Journal Pre-proof

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PII: S1040-6182(20)30058-6

DOI: https://doi.org/10.1016/j.quaint.2020.02.016

Reference: JQI 8148

To appear in: *Quaternary International*

Received Date: 17 September 2019

Revised Date: 10 February 2020

Accepted Date: 12 February 2020

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Abstract:

This article presents the results of the first dedicated study of organic residues in Portugal, extracted from pottery excavated from Anta 1 de Val da Laje passage grave. We fully exploit the organic residue extract, to obtain information regarding the diet of the people and their relationship with the environment, the socio-economic aspects of an otherwise elusive society, and we also used a new methodology to obtain direct absolute dates for the pottery, the residue extract being the only datable organic material from the site. Our results suggest a community with diet based on terrestrial resources, that was fully benefitting from a range of domestic animals including their secondary products. We present the first direct evidence of not only meat consumption, but also milk and dairy production in Iberia. The compound specific radiocarbon dating methodology, opens a door to possibilities for investigating otherwise poorly dated archaeological phenomena in the Iberian Peninsula.

Keywords: Portugal, Neolithic, megalithic monument, pottery, organic residue analyses, pottery direct dating

1. Introduction

Passage graves are part of the wide range of megalithic monuments scattered all-over the Atlantic façade of Europe, from South Portugal to Ireland and Scandinavia. They represent a local man-made Neolithic phenomena, which developed roughly contemporaneously in several different regions of Europe (Renfrew, 1976; Daniel, 1980; Scarre et al., 2003; Thomas, 2012). These structures were built from massive stone blocks and served as burial grounds for members of the community, as well as for gatherings and rituals associated with the burial ceremony or other occasions. Not only do they represent a link between the people and their ancestors, but these long-lasting structures, importantly, also served as anchors to the landscape for otherwise (semi)mobile communities (Scarre et al., 2011).

Following the early Neolithic phase in Iberia, megalithic monuments began to appear throughout the peninsula, with notably higher concentrations evident in the western and southwestern hinterland (the landscape around the Tagus and Guadiana rivers). Obtaining organic materials for absolute dating of this structures is often impossible. The major part of the Iberian interior belongs to the Hesperic Massif, a geological formation characterized by acidic soils, which prevents the preservation of organic material, e.g. bone, suitable for ^{14}C measurements. Attempts to date paint samples from passage graves with preserved wall decoration have had some success (e.g. Steelman et al., 2005). Dates from luminescence have been obtained for some contexts as well (e.g. Burbidge *et al.* 2014). Even though there are regional differences, the proposed chronological frame for the construction of passage graves in Iberia can be set between 4300 calBC when the earliest appeared, and the beginning of the 3rd millennium BC, when they were no longer built. Some of the graves were occasionally visited in later periods, but their construction was obviously not required any longer and the intensity of activities around the monuments drastically decreased. The relationship between chronology and trends in style is not straightforward, even if complexification of the monuments seems to occur in several cases, after c. 3.500 calBC.

The question of the ethnogenesis of the people who built passage graves is part of the wider debate on the neolithisation and the introduction of farming in the Iberian Peninsula (Carvalho et al., 2016). Most convincingly, they are attributed to the Neolithic groups occupying the hilly areas along the large rivers of the interior. These groups would have had a more complex ethno-genetic route than the coastal population of Franco-Iberian Cardial descent. Recent genetic studies reveal that the input of local hunter-gatherer groups was more significant in the genetic ancestry of the Neolithic population of the interior than in the costal areas (Szécsényi-Nagy et al., 2017). It is reasonable to suggest that this hunter-gatherer genetic inheritance was accompanied by some elements of culture and local knowledge, and had some manifestation in the economy, social structure, mobility and subsistence patterns. In this context, on a micro-scale we can observe the megalithic zones of the peninsula as an impact (*sensu* Kinnes 1982) or contact zone (Zvelebil, 2001) between farmers and hunter-fisher-gatherers, and the appearance of the passage graves as part of the process of interaction involving (at least) two interacting traditions.

Even though the megalithic monuments of Portugal have been investigated by archaeologists for more than a century, we still know very little today about any aspect other than the funerary traditions of the people who built them (Kalb, 1980; Scarre and Oosterbeek, 2010). This is due to the near invisibility of their settlements archaeologically, probably a result of a semi-nomadic, seasonal life pattern. In an attempt to come closer to these elusive societies and provide a glimpse of their subsistence base and use of pottery, we present the results from biomolecular and isotopic analyses of lipid residues from selected pottery samples. These types of analyses provide information on the subsistence pattern of the people and their relationship with the environment, the advance of domestic animal exploitation, as well as an insight to the roles of specific pottery types and culinary practices (Roffet-Salque et al., 2018). In order to provide a better chronological orientation about our assemblage, we undertook compound-specific radiocarbon analysis (CSRA) of fatty acids from two potsherds (Casanova et al., 2018; *in review*), a new approach in absolute chronology, providing direct dates for the use of pottery vessels (Casanova et al., *in review*).

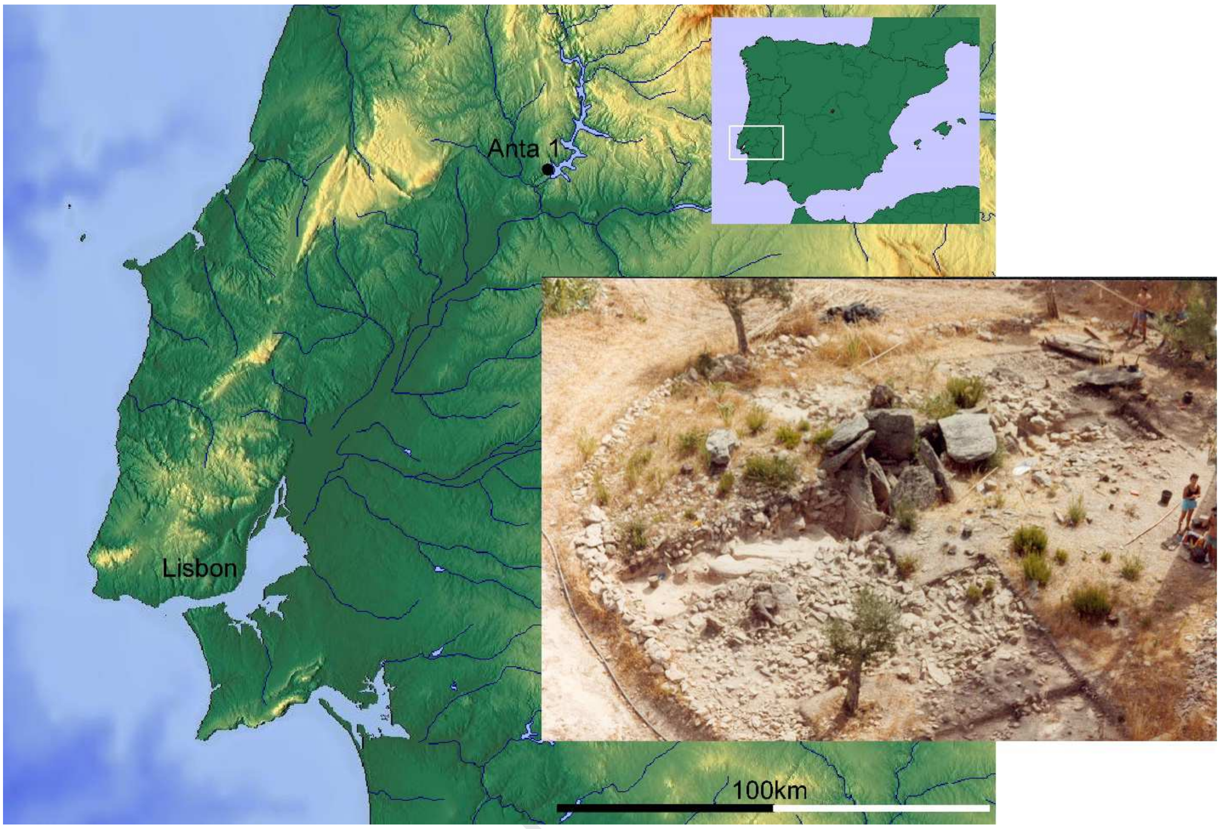

Figure 1. Anta 1 in the Iberian Peninsula and an aerial view of the excavations in 1991 (base map source: maps-for-free.com; photo: IPT archives)

1.1 Site description

We focus on a site from central Portugal, the western fringes of the passage grave distribution area on the peninsula. Anta 1 de Val da Laje (hereafter referred to as Anta 1) is part of a cluster of similar monuments on the right bank of the Zêzere River, a tributary of the Tagus. Today it stands on a hill on the bank of an artificial dam, about 10 km south-east of Tomar (Fig. 1). The grave has been excavated during several campaigns conducted over the past 30 years, the last of which started in 2017 and is still going on. The material used in this study derives from the excavations conducted in the early 1990's (Drewett et al., 1991; Cruz, 1997; Oosterbeek, 1997).

The structure as it is preserved today, is an oval mound with 10 m maximum diameter, containing a passage grave with pentagonal chamber and a short corridor, measuring

5.8 m in length, a maximum width of 2.2 m and height of 0.8 m; the chamber is 1.4 m high (Figs 2 and 3). Based on architectural and artefactual analogies, Anta 1 has been previously associated with Poço da Gateira and Gorginos 2, two passage graves in Alentejo dated with thermoluminescence to ca. 5th millennium BC and is considered to represent the early stages of building megalithic monuments in Portugal (Leisner and Leisner, 1951; Whittle and Arnaud, 1975).

The stratigraphy of the site consists of three archaeological layers A, B and C (Figs 2 and 3). At the base of the archaeological layers is layer D with no archaeological finds, and A is a disturbed layer, containing remains dating from the Copper Age until recent history. In this investigation we focussed on layers C and B, the former representing the phase when the monument was built, the latter the period of its most intensive use. During the later phase (B) some reconstruction efforts were made and the mound above the dolmen was reformed (Oosterbeek, 1994). A stratigraphic issue that still needs to be clarified is the relationship between layer C as a stratigraphic unit and the initial construction of the passage grave as an historical event. Refitting pieces of pottery were found on opposite sides of the chamber orthostates. There is a possibility that the location was used before the construction of the monument, during which period layer C (at least part of it) was formed. According to the excavated material, the two layers represent a prolonged period of time, spanning over the later Neolithic and possibly the beginning of the Copper Age. As part of this study, an exact chronological determination was provided by the new technique of compound-specific radiocarbon dating (see section 2.1).

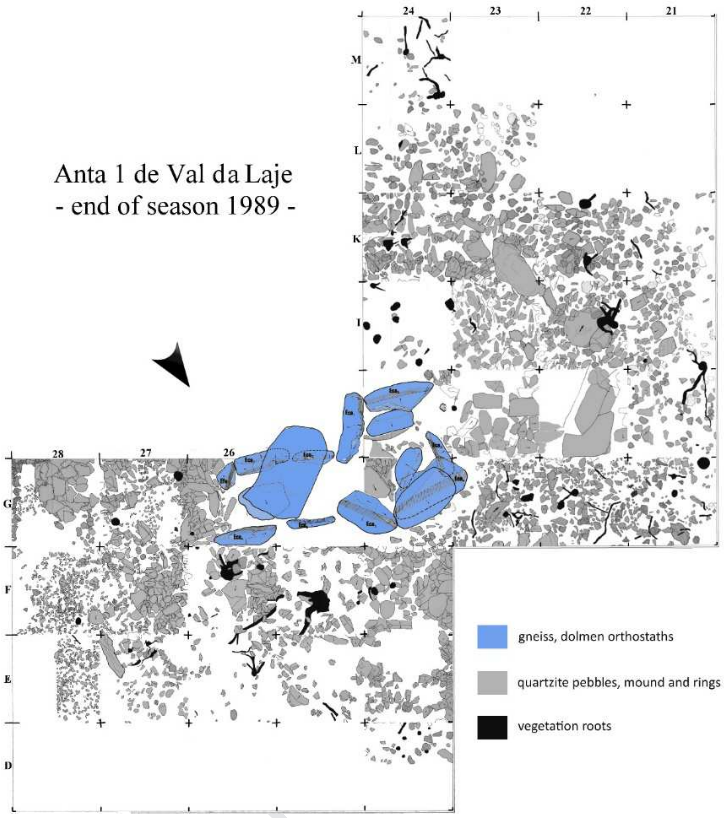

Figure 2. Plan of the Anta 1 mound and passage grave (modified after Ana Cruz 2004)

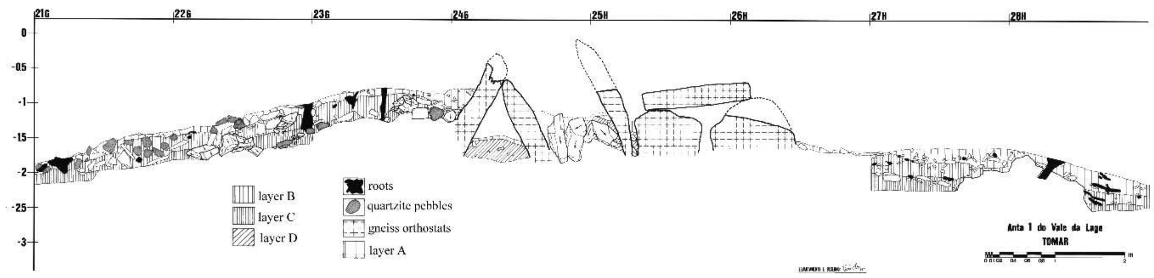

Figure 3. NW-SE section of the Anta 1 passage grave (modified original section drawing after Félix 1993)

1.2 Study materials

The pottery subject of this study was excavated between 1990 and 1992 and is stored at the Transdisciplinary Centre of Archaeologies (former Prehistory Centre) of the Polytechnic Institute of Tomar, Portugal. Following earlier partial studies (Oosterbeek, 1997; Oosterbeek et al., 2006; Fuying, 2008), the complete collection of 2,246 pottery units, 90% of which come from layer B, has been assessed for basic techno-typological characteristics. Being highly fragmented, typological assessment was limited to only 7% of the assemblage (149 fragments). Knowing the typological profile of the collection can help associating shapes and vessel categories with food commodities, and therefore can provide insights into the socio-economic aspects of pottery production and food preparation.

Four general categories of pottery containers were identified in the Anta 1 collection. **Dishes** are open vessels, rims represent the widest part of the body; they are among the most frequent shapes in this assemblage and, by modern analogy, may have been used for the serving, consumption and presentation of food. Based on relative depth and profile line, four types are recognized (SI 2, fig. S2.1): conical (D1), carinated (D2) and hemispherical dishes (D3) and shallow plates (D4). **Bowls**, including cups, have constricted orifices, shoulders being the widest part of the vessel (SI 2, fig. S2.2). However, openings are not less than 2/3 of the widest part and therefore these shapes were intended for easy access to their content (adding ingredients, stirring etc.). Seven sub-categories are recognized: globular (B1) and spherical bowls (B2), bowls with elaborated (bulged) rims (B3) and with high shoulders (B4), trunco-conic bowls (B5), carinated (B6) and shallow hemispherical bowls (B7). Furthermore, four size (rim diameter) groups were noted within the bowl category (Fig. 5): large (L; rim diameter between 205 and 350 mm), medium (M; 130-160 mm), small (S; 110-120 mm) and cups (C; 60-80 mm). The bowls, except for the cup-sized ones, are the most probable "cooking vessels" in the assemblage. **Jars** are closed forms of pottery vessels, often featuring necks and handles, and they are considered to have served for storage and transportation. A separate morphological type of pottery vessels is the small, closed globular pots interpreted as **lamps** (SI 2, Fig. S2.2, L1).

A total of 42 pottery samples from layers B and C (spits 2 to 9), representing all morphological types except the jars, were selected for lipid residue analyses (SI 2, Table S2.1, Fig. S2.1 and S2.2). Of them, 38 are from layer B, and four from layer C. Even though the aim was to sample fragments with known typology, this was not always possible: four out of the 42 potsherds are of unknown typological provenance. Rim sherds were preferred as cooking experiments and studies of ethnographic pottery vessels have shown that they contained higher concentrations of lipids (Charters et al., 1993; 1997; Evershed, 2008).

2. Results

2.1 Organic residue extraction

The acidified methanol extraction of the selected potsherds (see S1.1) yielded high recovery rates: 79% of the samples (33 out of the 42) contained appreciable amounts of lipids (>5 µg of lipids per g of sherd). Often the concentration of lipids in the extract was extremely high (up to 18 mg per gram of pottery; SI 2, Table S2.1). Only a few sherds contained ≤100 µg of lipids per gram of sherd, the lowest concentration being 66 µg g^{-1}. These are among the highest concentrations observed in archaeological pottery so far. The arid environment and the acidity of the soil is likely to have played a part in the preservation of organic residue in the ceramic fabric, as previously seen in other studies from similar environments (Dunne et al., 2012; Roffet-Salque and Evershed, 2015; Smyth and Evershed, 2016).

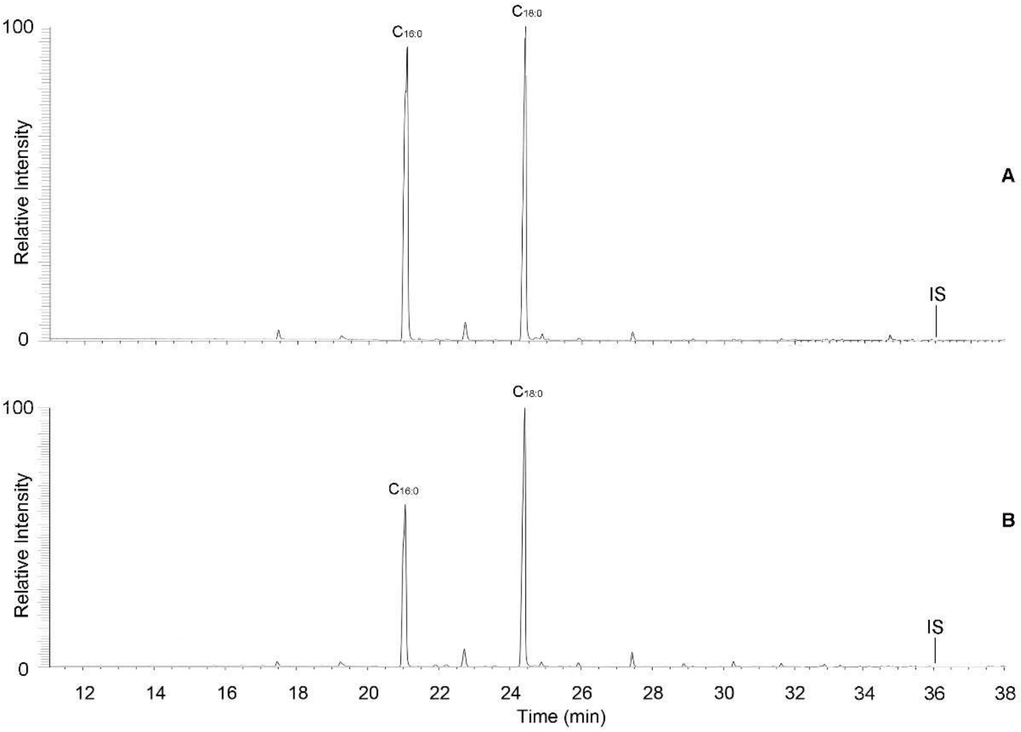

Figure 4. Partial chromatograms of VL021 (A) and VL03 (B), with clear dominance of the palmitic (FA $C_{16:0}$) and stearic (FA $C_{18:0}$) fatty acids. IS=internal standard.

All the extracts were identified as originating from animal fats based on their molecular composition, being dominated by palmitic ($C_{16:0}$) and stearic ($C_{18:0}$) fatty acids (Fig. 4). Using compound-specific stable carbon isotope analyses, it was determined that non-ruminant adipose fats have more enriched $\delta^{13}C$ values for $C_{16:0}$ and $C_{18:0}$ fatty acids than ruminant fats (Evershed et al., 1997b) and a higher $\Delta^{13}C$ (= $\delta^{13}C_{18:0}$ - $\delta^{13}C_{16:0}$) value (Evershed et al., 1999; 2008). As far as ruminants are concerned, due to the different pathways of biosynthesis, milk fats can be distinguished from adipose fats (Dudd and Evershed, 1998; Copley et al., 2003). Therefore, the determination of compound-specific carbon isotopic values of fatty acids by GC-C-IRMS allows animal fats to be further characterise as ruminant (sheep/goat/cattle) adipose, non-ruminant (*e.g.* pig) adipose and milk (dairy products).

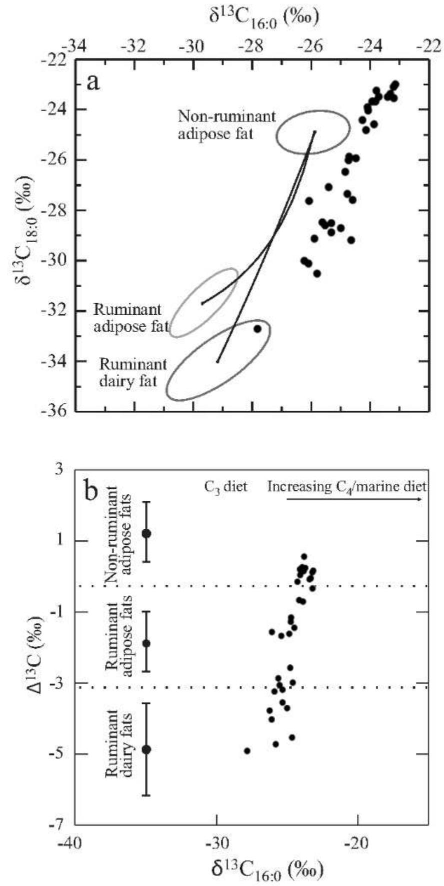

Figure 5. a. $\delta^{13}C$ values for the $C_{16:0}$ and $C_{18:0}$ fatty acids prepared from animal fat residues from sherds from the site of Anta 1. The three fields correspond to the P = 0.684 confidence ellipses for animals raised on a strict C_3 diet in Britain (Copley *et al.* 2003). Each datapoint represents an individual vessel. The analytical error (± 0.3‰) is approximately the size of the points on the graph; b. $\Delta^{13}C$ values from the same potsherds. Ranges show the mean ± 1 s.d. for a global database comprising modern reference animal fats from the UK, Africa, Kazakhstan, Switzerland and the Near East (Dunne *et al.* 2012).

The stable carbon isotope values of the $C_{16:0}$ and $C_{18:0}$ fatty acids in the animal fats extracted from the Anta 1 pottery assemblage range between -27.8‰ and -23.2‰, and -32.7‰ and -23.0‰, respectively. Most of the extracts (if not all) have $\delta^{13}C$ values that are c*a.* 4‰ more enriched than those observed in C_3-reared modern animals (Fig. 5a). The reasons for this enrichment in ^{13}C can be various, including the animal diet and ancient environment, which will be further discussed below (see section 3.2). In order to by-pass this offset and successfully determine the lipid origin, the $\Delta^{13}C$ value was calculated. The $\Delta^{13}C$ value expresses the differences in the metabolism and physiology of the animals (ruminant vs non-ruminant) or their tissues (adipose vs mammary; Evershed et al. 2008). The $\Delta^{13}C$ values of the animal fats extracted from the Anta 1 pottery range between -4.9 and 0.5 ‰ (fig. 5b; SI 2, table S2.1). Animal fats are thus interpreted as originating from non-ruminant adipose (11 samples, 33.5%), ruminant dairy (9 samples; 27%) and pure ruminant adipose fats or mixtures of ruminant and non-ruminant fats (13 samples; 39.5%).

2.2 Aquatic resources

Three specific classes of chemical compounds, ω-(*o*-alkylphenyl) alkanoic acids (APAAs), isoprenoid fatty acids and dihydroxy fatty acids (DHYAs), have been established as biomarkers for the processing of aquatic resources (most commonly fish) in the pottery (Cramp and Evershed, 2014; for instuments and protocols see SI 1). Using GC/MS, all the samples containing organic residue were screened for the above-mentioned biomarkers and none of them provided clear evidence for aquatic resource processing. Therefore, it is safe to assume that aquatic resources were not a major part of the subsistence base of this population.

2.3 Compound-specific radiocarbon dating

Compound-specific radiocarbon dating was performed on two pottery samples, one from each layer (VL03 from layer B and VL21 from layer C, see SI 2, Table 1, Fig. S2.1 and S2.2; see also Fig. 4). This new method uses the carbon contained in the $C_{16:0}$ and $C_{18:0}$ fatty acids of the extracted residue from the pottery (see SI 1; Casanova et al. 2018; *in review*). It offers a unique opportunity for dating the site, as the acidic nature of the soil prevented the preservation of conventional organic materials used for dating. The two potsherds were selected for CSRA based on their stratigraphic position and their high concentrations in $C_{16:0}$ and $C_{18:0}$ fatty acids allowing sufficient quantity of each FA (> 200 µg C) to be isolated for radiocarbon analysis.

Both potsherds passed the internal quality control criteria, established by Casanova et al. (2018, *in review*), based on the statistical identicality of the $C_{16:0}$ and $C_{18:0}$ FAs dates, ensuring the accuracy of the radiocarbon dates. Potsherd VL03 (BRAMS-2845) dates to

4491 ± 47 BP and was in use in 3356-3081 (88% probability) or 3068-3026 (7% probability) calBC for the 95% probability, or in use in 3335-3262 cal BC (26% probability) or 3252-3210 calBC (15% probability) or 3192-3152 calBC (14% probability) or 3138-3099 calBC (13% probability) for the 68% probability range. Potsherd VL21 (BRAMS-2846) dates to 4348 ± 46 BP and was in use in 3091-2892 calBC (95% probability), probably in 3012-2908 calBC (68% probability).

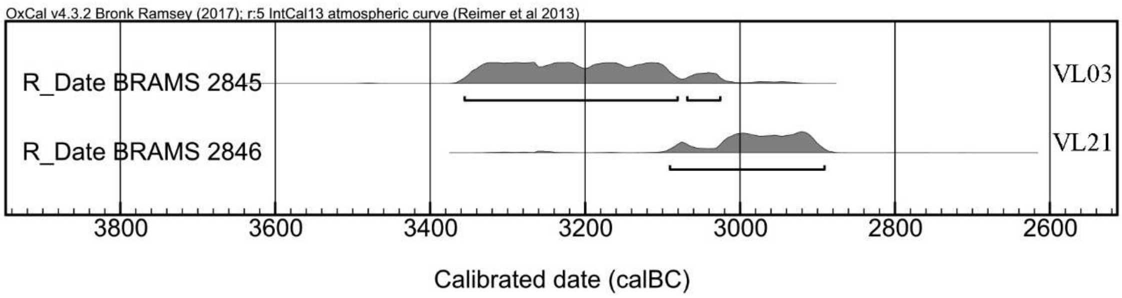

Figure 6. Calibrated ranges of the dates obtained from VL03 and VL21; calibration made in OxCal v4.3.2 platform (Bronk Ramsey, 2017), using the IntCal13 atmospheric curve (Reimer et al., 2013)

The obtained dates appear to contradict the stratigraphic sequence, since VL03 from layer B is older than the supposedly underlying VL21 from layer C (Fig. 6). It is, however, worth noting that these potsherds are single sherds. Thus, it is possible that one of them is residual or intrusive in the context it was recovered from. The inconsistency between the stratigraphic layer and the dates could probably be a result of one of the many disturbances of the stratigraphy from later pits, in which case, VL21 is intrusive in layer C and both samples represent the same layer B. According to the results, the monument existed and was in use during the final third of the 4th and the beginning of the 3rd millennium cal BC, over a millennium after the first passage graves were built in Iberia. The time span covered by the two potsherds corresponds to the end of the Iberian Middle Neolithic, the Final Neolithic, and the transition towards the Copper Age. A more extensive dating sequence of pottery vessels should provide a more complete chronological frame and a better understanding of the stratigraphic disturbances visible at the site.

3. Discussion

The results presented above confirm the presence of carcass fats in the Anta 1 pottery vessels from both ruminant and non-ruminant animals, as well as dairy fats. In view of the lack of animal bones, lipid residue analyses provide the only method to gain insights into animal management at the site. The inhabitants of Anta 1 were thus exploiting both ruminant and non-ruminant animals. The identification of dairy fats confirms that Neolithic people of central Portugal exploited not only the meat from the domestic animals, but, judging from the high percentage of vessels used for milk processing, also dairy products. Dairying was thus an important part of the subsistence economy of the communities frequenting the megalithic monument at the end of the 4th millennium calBC.

3.1 Pottery morphology/food product/lipid concentration associations

An interesting pattern emerges when food commodities are compared against pottery morphology. Even though there are exceptions, it seems that the open dishes were preferred for milk processing, while meat was preferentially cooked in the semi-open bowls (fig. 7a). The only lamp fragment with animal fats contained non-ruminant lipids, suggesting that lard may have been used as fuel. So far, beeswax (Evershed et al., 1997a), plant oils (Kimpe et al., 2001; Colombini et al., 2005) and blubber (Heron et al., 2013) have been identified as illuminants. To our knowledge, this is the first investigated lamp from this part of the world, and the first one to contain fat traces from terrestrial, non-ruminant animal.

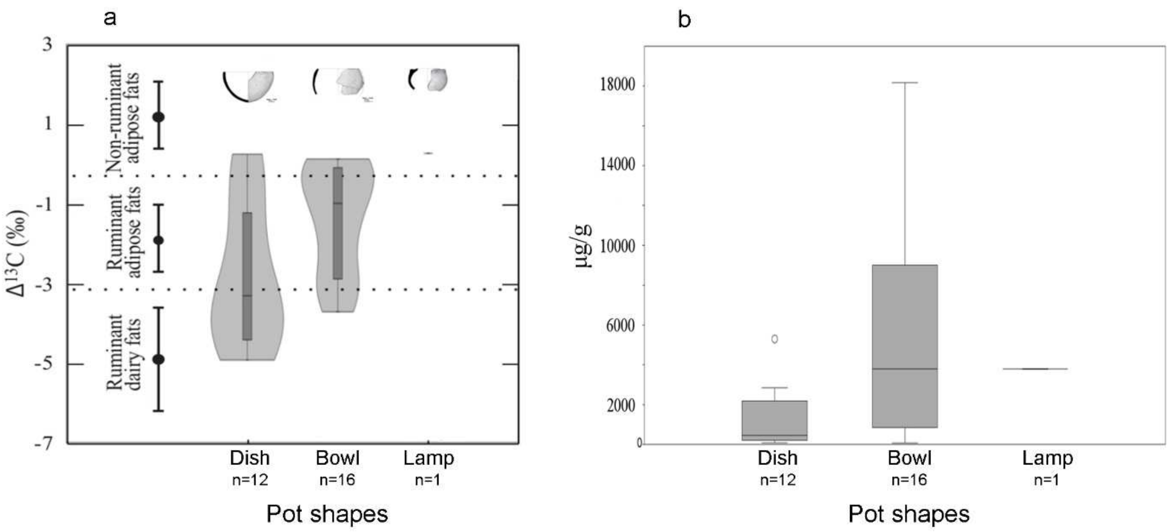

Figure 7. Violin (a) and box plots (b) of $\Delta^{13}C$ values and lipid concentrations, respectively, for the three main typological groups of pottery.

As far as the concentration of lipids is concerned, the dishes show much lower values than the bowls (Fig. 7b). The correlation between dishes, dairy products and low concentrations (relative to the bowls and carcass fats) is intriguing. It might reflect different culinary practices related to different food products, or shorter lifespan (and number of uses) of the dishes compared to the bowls, or both. Further studies in this direction should provide an interesting view on social, economic, cultural and technological aspects of food preparation during the Neolithic.

3.2 Offset from reference values from modern animals reared on C_3 diet in Britain

There is a significant discrepancy between the $\delta^{13}C$ values of the animal fats extracted from Anta 1 and the reference values obtained on modern ruminants raised on C_3 plants in Britain (Copley *et al.* 2003; see section 2.2). Several hypotheses can be proposed to explain this:

A) Increased amount of C_4 plants in the diet of domestic animals will produce enriched $\delta^{13}C$ values of tissue biochemicals (Evershed et al., 2008; Dunne et al., 2012; Roffet-Salque et al., 2016). The major C_4 plants included in the diet of modern-day domestic animals (like millet and maize), were introduced in Europe much later than the Neolithic (e.g. Lawler 2009). However, one C_4 species of grass, the so-called common thatching grass (*Hyparrhenia hirta*), is native to the northern Mediterranean, including central Portugal (Chejara et al., 2010). *Poaceae* taxa are recognised in growing percentages from the 6th millennium onwards in the region, as part of the degradation of previous vegetation cover and replacement of woodland by shrub vegetation and herbs (Ferreira, 2017). However, since the specific weed *Hyparrhenia hirta* is yet to be confirmed in the archaeobotanical record of Neolithic Portugal, its presence cannot be evaluated currently, let alone its influence on the diet of Neolithic livestock.

B) The salinity in a marine environment affects the plant metabolism, resulting in increased $\delta^{13}C$ values (Farquhar et al., 1989). Eventually this affects the $\delta^{13}C$ values of tissue samples from animals grazing in marine environments. Indeed, Cramp *et al.* (2014) observed this effect when comparing the $\delta^{13}C$ values of animal fats extracted from pottery excavated from island vs. mainland from Great Britain. The Tagus estuary has the characteristics of a marine environment and it could have served as a seasonal pasture for the livestock of these semi-nomadic Neolithic herders.

C) In a similar way to salinity, water stress also affects plant metabolism. Aridity decreases the photosynthetic process and indices changes in the metabolism, which is then reflected in the carbon isotope composition of plant tissues. In other words, in arid conditions C_3 plants can switch to the CAM photosynthetic process, which leads to similar fractionation against ^{13}C as in C_4 plants (Farquhar et al., 1989). This plant behaviour has already been mentioned as a possible reason for variations in $\delta^{13}C$ values in Neolithic pottery from Anatolia and Central Europe (Evershed et al., 2008; Roffet-Salque and Evershed, 2015) and is also a possibility for the Tagus region, in the context of the forest degradation mentioned above.

3.3 The view on Anta 1 from the pottery use perspective

Since no settlements have been found, it is impossible to have the complete representation of the Neolithic community in central Portugal, or to evaluate the exact role of the passage graves in the activity pattern in the landscape. Beside the fact that they served as a burial ground, through pottery analyses some additional information becomes apparent. The pottery fragments were scattered all over the mound and inside the structure. No clear pattern of deposition is evident, except that the front side and the interior have a higher concentration of pottery than the rear of the passage grave (Fig. 8). Furthermore, we have seen that the pottery vessels contain very high concentrations of lipids, meaning they were used frequently and likely for a long period. It is probable, therefore, that this was not a "ritual" pottery, produced specially for the occasion (burial) and deposited, but were common "kitchen" utensils, regularly transported around the inhabited territory. This suggests passage graves were not only burial grounds, but more likely a social hub, which frequently aggregated people and activities, becoming the only permanent point in the landscape which anchored the entire circulating community.

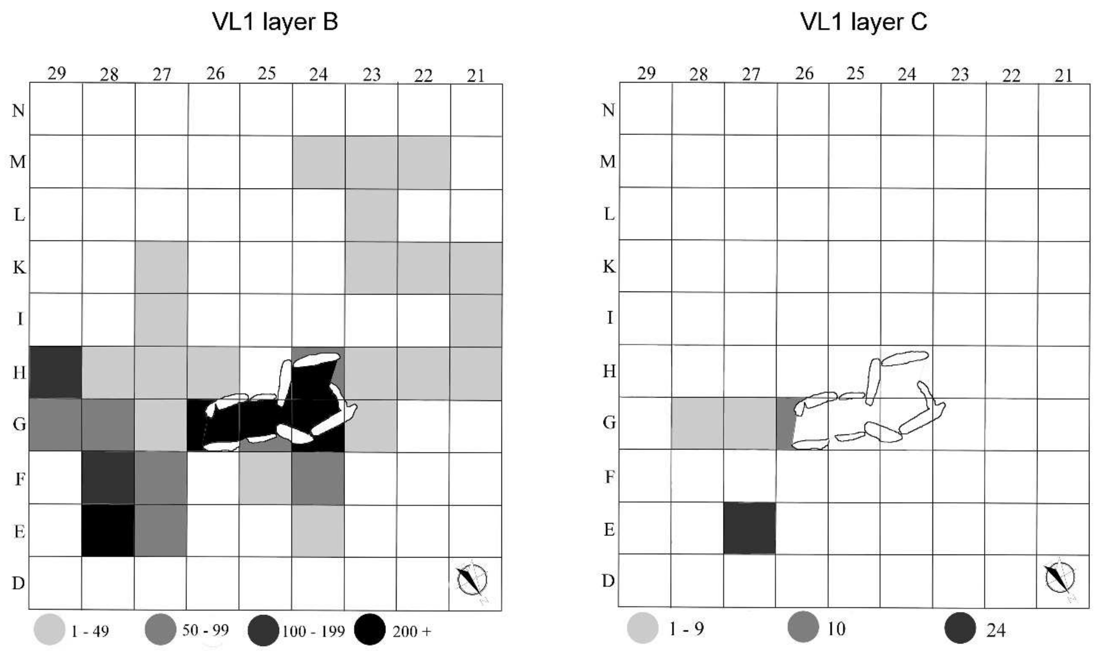

Figure 8. Dispersal and density of pottery fragments on the Anta 1 mound (quantities are expressed in number of sherds).

4. Conclusion

The presence of domestic sheep/goats and cattle in Portugal since the initial stages of farming has already been established through archaeozoology (e.g. Davis & Simões

2016; Almeida 2017; Davis *et al.* 2018). Even if primarily relevant for an advanced stage of the Portuguese Neolithic, this study presents the first direct evidence of not only meat, but also dairy exploitation and processing in this part of Iberia during the late 4th/early 3rd millennium BC. From the animal products that we observed, we could say that animal meat and tissue were more common but given that almost 1/3 of the organic residues were dairy products, the latter were thus far from being an exotic, or exclusive products.

It has also been possible to relate food contents to specific ceramic morphologies, thus approaching morpho-functionality. In the social context of a megalithic passage grave, there was a tendency to use open vessels for milk processing and/or consumption, and semi-open bowls for cooking animal meat.

In contrast to the Mesolithic coastal groups, aquatic commodities were not part of the subsistence base of the megalithic communities of the interior (as the lack of relevant biomarkers suggests), and no indications of plant processing were detected from the pottery organic residues (Dunne et al., 2016). This suggests a predominant use of the pottery for terrestrial animal products, which is consistent with an understanding of a strong pastoral component of the economy (Scarre and Oosterbeek, 2010)

The close relationship between the Neolithic communities and their environment is also reflected on a molecular level. The stable carbon isotope values of the fatty acids extracted from the sherds reflect the diet of the animals that provided the meat and milk for the site, either as an influence of the salinity of the Tagus estuary, extreme aridity episodes, significant input of C_4 plants in the animal diet, or a combination of the three.

Acknowledgements

The presented results were obtained through a PhD research project, during which DS benefitted from an Erasmus Mundus European Commission fellowship fund, in the frameworks of the Quaternary and Prehistory programme. Research on the Anta 1 de Val da Lage is undertaken with the approval of the Portuguese Directorate for Cultural Heritage (Contract PIPA Es.ter.tejo) and the funding of the Portuguese Foundation for

Science and Technology (FCT contract UID/Multi00073/2019). DS would also like to thank Stefanija Stojanovska for the pottery drawings, Borys Banecki for the technical support during the laboratory analyses at the University of Bristol and Jens Holtvoeth and Julie Dunne for a very useful discussion on C_4 plants. MRS and RPE were funded by the European Research Council Advanced Grant "NeoMilk" FP7-IDEAS-ERC/324202. The Royal Society is thanked for funding a fellowship to M.R.S (DHF\R1\180064). We thank the NERC for partial funding of the MS facilities at Bristol (Contract R8/H10/63; www.chm.bris.ac.uk/lsmsf/). LO wishes to thank FCT for the funding of the MTAS project (contract PTDC/EPH-ARQ/4356/2014), allowing for the recent field campaigns and the compound-specific radiocarbon dating.

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Declaration of interests

☒ The authors declare that they have no known competing financial interests or personal relationships that could have appeared to influence the work reported in this paper.

☐The authors declare the following financial interests/personal relationships which may be considered as potential competing interests: