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Fats, Fire and Bronze Age Funerary Rites: Organic Residue Analysis of Wide Horizontal Rim Vessels From Burial Contexts in Northwest Portugal

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ABSTRACT

This study presents the first GC-MS–based analyses of wide horizontal rim vessels with well-defined funerary contexts, from Middle Bronze Age Portugal (Quinta do Amorim 2 and Pego). Organic residues from two vessels revealed ruminant fats and plant oils, alongside molecular markers of heat exposure. These results provide direct chemical evidence for vessel contents and use, offering new insights into funerary practices.

1 | Introduction

1.1 | Background and Objectives

Wide horizontal rim (WHR) vessels are ceramic containers commonly found in funerary contexts dating to the regional Bronze Age, primarily from the Middle Bronze Age, across a broad region of Northwest Iberia. This encompasses Southern Galicia (Spain) and Northwestern Portugal, extending to the southern bank of the Douro River basin (Cardoso 1936; Ataíde and Teixeira 1940; Bettencourt 1999, 2011; Cruz and Gonçalves 1998; Sampaio 2014; Sampaio and Bettencourt 2014; Sampaio et al. 2014; Nonat et al. 2015, among others).

Since the early 20th century, dark remains on the surfaces of WHR vessels have been repeatedly reported, variously interpreted as "soot" or "organic contents" (Cardoso 1936; Ataíde and Teixeira 1940; Cruz and Gonçalves 1998; Bettencourt 1999, 2011; Sampaio 2014). Recently, this type of vessel was the subject of a comprehensive compilation by Nonat et al. (2015), which explores their stylistic evolution, functional significance and cultural context. However, despite the large number of published examples, the lack of chemical analysis to date (Nonat et al. 2015, 2, 140–143; Oliveira et al. 2022, 14) restricts insights into the vessels' original contents.

This article outlines the findings from the analysis of organic residues identified inside two WHR vessels from Bronze Age burial sites in Northwest Iberia. The vessels were recovered from two archaeological sites, one located at Quinta do Amorim 2, on the border between the parishes of São Victor and Gualtar, and the other located at Pego, within the parish of Arentim and Cunha, in the municipality and district of Braga (Portugal).

While the sample size is limited, this study represents the first chromatographic analysis of organic residues from WHR vessels within well-documented archaeological contexts. The entire handling and sampling protocol adhered to best archaeological practices (Oliveira 2023) to minimise potential postdepositional contamination, particularly from human interference. Exceptional conditions were maintained throughout the study to ensure strict control over the newly excavated samples, alongside a comprehensive understanding of their archaeological context. These factors represent a substantial improvement over previous studies, which relied solely on chromatographic analyses (GC-MS) conducted on a single sample from a WHR vessel lacking a defined archaeological context. That vessel was part of a collection and had been repeatedly handled prior to analysis (Oliveira et al. 2022; Vilaça et al. 2023). The absence of clear contextual information made it difficult to interpret the findings and limited the ability to link the identified organic residues to the social practices of the communities under study.

The results presented here allow us to propose new hypotheses regarding their possible function and their role in funerary rituals during this chronological and cultural period.

1.2 | Archaeological Contexts

The vessels under examination come from two distinct archaeological contexts, both located within the Ave River basin and situated approximately 13 km apart (Figure 1). These vessels were found in flat graves cut into the bedrock, with absolute chronologies placing them in the first half of the 2nd millennium BCE. As such, they can be assigned to the regional Middle Bronze Age (Sampaio 2014, 2017; Sampaio and Bettencourt 2014). One of the vessels comes from the southwest end of grave 9 of the Pego necropolis (Sampaio 2014, 261; Sampaio and Bettencourt 2014), while the other was found at the north-northwest end of grave 1 at Quinta do Amorim 2 (Sampaio 2014, 637; Sampaio et al. 2014).

The two graves were located on the south slopes of small hilltops, close to spaces with non-funerary functions marked by the presence of numerous pits dug into the rocky substrate, as well as post holes, hearths and other remains. These features were interpreted by Sampaio (2014) as most likely belonging to possible settlements.

The interpretation of grave 9 from the Pego necropolis as a burial context, despite the absence of skeletal remains, is based on a combination of taphonomic evidence and archaeological analysis. The taphonomic processes that affect the preservation of human remains may have led to the degradation or disappearance of the bones. However, the morphological and stratigraphic characteristics of the burial structure provide crucial information for its interpretation as a grave.

The central distribution of darker sediments in grave 9 is particularly interesting, as it is believed to be related to the decomposition of a body buried in a primary context, suggesting its placement in the grave shortly after death, without significant disturbance or secondary burial practices. The darker sediments may be an indicator of organic decomposition, a typical result of the breakdown of soft tissues in burial contexts. This evidence points towards the position of the body being in lateral decubitus (lying on its side), a common position for burials during the above-mentioned period (Sampaio 2014, 628; Sampaio and Bettencourt 2014, 54).

Furthermore, the presence of an intact WHR vessel in the grave provides an additional context for identifying it as a funerary object. Such vessels, often associated with burial practices, were typically used to hold food, drink or offerings for the deceased. The morphology and physical integrity of the vessel suggest it was part of a deliberate burial offering or ritual. Similar vessels have been found in other funerary contexts, such as the necropolis of Agra de Antas, located in Esposende (Portugal). This connection strengthens its interpretation as a grave, even in the absence of human remains, since similar practices were recorded in other burial sites (Ataíde and Teixeira 1940; Cruz and Gonçalves 1998; Bettencourt 1999, 2011). Thus, the absence of skeletal remains in this context does not diminish the archaeological interpretation, as the

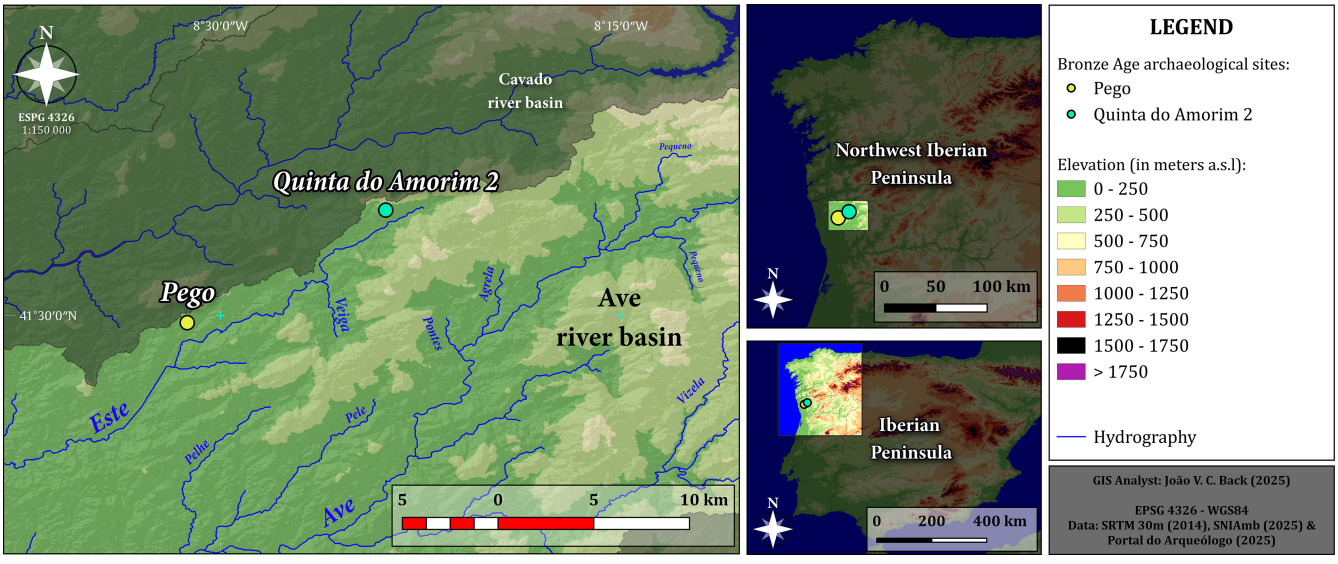


FIGURE 1 | Hypsometric map showing the locations of the Pego necropolis and the Quinta do Amorim 2 necropolis.

combination of sedimentary evidence, vessel morphology and comparisons with other funerary sites allows for a confident assessment of the site as a grave.

1.3 | Ceramic Vessels Under Investigation

The vessel from the south-west end of grave 9 at the Pego site has a height of 9.1 cm, the diameter of the mouth is between 10.0 and 10.3 cm, while the width of the flange varies between 3.4 and 3.9 cm (Sampaio 2014, 261). It was handmade with a coarse sandy paste and fired in an atmosphere of reduced oxygen. It possesses a vertical outer handle with a ribbon section, and the outer wall is polished while the inner wall is smoothed (Sampaio 2014, 636). The flange is decorated with metopes (*metopada* organisation), which comprise horizontal and vertical grooves, complemented by impressions at both ends (Sampaio 2014, 261) (Figure 2). According to the morphological table of ceramic vessels defined by Bettencourt (1999) this example is classified as form 13c, categorised as a WHR vessel. An estimation of the maximum volumetric capacity was conducted through 3D digital modelling, utilising the scale of the drawing published by Sampaio and Bettencourt (2014, 50). This approach gave an estimation of approximately 0.230 dm^3, determined by utilising *Blender 4.2* software, in accordance with the method employed by Tavella et al. (2021, 2022). This estimate was also confirmed using *Capacity*, a tool developed by the *Université Libre de Bruxelles* (Engels et al. 2009) and widely cited (Molina Vidal and Corredor 2018; Steiner and Bidgood 2018; Sukhanov 2018). The ^{14}C AMS analysis carried out on the organic residues from the inside of this container indicated a dating between the middle of the 18th century and the beginning of the 15th century BCE (2σ cal) (Sampaio and Bettencourt 2014, 53).

The vessel from the Quinta do Amorim 2 grave, located at the north-northwest end of the burial, has a maximum internal diameter of 13.5 cm, a flange width varying between 1.9 and 3.2 cm, and a height of 9.4 cm (Sampaio 2014, 426; Sampaio et al. 2014, 40). The fabrication method of this vessel shares similarities with the previously documented example, employing a sandy paste and a reduction firing technique. This vessel exhibits a smoothed finish on both sides and a slightly flattened concave base. However, a notable distinction emerges in the absence of a handle, which would have been vertical and ribbon-sectioned. In contrast to the previous example, this

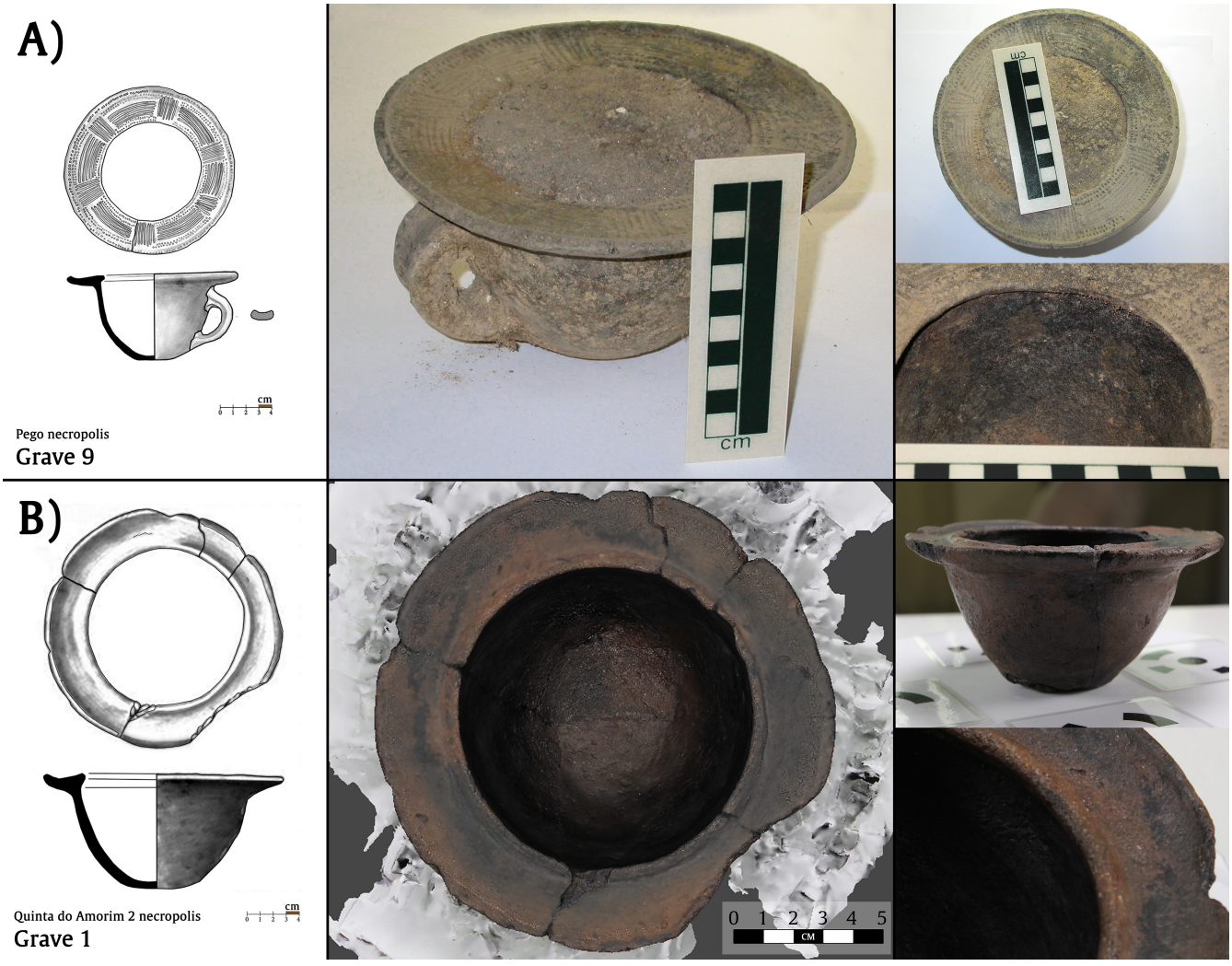
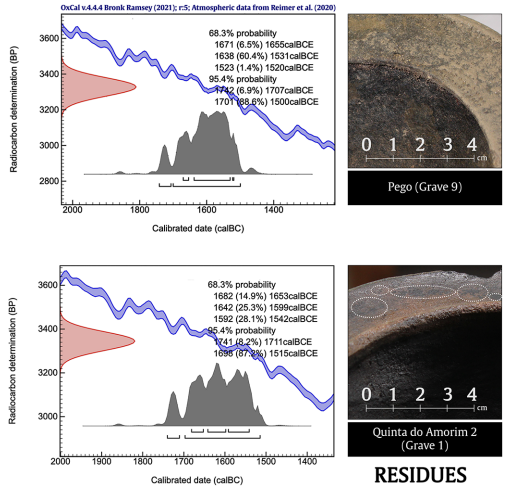


FIGURE 2 | Vessels from grave 9 of Pego (A) and grave 11 of Quinta do Amorim 2 (B). Drawings adapted from Sampaio et al. (2014, 40).



Site	Code	Age BP (AMS ^{14}C)	Calibrated age BCE (2σ)	Bibliography
Pego **Grave 9** -Vessel-2007.0442	AA89666	3328 ± 51	1742-1707 (6.9%) 1701-1500 (88.6%)	Sampaio (2014, p. 336); Sampaio & Bettencourt (2014, p. 53)
Quinta do Amorim 2 **Grave 1** -Vessel-2009.0765	AA89661	3345 ± 42	1741-1711 (8,2%) 1638-1511 (87,2%)	Sampaio et al. (2014, p. 40)

FIGURE 3 | Radiometric dating results (calibrated with *OxCal 4.4* using the *IntCal20* curve, based on Reimer et al. 2020).

vessel is devoid of any decoration (Sampaio 2014, 636; Sampaio et al. 2014, 40) (Figure 2).

Using the morphological table published by Bettencourt (1999), it is also a form 13c, categorised as a WHR vessel. 3D modelling in *Blender 4.2*, in conjunction with the Structure-from-Motion (SfM) close-range photogrammetry (carried out using *Agisoft Metashape Pro*), facilitated the estimation of a maximum volumetric capacity approaching 0.650 dm^3, as determined through the application of the method outlined by Tavella et al. (2021, 2022). This value is higher than the maximum volume obtained by *Capacity* (Engels et al. 2009), which is approximately 0.610 dm^3. The latter estimate is arguably less credible than the former because it does not take into account the variation in radius across different parts of the vase. ^{14}C AMS dating was also conducted on the organic residues of this vase, indicating an estimated period of use between the 18th century and the end of the 16th century BCE (2σ cal) (Sampaio et al. 2014, 40). It is therefore valid to consider both vessels as virtually contemporary (Figure 3).

2 | Materials and Methods

2.1 | Sampling Strategy and Handling Procedures

The sampling and handling of the ceramic materials for chromatographic analyses were carried out with particular care, following the best archaeological practices to prevent contamination and ensure the integrity of the samples (Oliveira 2023). The archaeological materials were handled with powder-free nitrile gloves and wrapped without washing in aluminium foil previously baked at 550°C for a minimum of 6 h to eliminate any residual organic compounds. Once the materials were properly wrapped and sealed, they were transported to the laboratory. The samples were kept in aluminium foil during transit, further ensuring that the materials did not contact any plastic containers or surfaces that might cause contamination. By adhering to these strict protocols, the archaeological team ensured that the ceramic samples remained uncontaminated, which is crucial for obtaining reliable and accurate results from the chromatographic analyses conducted on the samples.

2.2 | Analytical Procedures for Organic Residue Analysis

The vessels were found to be almost entirely intact and well-preserved, which allowed for the careful collection of a relatively large quantity of potsherd, approximately 1.5 g, from their interior surfaces using new, sterilised scalpel blades, following surface removal (~0.5 mm) to minimise exogenous contamination (e.g., soil and fingerprints). Thereafter, the material was ground in a clean agate mortar. The samples containing the organic residues were extracted in an ultrasonic bath (30 min—80 Hz) with 7 mL of a chloroform/methanol mixture (2:1). This method primarily targets free fatty acids and aligns with long-established practices in archaeological organic residue analysis. However, it is less effective for recovering glycerol-bound forms, which are generally more resistant to chemical and microbial degradation than free fatty acids. Although bound and intact acyl lipids can be hydrolysed prior to derivatisation, as noted by Evershed et al. (2002), this approach was not undertaken in the present study. Nevertheless, this limitation can be partially mitigated by comparative analysis of other molecular classes. The resulting extracts obtained were centrifuged (4500 rpm—1 h), filtered through PTFE filters with a pore size of 0.45 μm, and the supernatants were carefully decanted and dried using a gentle stream of nitrogen. The total lipid extracts were dissolved in *n*-hexane and derivatised with *N*,*O*-bis (trimethylsilyl)trifluoroacetamide containing 1% trimethylchlorosilane (BSTFA +1% TMCS). After removing the derivatising agent in excess under a gentle stream of nitrogen, the extract was redissolved in *n*-hexane and analysed by GC/MS.

2.3 | Instrumentation

Chromatographic analyses were performed using a Shimadzu GC2010 gas chromatograph coupled to a GCMS-QP2010 Plus Mass Spectrometer. The system was equipped with a Zebron ZB-5HT column (15 m, 0.25-mm internal diameter, 0.10-μm film thickness). Helium served as the carrier gas at a constant flow rate of 1.5 mL min^{-1}, the injection volume was 1 μL, and the injector temperature was set at 250°C.

The temperature programme was as follows:

a. Initial temperature of 50°C, held for 2 min.
b. Ramp from 50°C to 300°C at a rate of 10°C min^{-1}, held at 300°C for 5 min.
c. Further ramp from 300°C to 400°C at a rate of 10°C min^{-1}, held at 400°C for 5 min.
d. Total run time: 47 min.

The system was operated in full scan mode, scanning masses in the range of m/z 50–1090. The ion source temperature was maintained at 240°C, while the interface temperature was set at 280°C. Ionisation was performed using an electron impact at 70 eV. Compound identification was based on fragmentation patterns and comparison of the spectra obtained with the commercial libraries Wiley8 and Nist17.

3 | Results

3.1 | Chromatographic Findings: WHR Vessel From Pego Necropolis

The analysis of the chromatogram obtained (Figure 4) indicates a high abundance of lipids, characteristic of the degradation of triacylglycerols (TAGs) (Evershed et al. 2002; Romanus et al. 2007; Rosiak et al. 2019). The identified compounds include diacylglycerols (DAGs), such as 1,3-dipalmitin, 1,2-hexadecanoyloctadecanoylglycerol, 1,3-hexadecanoyloctadecanoyl glycerol and 1,3-distearin; monoacylglycerols (MAGs), such as 1-monopalmitin, 2-heptadecylglycerol, 1-heptadecylglycerol, 1-monostearin and 2,3-dihydroxypropyl icosanoate; as well as saturated fatty acids, which comprise an almost complete homologous series from $C_{9:0}$ to $C_{32:0}$, with a predominance of even-numbered carbon chains. Additionally, unsaturated fatty acids, specifically $C_{16:1}$ and $C_{18:1}$, were detected. Among the identified compounds, stearic acid ($C_{18:0}$) and palmitic acid ($C_{16:0}$) are the most abundant.

Although both palmitic (P) and stearic (S) acids are common in plant oils as well as in animal fats (Gunstone 1996, 72–73; Rosiak et al. 2019), their relative abundances can provide insights into their likely origins. Palmitic acid tends to be more prevalent in plant oils, whereas stearic acid is typically found in higher concentrations in animal fats, allowing for interpretations based on the ratio between the two acids (P/S) (Reber 2022; Romanus et al. 2007). In this context, the sample exhibits a P/S ratio of 0.302, suggesting a strong contribution from animal fats (Kimpe 2003; Romanus et al. 2007).

The same ratio can offer further aid in determining the provenance of fats. Isaksson (2000), for example, employed this ratio to differentiate fats from terrestrial animals and fish oils, considering a P/S ratio below 2.08 indicative of the former and a higher ratio associated with the latter. According to this author, the value observed in the Pego vessel sample from grave 9 strongly suggests a significant contribution from terrestrial animal fats. However, palmitic acid is more soluble and thus more susceptible to leaching, which can reduce its initial concentration due to post-depositional processes (Steele et al. 2010, 3479). Consequently, interpretations based solely on this parameter should be approached with caution.

Within the animal fat category, Dudd et al. (1999) and Hjulström et al. (2008) propose a distinction between fats from ruminant animals (e.g., cattle, sheep and goats) and those from monogastric animals (e.g., pigs) based on the ratio of the branched form of heptadecanoic acid to stearic acid ($C_{17\text{-branched}}/C_{18:0}$). According to Hjulström et al. (2008), a ratio above 0.020 indicates a greater contribution from ruminant fats, whereas values below 0.015 (Dudd et al. 1999) and below 0.0077 (Hjulström et al. 2008) suggest the presence of fats from monogastric animals. The sample studied exhibited a ratio of 0.0217, indicating a predominant contribution of fats from ruminant animals.

Eerkens (2005) employs a complementary approach to this assessment of the presence of ruminant fats by considering a broader ratio, specifically that of the sum of pentadecanoic and heptadecanoic acids, to the sum of dodecanoic, myristic, palmitic and stearic acids $(C_{15:0}+C_{17:0})/(C_{12:0}+C_{14:0}+C_{16:0}+C_{18:0})$. According to this method, values exceeding 0.04 are indicative of ruminant fats. The sample from grave 9 showed a value of 0.089, further supporting an origin associated with ruminant fats.

The presence of cholesterol, a sterol of animal origin (Evershed 1993; Kimpe et al. 2004; Baeten et al. 2013; Rosiak et al. 2019, 4; Oliveira et al. 2022; Reber 2022), along with two of its oxidation products cholesta-3,5-dien-7-one and cholesta-4,6-dien-3-one, further reinforces this hypothesis (Stott et al. 1999; Steele 2013; Pecci and Grassi 2016). Additionally, the detection of phytanic acid and the odd carbon-numbered fatty acids $C_{15:0}$, $C_{17:0}$ (both in branched and linear forms) and $C_{19:0}$ (Evershed et al. 2002, 664; Malainey 2011) may suggest the presence of ruminant fats. This interpretation is supported by the high abundance of branched-chain fatty acids, which are characteristic of ruminant fats, as well as by diagnostic ratios previously discussed, although these compounds may also derive from soil microbes. Similarly, phytanic acid, derived from phytol (a chlorophyll component), can result from both ruminant fat metabolism and aquatic food chains. However, it is notably abundant in ruminant fats due to microbial processing in the rumen. To address this ambiguity, phytanic acid is evaluated alongside a broader set of molecular markers and diagnostic ratios, allowing a more reliable distinction between aquatic and terrestrial sources.

The predominance of the five most abundant saturated fatty acids—$C_{18:0} > C_{16:0} > C_{17:0} > C_{20:0} > C_{15:0}$ (Figure 5)—strongly suggests that animal fats are well represented in the sample, to the detriment of other sources (Gunstone 1996; Rosiak et al. 2019). Additionally, the presence of various isomers of octadecenoic acid ($C_{18:1}$) may indicate ruminant fats, as monogastric animals typically exhibit only trace amounts of a single isomer of this acid (Evershed et al. 2002). These isomers arise from the biohydrogenation of unsaturated fats consumed in the diet of ruminants, which are subsequently metabolised in the rumen (Dudd and Evershed 1998; Evershed et al. 1997, 1999; Mayyas et al. 2017).

However, the detection of β-sitosterol and stigmasterol (phytosterols) clearly indicates the presence of plant oils, albeit to a lesser extent, which are associated with unsaturated fatty acids ($C_{16:1}$ and $C_{18:1}$) and long-chain saturated fatty acids. Thus, the

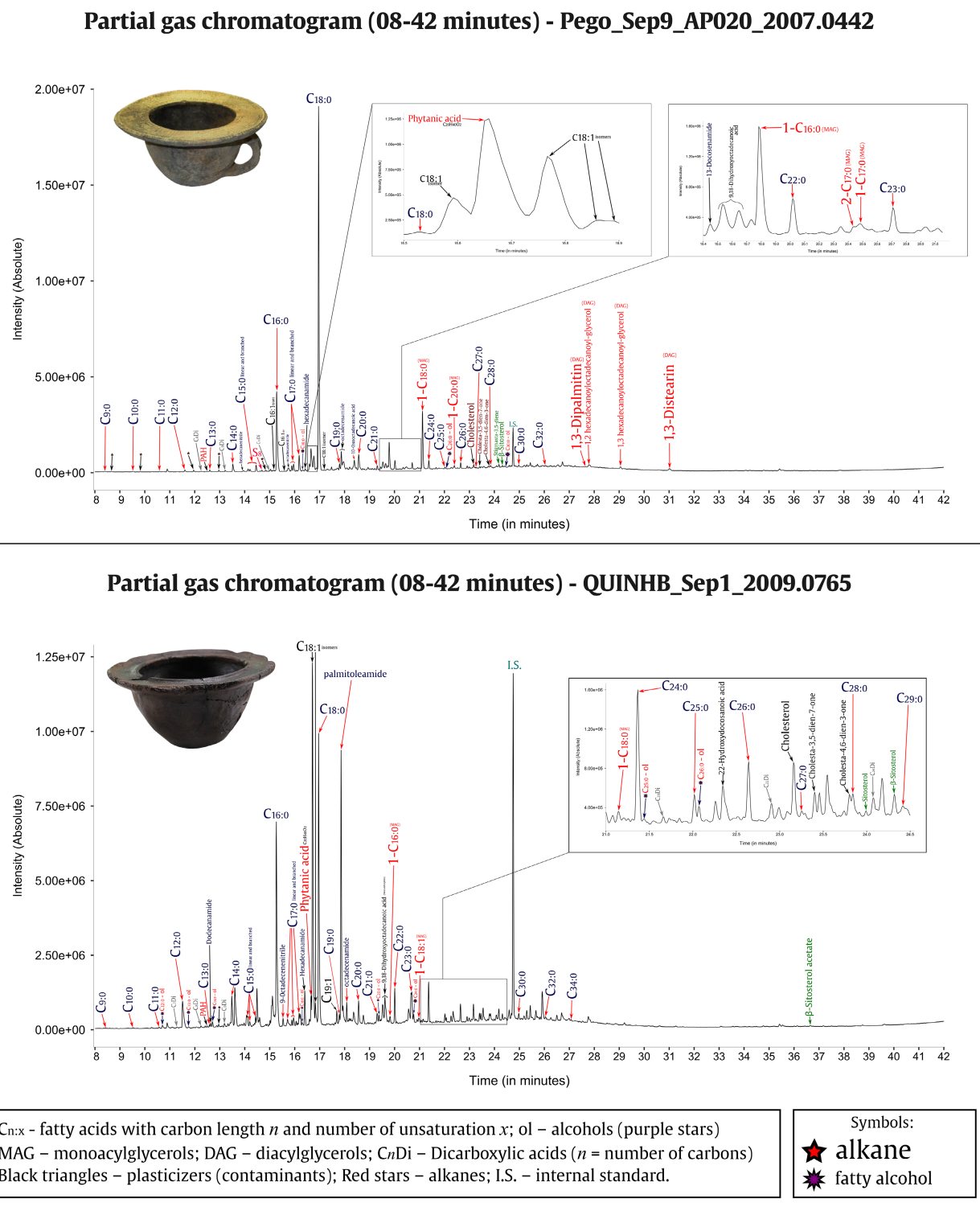


FIGURE 4 | Gas chromatograms from the total lipids extracts (TLE). Above: vessel from grave 9 (Pego). Below: vessel from grave 1 (Quinta do Amorim 2).

detection of octadecenoic acid ($C_{18:1}$), which is typically abundant in plant oils, is equally, or even more, likely to derive from plant contributions. Even so, the prevalence of animal fats is further corroborated by the comparative analysis of monoacylglycerols and diacylglycerols, with compounds derived from stearic acid ($C_{18:0}$) exhibiting higher concentrations than those formed by palmitic acid ($C_{16:0}$). Moreover, the absence of squalene—a biomarker of contemporary contamination, often introduced through direct contact with human skin (Steele 2013, 93; Reber 2022, 58)—as well as the absence of compounds such as homosalate, a component of cosmetics like sunscreens (Reber 2022, 50), suggests that the sample has not been affected by recent contamination.

The presence of the polycyclic aromatic hydrocarbon (PAH) anthracene, along with traces of external soot (inorganic carbon

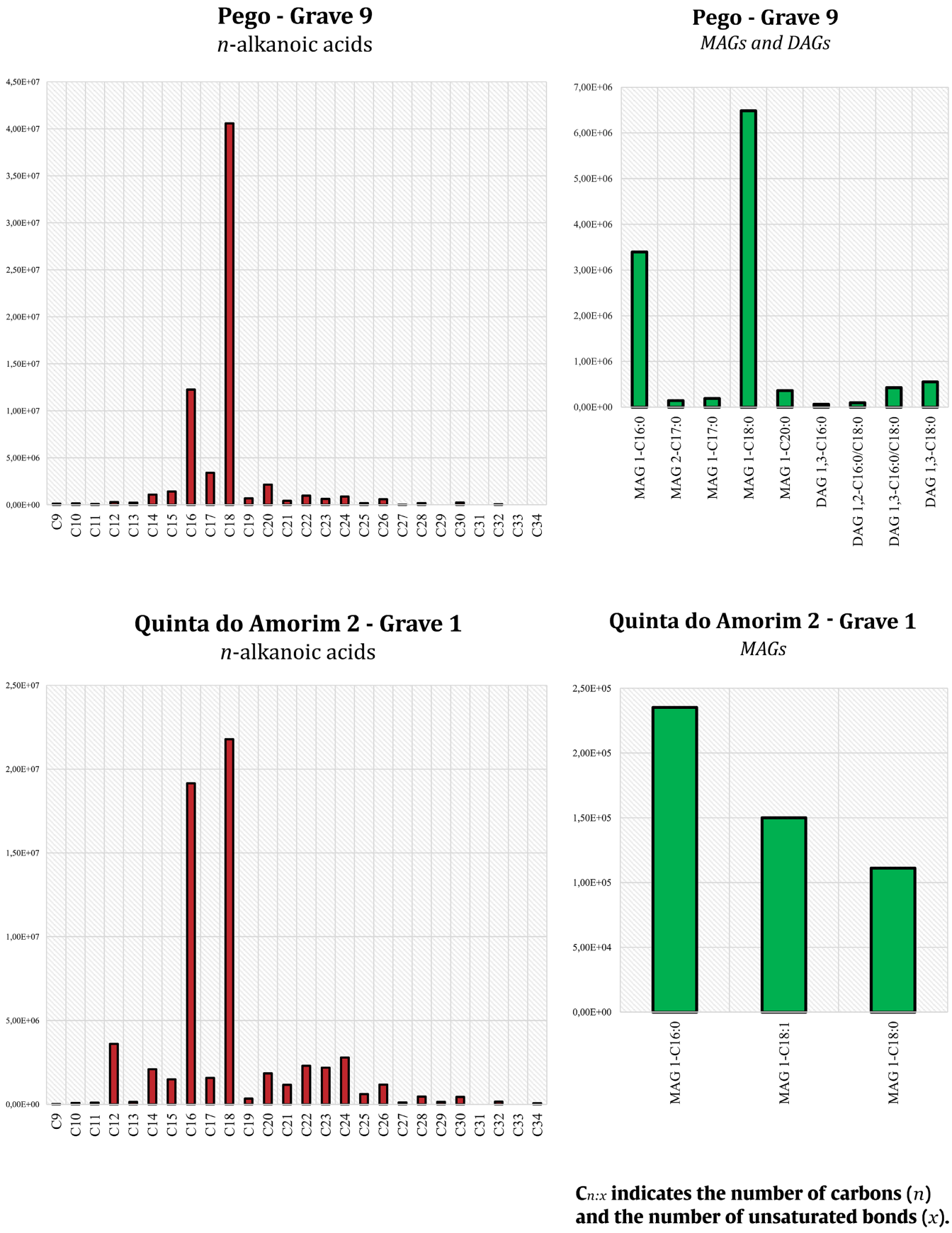


FIGURE 5 | Red: Distribution of *n*-alkanoic acids (red) and monoacylglycerols (MAGs) and diacylglycerols (DAGs) (green). Above: vessel from grave 9 (Pego). Below: vessel from grave 1 (Quinta do Amorim 2).

produced by the incomplete combustion of organic matter), suggests that the vessel and possibly its contents were exposed to or directly heated by fire. The occurrence of PAHs in archaeological samples has been associated with the incomplete combustion of organic matter, with their formation peaking between 400°C and 600°C (Poulain et al. 2016, 40). However, their detection in archaeological contexts does not necessarily imply temperatures were reached during food preparation, as PAHs can also form under less extreme conditions. The simultaneous detection of nitrogen-rich compounds, including amides (hexadecanamide, octadecenamide and docosenamide) and nitriles (hexadecanenitrile and octadecanenitrile), though less conclusive, may also point to thermal processing, provided that contamination was carefully controlled, specifically through the avoidance of plastic contact and careful handling of aluminum foil. Nonetheless, the PAHs remain the most convincing evidence of burning or fire, whereas nitriles and amides offer more tentative support.

Supporting this evidence, recent studies by Rasmussen et al. (2022, 14–16) on organic residues in ceramics from the Christmas Cave (Judean Desert) across different periods indicate that the presence of dicarboxylic acids, hydroxy acids and unsaturated isomers (*cis* and *trans*) of octadecenoic acid ($C_{18:1}$), along with 10-oxo-octadecanoic acid, is indicative of the oxidation of organic contents during heating processes, such as cooking, in the presence of oxygen. However, the authors acknowledge that these compounds can also result from degradation processes occurring post-burial due to microbial activity (Rasmussen et al. 2022, 16). It is noteworthy that, from a stratigraphical perspective, previous studies of the concentration of charcoal inside grave 9 did not provide sufficient evidence for the existence of a fire (Sampaio 2014). This lends credibility to the interpretative hypothesis that '[...] something [...] was burnt inside [...] [this structure] before or during the funeral ceremonies that took place there' (Sampaio 2014, 280). The findings from the GC-MS analysis of the vessel from this context align with this interpretation, reinforcing the possibility of fire-related activities associated with the burial.

3.2 | Chromatographic Findings: WHR Vessel From Quinta Do Amorim 2 Necropolis

The chromatographic results obtained for this vessel (Figure 4) clearly distinguish it from the previously described example from the Pego site. While a significant portion of the identified organic residues is similar in terms of suggesting the presence of triacylglycerol (TAG) degradation products, the absence of detectable peaks corresponding to diacylglycerols (DAGs) indicates a more advanced degree of chemical and/or bacterial degradation compared to the Pego sample. Despite this, the presence of degraded fats can be confirmed. Notably, the detection of monoacylglycerols (MAGs), including 1-monopalmitin, 1-monooleoylglycerol and 1-monostearin, is significant. Furthermore, an almost complete homologous series of saturated fatty acids (ranging from $C_{9:0}$ to $C_{34:0}$) was identified (Figure 5), alongside peaks corresponding to monounsaturated fatty acids, such as $C_{18:1}$ and $C_{19:1}$. These findings highlight the compositional differences between the two vessels while reinforcing the evidence for lipid degradation processes.

This analysis identifies the most significant chromatographic peaks among the saturated fatty acids as stearic acid ($C_{18:0}$) and palmitic acid ($C_{16:0}$). However, the most abundant fatty acid detected in the sample is the monounsaturated oleic acid ($C_{18:1}$). The relatively small difference between the concentrations of $C_{16:0}$ and $C_{18:0}$, combined with the high abundance of monounsaturated fatty acids ($C_{18:1}$ and $C_{19:1}$), indicates a distinct composition compared to the sample from the Pego necropolis.

The presence of phytosterols such as stigmasterol, β-sitosterol and β-sitosterol acetate (Colombini and Modugno 2009, 197; Romanus et al. 2009; Poulain et al. 2016, 39; Irto et al. 2022; Reber 2022), along with the significant concentration of oleic acid ($C_{18:1}$), suggests a substantial contribution from plant-derived oils. Additionally, the identification of long-chain alcohols ($C_{22\text{-ol}}$–$C_{32\text{-ol}}$), including docosanol ($C_{22\text{-ol}}$), tetracosanol ($C_{24\text{-ol}}$) and pentacosanol ($C_{25\text{-ol}}$), in association with a high concentration of even-numbered fatty acids, supports the presence of plant waxes (Colombini and Modugno 2009, 11). Conversely, the detection of cholesterol (Evershed 1993; Kimpe et al. 2004; Baeten et al. 2013; Oliveira et al. 2022; Reber 2022) and its oxidation products—cholesta-3,5-dien-7-one and cholesta-4,6-dien-3-one (Stott et al. 1999; Steele 2013; Pecci and Grassi 2016)—alongside the higher concentrations of $C_{18:0}$ relative to $C_{16:0}$, indicate the co-presence of animal fats. The calculated P/S ratio ($C_{16:0}/C_{18:0}$) of 0.879, falling below the 2.08 threshold (Isaksson 2000), suggests a predominance of terrestrial animal fats rather than those of aquatic origin.

The detection of odd branched-chain fatty acids, including $C_{15:0}$, $C_{17:0}$ and $C_{19:0}$, along with the prominent peak of phytanic acid, further indicates an origin derived from animal sources. The ratio of branched $C_{17:0}$ to $C_{18:0}$, calculated at 0.057, exceeds the thresholds proposed by Dudd et al. (1999), 0.015, and Hjulström et al. (2008), 0.0077, for monogastric animals. Additionally, the value surpasses the 0.020 limit indicative of ruminant fats (Hjulström et al. 2008), reinforcing this interpretation.

The detection of anthracene, a PAH, alongside macroscopic soot deposits supports the interpretation of exposure to fire (Poulain et al. 2016, 40; Reber 2022, 94–95). Additionally, as previously discussed, the simultaneous presence of nitrogen-rich compounds, including nitriles (e.g., 9-octadecenitrile) and amides (e.g., hexadecanamide, palmitoleamide and octadecenamide) suggests exposure to elevated temperatures (Lejay et al. 2016, 2019; Oliveira et al. 2022, 2024). However, alternative sources such as algae or aquatic plants may also contribute to the presence of some of these compounds (Dembitsky et al. 2000).

The detection of $C_{19:1}$, a rare monounsaturated fatty acid in archaeological contexts, presents interpretative challenges due to its high abundance in the sample. This compound, along with $C_{17:1}$ acid (absent in the sample), is characteristic of fish species such as carp and catfish (Rasoarahona et al. 2004, 2008), and other marine species, including cuttlefish, crabs, limpets, shrimps and sponges (Ando and Nozaki 2007; Le Bihan et al. 2007; Kawashima et al. 2008; Denis et al. 2009; Baeten et al. 2013). Studies by Baeten et al. (2013) have reported previous findings from analyses of fish remains from Roman and predynastic Egyptian contexts, suggesting that these fatty acids are indicators of aquatic food sources. Similarly, Poulain et al. (2016,

37) employed this argument when analyzing organic residues from 16th- and 17th-century ceramics at Middelburg Castle (Belgium). The combined presence of nitrogen-rich compounds and specific fatty acids such as $C_{19:1}$ thus preliminarily suggests the presence of aquatic resources, including fish and seafood, in the analysed sample. The presence of soot (Sampaio 2014, 636) and anthracene (Poulain et al. 2016) further supports the hypothesis of heat exposure, possibly related to food processing or cooking.

Given this context, the phytanic acid present in the Quinta do Amorim 2 sample may not be solely attributable to ruminant fat (Heron et al. 2016), and the contribution of fish oils must be considered (Hansel et al. 2004; Hansel and Evershed 2009; Araújo 2015). The River Este, for example, is located less than 1 km away from the site.

Phytanic acid can originate from aquatic resources, as it is derived from the degradation of phytol, a chlorophyll constituent found in algae (Schwender et al. 1997), and it is also naturally present in fish oils (Araújo 2015). Therefore, the assumption that phytanic acid serves exclusively as a biomarker for ruminant fats may be overly restrictive, particularly given its potential presence in freshwater and marine environments.

Although the interpretation of an aquatic origin for the lipids found in this sample is less plausible based on several key factors:

a. Absence of specific markers for aquatic resources: the absence of ω-(o-alkylphenyl) alkanoic acids (APAAs), which are typically found in fish and seafood lipids, notably heat-altered ones (Cramp and Evershed 2014; Hansel et al. 2004; Bondetti 2021; Reber 2022; Admiraal et al. 2025), significantly weakens the case for an aquatic origin. Additionally, the lack of specific omega acids—such as erucic acid ($C_{22:1}$, *cis*-9, ω9), myristoleic acid ($C_{14:1}$, *cis*-9, ω7) and palmitoleic acid ($C_{16:1}$, *trans*-9, ω7), which are characteristic of fish and fish preparations (Hansel et al. 2004)—further diminishes the likelihood that the sample originates from aquatic sources.

b. Fatty acids profiles: the presence of $C_{19:1}$ (nonadecenoic acid), without the corresponding $C_{17:1}$ (heptadecenoic acid), reduces the probability of an aquatic origin, as these fatty acids typically co-occur in fish and seafood. Furthermore, the profile reveals a greater prevalence of stearic acid ($C_{18:0}$) compared with palmitic acid ($C_{16:0}$). This is contrary to the typical pattern seen in freshwater and saltwater fish, where palmitic acid is usually more abundant than stearic acid (Regert 2011, 190).

c. Diagnostic ratios: the ratio of $C_{16:0}$ to $C_{18:0}$, or the P/S ratio, is found to be 0.879—a value less than 1. This is consistent with the expected lipid composition of ruminant fats rather than aquatic resources. In general, aquatic fats tend to have a different P/S ratio, with $C_{16:0}$ often more prominent than $C_{18:0}$ in aquatic species (Regert 2011, 190). Additionally, the ratio of the sum of pentadecanoic and heptadecanoic acids, to the sum of dodecanoic, myristic, palmitic and stearic acids $(C_{15:0}+C_{17:0})/(C_{12:0}+C_{14:0}+C_{16:0}+C_{18:0})$ was 0.065, exceeding the 0.04 threshold adopted by Eerkens (2005) as indicative of ruminant fats. Therefore, the diagnostic ratios further reinforce the hypothesis that the animal fats in the sample are of terrestrial origin, most likely from ruminants.

In conclusion, the lack of aquatic-specific markers, the fatty acid profile and diagnostic ratios all point toward a terrestrial origin for the fats in the sample, with a significant contribution from ruminant animals rather than fish or other aquatic sources. This is further supported by the previously discussed findings, such as the presence of ruminant-associated fatty acids and the P/S ratio, which align with the interpretation of a predominantly animal-based fat composition, specifically from terrestrial animals.

4 | Discussion

4.1 | Interpretation of Organic Residues

The results of the analyses conducted on the two ceramic vessels, which date to approximately the same period, suggest that both WHR vessels, despite exhibiting distinct features in terms of decoration, maximum volumetric capacity and depositional context, contained animal-derived fats and plant oils, albeit in varying proportions. These observations can most likely be attributed to some cooking practices carried out during the funerary ceremonies. Among the identified animal fats, those derived from ruminants (e.g., cattle, sheep, goats and deer) were the most prevalent in the extracted compounds from both samples. This suggests that in societies increasingly reliant on agro-sylvopastoral practices, these animals played not only a crucial role in daily subsistence but also held significance in funerary rituals. However, the absence of data from ancient DNA analysis or advanced mass spectrometry techniques—such as gas chromatography-combustion-isotope ratio mass spectrometry (GC-C-IRMS), Nano-electrospray Ionisation time-of-flight mass spectrometry (nESI-TOF-MS)—or from other approaches, including compound-specific isotope analysis (CSIA), limits the ability to further discriminate between organic residues (Craig et al. 2020, 79–83) from different species, including those within the ruminant category.

The high acidity of the soils in the region (Cardoso et al. 1973; Inácio et al. 2008; Faria et al. 2023), which hinders the preservation of bone remains (Denys 2002, 477), further limits the ability to identify the specific species within the suborder *Ruminantia*. This preservation constraint makes it difficult to definitively exclude the use of fats from wild ruminants, such as red deer (*Cervus elaphus*). Notably, a small number of skeletal remains from this species have been recovered at other Bronze Age archaeological sites in northern Portugal (Cardoso et al. 1998; Bettencourt 1999, 1225; Senna-Martinez and Luís 2009, 75).

4.2 | Cultural and Functional Implication of Ceramic Use

The contents of the vessels appear to have been partially or entirely spilled during the funerary rites (Figure 6), as evidenced by the distribution of organic residues both inside and outside the

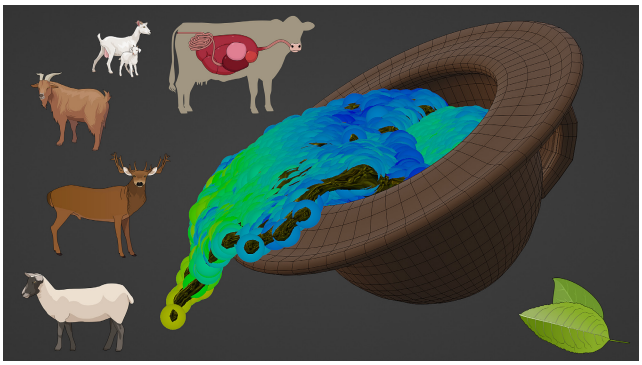


FIGURE 6 | Three-dimensional simulation of the action that could explain the spatial distribution of organic contents inside and outside the vessels, recurrently on the side opposite the handle. Developed in *Blender 4.2* by J.V.B. (2024). Animal images from Science Figures (open license, accessed from https://sciencefigures.org on 17 March 2025).

vessels, particularly on the outer surfaces opposite the handle. Additionally, the organic composition of the residues suggests notable differences between the two vessels. The vessel from the Pego exhibited a predominant contribution of ruminant animal fats, with a minor yet detectable presence of plant-derived oils. In contrast, the vessel from the Quinta do Amorim 2 contained traces of animal fats, likely from ruminants, alongside substantial quantities of plant residues. This distinction may reflect variations in ritual practices or differences in food preferences associated with each funerary context.

These particularities observed in the contents of the two vessels raise several questions regarding the factors that may explain these differences. Notably, one vessel featured decoration, while the other, despite its larger size, was completely undecorated. This contrast suggests that each vessel may have conveyed specific symbolic messages, whether through its decoration, size or contents.

What was the significance of these distinctions? Could they be linked to the identity of the buried individual, reflecting their role, status or social prestige within the community? Might they be associated with differences in sex and/or age? Alternatively, could they indicate variations in funerary practices between populations from geographically distant areas, pointing to distinct cultural traditions? It is important to emphasise that the exploratory nature of the residue analysis does not support broad generalisations. The results from only two specimens are insufficient to claim that undecorated pots were typically used for plant-based contents while decorated ones held meat residues. Such interpretations are best pursued through large-scale statistical analysis of WHR vessels recovered from burial contexts. Nonetheless, this study offers a preliminary contribution to a broader research initiative aimed at investigating the use of WHR vessels in funerary settings. It is anticipated that future work will incorporate a larger number of samples, enabling statistically robust conclusions.

Conversely, it is vital to note that the two sites are approximately 13 km apart, suggesting that while they may have shared a broader funerary tradition, characterised by using flat graves and WHR vessels, they could also have exhibited distinct intrinsic customs. Additionally, variations in funerary practices may not be limited to different sites but could also occur within distinct grave clusters in the same necropolis, as highlighted by Sampaio and Bettencourt (2014) in relation to the Pego necropolis. Addressing these questions will require further studies and a larger number of well-contextualised samples to refine our understanding of these ritual variations.

5 | Conclusions

In comparison to other similar studies analysing organic residues in WHR vessels (Gonçalves et al. 2010), which employed techniques like Fourier transform infrared spectroscopy (FTIR), this study represents a significant technical advance through the adoption of GC-MS. While FTIR provides only general descriptions of the chemical properties of broad compound classes, often making precise identification of specific origins challenging, chromatography allows for the detailed characterisation of the organic compounds present in the samples. This methodological leap offers a more robust foundation for interpreting organic residues in archaeological contexts (Evershed 2008, 895–896).

This study also allowed for a direct comparison of the results with those from a previous analysis of a WHR vessel from the Sousa de Oliveira Foundation (São Miguel Island, Azores), archaeologically uncontextualised, which followed exactly the same extraction method. In that vessel, traces of fats were detected, including degradation products of animal and plant-derived triacylglycerols (TAGs), as well as cholesterol, phytanic acid, linear fatty acids ($C_{15:0}$ and $C_{17:0}$) and their branched equivalents (Oliveira et al. 2022; Vilaça et al. 2023). This evidence suggests that the vessel likely contained fats from ruminant animals. Additionally, nitrogen-containing compounds, such as urea, oleamide and anthracene, were identified. Oliveira et al. (2022) and Vilaça et al. (2023) proposed that these compounds might indicate a mixture of ruminant animal fats and plant oils that had been exposed to high temperatures, possibly during meat cooking. It is noteworthy that many of the same compounds were identified in the samples analysed in this study.

A comparison of the results from this study with those of the aforementioned study reveals a notable similarity between the vessel from the Vila Franca do Campo Museum, which is richly decorated (Oliveira et al. 2022, 6–8), and the vessel from grave 9 (Pego necropolis), which also features decoration on the rim. This similarity is supported by the comparative concentrations of free fatty acids and the absence of a significant amount of oleic acid, which is clearly present in the vessel from grave 1 (Quinta do Amorim 2 necropolis). These findings suggest a lower presence of plant oils in the Pego vessel, thereby reinforcing the earlier results to some extent.

Similar to the results presented here, the vessel of indeterminate context also exhibited chemical markers indicative of fat burning, supporting the significant recurrence of soot traces observed macroscopically on these vessels, as documented by various authors (Cardoso 1936; Cruz and Gonçalves 1998; Bettencourt 1999, 2011; Sampaio 2014, 2017, among others). This phenomenon will be explored in future research, as this study also suggests that much of the dark traces adhering to the ceramic walls of these vessels could be either organic

(e.g., degraded organic residues) or inorganic (e.g., soot). Such duality underscores the importance of conducting a more detailed and differentiated analysis of the residues associated with these archaeological contexts.

Finally, although this study is pioneering, the limited sample size prevents robust generalisations about the variability, or potential uniformity, in the use of WHR vessels and the factors underlying these patterns. Expanding the sample size is therefore essential to fully explore the complexity of WHR vessel use in Bronze Age funerary contexts.

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Data Availability Statement

The data that support the findings of this study are available within the article and in the Supporting Information.

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Supporting Information

Additional supporting information can be found online in the Supporting Information section. **Data S1:** Supporting Information.