical compounds typically formed through the reaction of free fatty acids with ammonia or amino groups, a process that is





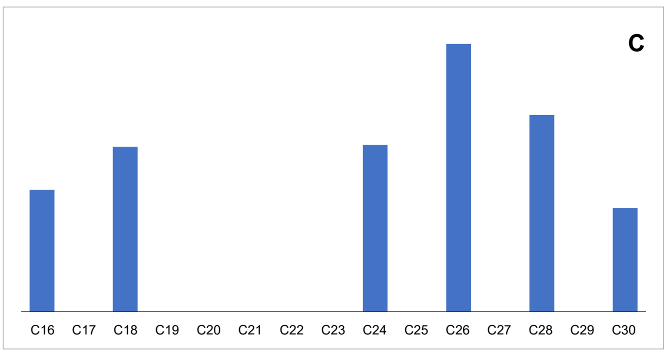


Fig. 5. The distribution of a) mono and diacylglycerols; b) *n*-alkanoic acids; c) *n*-alkanols. C*n*:*x* stands for carbon length *n* and number of unsaturation *x*.

Fig. 5. A distribuição de a) mono e diacilgliceróis; b) ácidos de cadeia linear; c) álcoois de cadeia linear. C*n*:*x* representa uma cadeia orgânica com *n* átomos de carbono, e *x* o grau de insaturação.

significantly enhanced at elevated temperatures. In archaeological contexts, this formation may occur during cooking or other thermal treatments involving animal fats. The thermal decomposition of triglycerides, combined with the availability of nitrogenous compounds, facilitates the generation of these amides, making their detection a reliable marker for heat-induced transformations in lipid residues. The presence of amides, such as hexadecanamide, octadecenamide and octadecanamide (Fig. 4), indicates that the fats were exposed to high temperatures during processing or use (Lejay *et al.*, 2016; Lejay *et al.*, 2019). The small peak of retene (Fig. 4), a polycyclic aromatic hydrocarbon (PAH) produced by the thermal degradation of resin acids, indicates the use of conifer wood or resin as fuel (Carpy & Marchand-Geneste, 2003; Marchand-Geneste & Carpy, 2003).

5. Discussion of the Results

The chromatographic analyses of the organic residue found at the bottom of the ceramic pot suggest that it contained a mixture predominantly made from animal meat, most likely from monogastric animals such as pigs or horses, with some traces of plant matter. The pot's contents appear to have been exposed to heat or cooked at high temperatures, possibly using biomass from conifer species, such as pine, for fuel.

The faunal remains available for analysis do not include any fragments from horses, while only a tooth from a suid was found, which is an anatomical part not associated with human consumption. Other faunal remains identified include those from sheep/goats and bovids, both ruminant or polygastric animals. The absence of horse remains and the presence of suid and ruminant remains suggests that while pig meat may have been involved in the preparation, the faunal evidence does not fully align with the chromatographic analysis indicating the presence of monogastric animal fats.

Interestingly, the presence of suids in funerary practices in other Iberian regions, such as the Southwestern Bronze Age in Portugal (e.g. Baptista *et al.*, 2012) and the Southeastern Bronze Age Culture of El Algar (e.g. Aranda Jiménez, Montón-Subias, 2011), suggests that pigs may have been involved in ritual or funerary contexts during this period, even though it was not possible to know if they were consumed in the immediate context of this particular pot.

Additionally, the external soot found on the bowl and base of the pot is suggestive of exposure to high temperatures, but it remains unclear whether this soot is a result of the cooking process or a result of the context in which the pot was deposited. This ambiguity makes it difficult to draw firm conclusions about the specific role of this pot in food preparation, although the presence of soot does indicate some form of thermal activity in its history.

6. Conclusions

The deposition of this ceramic container inside the funerary monument suggests the practice of commensality or offerings of cooked food in funerary contexts in Northwest Portugal, particularly during the second half of the 2nd millennium BC. This is further supported by the identification of organic residues in the pot deposited in the center of the funerary monument of Cova da Moura, which point to a preparation involving animal meat, likely from monogastric species, such as pigs, with traces of plant matter, and the exposure to high temperatures. This evidence points to a possible funerary practice linked to ritual cooking, commensality or offering practices to the deceased.

Additionally, the presence of mammal remains inside the monument, including one calcined fragment, raises important questions about the nature of the funerary activities. Although the calcined bone fragment likely represents a cremation deposit, it is not directly related to the pot's contents, as it does not exhibit signs of anthropogenic manipulation associated with food preparation. The whitish coloration of the bone fragment is consistent with calcination, which is typically linked to cremation rather than meat preparation. It would be valuable to date these faunal remains to determine whether they are contemporary with the pot deposit, which could help clarify the synchrony of activities and whether different forms of commensality existed during the funerary rites.

The chromatographic analysis suggests that the pot was not a funerary urn for cremation deposits, as previously suggested in reference to containers found at the site (Viana, 1955: 489). Instead, the contents of this specific pot indicate that it was used for the preparation of animal products, particularly meat, making it the first known occurrence of such a pot in a funerary context in Northwestern Portugal. This discovery may point to the practice of commensality rituals or offerings of prepared food as part of funerary practices, shedding new light on the nature of Bronze Age funerary behavior in this region.

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