comptes rendus
palevol
2024 • 23 • 1

Faire gras à Molène: dairy products and ruminant fats detected by lipid and isotopic analysis of pottery dating to the Final Neolithic-Early Bronze Age from the island site of Beg ar Loued (Molène, western Brittany, France)

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art. 23 (1) — Published on 15 January 2024
www.cr-palevol.fr

PUBLICATIONS SCIENTIFIQUES

Couverture / *Cover* :
Aerial view of the Early Bronze Age settlement of Beg ar Loued being excavated from the shore at low tide. Credits: Marine nationale, 2009 (no. 09LVCNO50).

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diff.pub@mnhn.fr / https://sciencepress.mnhn.fr

Académie des sciences, Institut de France, 23 quai de Conti, 75006 Paris.

ISSN (imprimé / *print*) : 1631-0683/ ISSN (électronique / *electronic*) : 1777-571X

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Camielsa PRÉVOST
Cultures et Environnements Préhistoire, Antiquité, Moyen Âge (CEPAM), CNRS,
Université Côte d'Azur (UniCA), Pôle Universitaire Saint-Jean d'Angély (SJA3),
24, avenue des Diables Bleus, F-06300 Nice (France)
camielsa.prevost@cepam.cnrs.fr ; camielsa.prevost@hotmail.fr (corresponding author)

Akshyeta SURYANARAYAN
Cultures et Environnements Préhistoire, Antiquité, Moyen Âge (CEPAM), CNRS,
Université Côte d'Azur (UniCA), Pôle Universitaire Saint-Jean d'Angély (SJA3),
24, avenue des Diables Bleus, F-06300 Nice (France)
and Culture, Archaeology and Socio-Ecological Dynamics (CASEs) Research Group,
Department of Humanities, Universitat Pompeu Fabra,
Plaça de la Mercè, 12, 08002 Barcelona (Spain)
and Faculty of Asian and Middle Eastern Studies, University of Oxford,
1 Pusey Lane, Oxford OX1 2LE (United Kingdom)
and McDonald Institute for Archaeological Research, University of Cambridge,
Downing Street, Cambridge CB2 3ER (United Kingdom)
as2429@cam.ac.uk

Yvan PAILLER
Littoral, Environnement, Télédétection, Géomatique (LETG), CNRS, Nantes Université,
Université de Bretagne Occidentale, Université Rennes 2, Chaire Professeur Junior ArMeRIE,
Institut Universitaire Européen de la Mer, Technopôle Brest-Iroise,
rue Dumont d'Urville, F-29280 Plouzané (France)
yvan.pailler1@univ-brest.fr

Clément NICOLAS
Laboratoire Trajectoires. De la sédentarisation à l'état, Université Paris 1 - Panthéon-Sorbonne,
Centre de Recherche, 9 rue Malher, F-75004 Paris (France)
clement.nicolas@cnrs.fr

Thierry BLASCO
Sorbonne Université, CNRS, Laboratoire d'Océanographie de Villefranche (LOV),
181 Chemin du Lazaret, F-06230 Villefranche-sur-Mer (France)
thierry.blasco@obs-vlfr.fr

Arnaud MAZUY
Cultures et Environnements Préhistoire, Antiquité, Moyen Âge (CEPAM), CNRS,
Université Côte d'Azur (UniCA), Pôle Universitaire Saint-Jean d'Angély (SJA3),
24, avenue des Diables Bleus, F-06300 Nice (France)
arnaud.mazuy@cepam.cnrs.fr

Pauline HANOT
Mécanismes Adaptatifs et Evolution, CNRS, Muséum national d'Histoire naturelle,
57 rue Cuvier, F-75005 Paris (France)
pauline_hanot@hotmail.fr

Yvon DRÉANO
Centre de Recherche en Archéologie, Archéosciences, Histoire (CReAAH), CNRS,
Université de Rennes, Campus de Beaulieu, Bâtiments 24/25, Labo Archéosciences,
Avenue du Général Leclerc, CS 74205, F-35042 Rennes Cedex (France)
and Études et valorisations archéologiques (EVEHA), Bureau d'Études archéologiques,
13 rue des Granges Galands, F-37550 Saint-Avertin (France)
yvon.dreano@eveha.fr

Catherine DUPONT
Centre de Recherche en Archéologie, Archéosciences, Histoire (CReAAH), CNRS,
Université de Rennes, Campus de Beaulieu, Bâtiments 24/25, Labo Archéosciences,
Avenue du Général Leclerc, CS 74205, F-35042 Rennes Cedex (France)
catherine.dupont@univ-rennes.fr

Martine REGERT
Cultures et Environnements Préhistoire, Antiquité, Moyen Âge (CEPAM), CNRS,
Université Côte d'Azur (UniCA), Pôle Universitaire Saint-Jean d'Angély (SJA3),
24, avenue des Diables Bleus, F-06300 Nice (France)
martine.regert@cepam.cnrs.fr

Submitted on 12 May 2023 | Accepted on 11 September 2023 | Published on 15 January 2024

urn:lsid:zoobank.org:pub:D63CA04C-B8A0-4BEC-B054-F32D38335F95

Prévost C., Suryanarayan A., Pailler Y., Nicolas C., Blasco T., Mazuy A., Hanot P., Dréano Y., Dupont C. & Regert M. 2024. — Faire gras à Molène: dairy products and ruminant fats detected by lipid and isotopic analysis of pottery dating to the Final Neolithic-Early Bronze Age from the island site of Beg ar Loued (Molène, western Brittany, France). *Comptes Rendus Palevol* 23 (1): 1-30. https://doi.org/10.5852/cr-palevol2024v23a1

ABSTRACT
The subsistence strategies of early farming communities have been highlighted since the beginning of the Neolithic, thanks to numerous studies on lipid residues from ceramic vessels conducted in various parts of continental Europe. However, after the Early Neolithic, evidence of subsistence strategies along the northern Atlantic coast are still lacking, especially for island contexts. This paper presents the results of lipid residue analysis of 129 potsherds from Beg ar Loued (Molène, France), an island site dating primarily to the Early Bronze Age (*c.* 2700-2600 to 1800 BCE). Aiming to understand the use of vessels, vessel treatment and culinary practices on the settlement, analyses of visible charred residues, sherds and ceramic surfaces/coating layers were carried out using chromatographic (n = 174) and isotopic techniques (n = 24) after lipid extraction by solvent (n = 174) or acid methanolysis (n = 31). The results demonstrate the extensive use of terrestrial products (ruminant carcass and dairy) in pottery, including occasional plant products (with possible mixtures of different waxes), while the detection of aquatic products is limited. Thus, combined with evidence from faunal remains at the site, the results indicate that terrestrial resources like ruminant meat and dairy products were preferentially processed in vessels, and aquatic products mostly without the use of ceramics. These findings demonstrate the significance of lipid residue analysis for studying the role of pottery in food production and consumption at sites along the Atlantic coast.

KEY WORDS
Lipids,
pottery,
Final Neolithic,
Early Bronze Age,
France,
Atlantic coast.

RÉSUMÉ
Faire gras à Molène : produits laitiers et graisses de ruminants détectés par l'analyse lipidique et isotopique des céramiques du Néolithique final et de l'âge du Bronze ancien du site insulaire de Beg ar Loued (Molène, Bretagne occidentale, France).
Les stratégies de subsistance des premières communautés agricoles ont été mises en évidence depuis le début du Néolithique grâce à de nombreuses études sur les résidus lipidiques des récipients en céramique menées dans diverses parties de l'Europe continentale. En revanche, très peu de données sont disponibles pour la fin du Néolithique et le début de l'âge du Bronze sur la côte atlantique, en particulier en contexte insulaire. Cet article présente les résultats de l'analyse de résidus lipidiques provenant de 129 fragments de poteries de Beg ar Loued (Molène, France), un site insulaire dont les principaux vestiges datent de l'âge du Bronze ancien (*c*. 2700-2600 à 1800 BCE). Dans le but de comprendre l'utilisation des récipients, les pratiques culinaires sur ce site et d'appréhender les techniques de finition des céramiques, des analyses de résidus visibles carbonisés, de tessons, et de surfaces/

couches d'engobe ont été effectuées via des analyses chromatographiques (n = 174) et isotopiques (n = 24), après extraction des lipides par solvant (n = 174) ou méthanolyse acide (n = 31). Les résultats démontrent l'utilisation extensive de produits terrestres (carcasses de ruminants et produits laitiers), comprenant occasionnellement des produits végétaux (avec un mélange probable de différentes cires), alors que la détection des produits aquatiques est faible. Comparés aux données fauniques, ces résultats indiquent donc que les produits terrestres, tels que la viande de ruminant et les produits laitiers, sont transformés en utilisant des récipients en céramiques, tandis que les produits aquatiques semblent de préférence exploités sans avoir recours à une poterie. Ces résultats démontrent l'importance de l'analyse des résidus lipidiques pour connaître le rôle des récipients en céramique dans la production et la consommation d'aliments sur les sites de la côte atlantique.

MOTS CLÉS
Lipides,
poterie,
Néolithique final,
âge du Bronze ancien,
France,
côte atlantique.

INTRODUCTION

The subsistence strategies of early farming communities and consumption of dairy products in Europe have been highlighted since the beginning of the Neolithic period, thanks to numerous studies on lipid residues conducted in Southern, Central and Northern Europe (Craig *et al.* 2005a; Evershed *et al.* 2008; Cramp *et al.* 2014a, b; Roffet-Salque *et al.* 2015; Debono Spiteri *et al.* 2016; Cubas *et al.* 2020). Along the Atlantic coast, the importance of terrestrial animal products, such as ruminant meat and milk in vessels, have been reported, with an increase in dairy products apparent along a northerly latitudinal gradient (Cubas *et al.* 2020). Similar results are reported for the Bronze Age from sites across the Channel in England (Copley *et al.* 2003; Šoberl 2011). Despite a short distance to the sea, a general absence of evidence of aquatic products in vessels has been noted (Schulting 2005; Cubas *et al.* 2020; Evershed *et al.* 2022).

From the Final Neolithic and into the Bronze Age, evidence of subsistence strategies is still lacking. In Brittany, northwestern France, this period is marked by climatic fluctuations and landscape transformations (Stéphan *et al.* 2019b), changes in building methods and settlement patterns (reduction in the size of the settlement/population, Blanchet *et al.* 2018), the introduction of metal production, and the spread of new technical and decorative styles of ceramics (Salanova *et al.* 2011; Ripoche 2017, 2022; Blanchet *et al.* 2019; Nicolas *et al.* 2019; Favrel 2020). Due to the acidic pH of the region's sediments, faunal remains rarely survive, and as a result, subsistence strategies during this period are not well documented. Slaughter profiles at sites where faunal remains have been found show an increase in the breeding of ovicaprines and pigs alongside cattle, underlining the importance of dairying from the Final Neolithic to the Bronze Age in food production (Braguier 2000; Hanot & Tresset 2019).

The study of lipid residues in pottery has the potential to add deeper insights into past subsistence and culinary practices, as demonstrated by previous studies (Craig *et al.* 2005a, b; Evershed 2008; Cramp *et al.* 2014a, b, 2019; Debono Spiteri *et al.* 2016; Roffet-Salque *et al.* 2016; Evershed *et al.* 2022). Most of the data on lipid residues in ceramic vessels in the region is available for inland sites (Cramp *et al.* 2014a; Cubas *et al.* 2020), while island contexts, which are unique due to their proximity to the sea, have received little attention.

The site of Beg ar Loued is one of the few EBA settlements on the coast of Brittany which shows exceptional preservation of faunal material (Fig. 1; Pailler *et al.* 2011; Pailler & Nicolas 2019a, b). It was excavated between 2008 and 2011 by some of us (YP and CN). The first occupation of the site corresponds to a shell deposit dating back to the Final Neolithic (FN: *c*. 3300-2800 cal. BCE). The most significant remains on the site date to the Early Bronze Age (EBA1: *c.* 2200-1950 cal. BCE; EBA2: *c.* 2000-1800 cal. BCE) and consist of two dry-stone domestic buildings, interpreted as houses. There is also a phase of site desertion (EBA3: *c.* 1665-1302 cal. BCE). In some areas of the site, this phase is difficult to separate with a layer of older colluvium from an upper area (FN: *c.* 3000 BCE). The chronology is based on the dating of 39 remains of bones, charcoal and charred residues on ceramic vessels (Pailler & Nicolas 2019a). The faunal remains and location of the site provide clear evidence that the relationship with the ocean was important for populations at Beg ar Loued (Pailler & Nicolas 2019a). Nevertheless, the ceramics, lithics and faunal remains indicate a strong link with the mainland.

This paper explores the cultural choices of the population for the exploitation of coastal/inland resources in pottery vessels, culinary practices, and surface treatment through lipid and isotopic analyses carried out on 129 potsherds. We focus our research on animal products used in ceramic vessels, specifically dairy products, and also investigate whether aquatic resources were processed in ceramics. In addition, we open up new questions about the use of organic products in surfaces/coating layers for the finishing treatment of specific potsherds.

Beg ar Loued and its landscape in the final Neolithic-Early Bronze Age

Environmental studies of the coastline of Brittany have shown that the end of the Neolithic was concomitant with rapid environmental transformation, including major marine transgressions (Bond *et al.* 1999; Magny 2004; Joly & Visset 2005; David 2014), climate change (e.g. Bond Event 3; Bond *et al.* 1999) and cooling weather (Martín-Chivelet *et al.* 2011). Changes in land use and occupation are detected with Neolithic oak stands (*Corylus* L., *Quercus* L., *Tilia* L.,

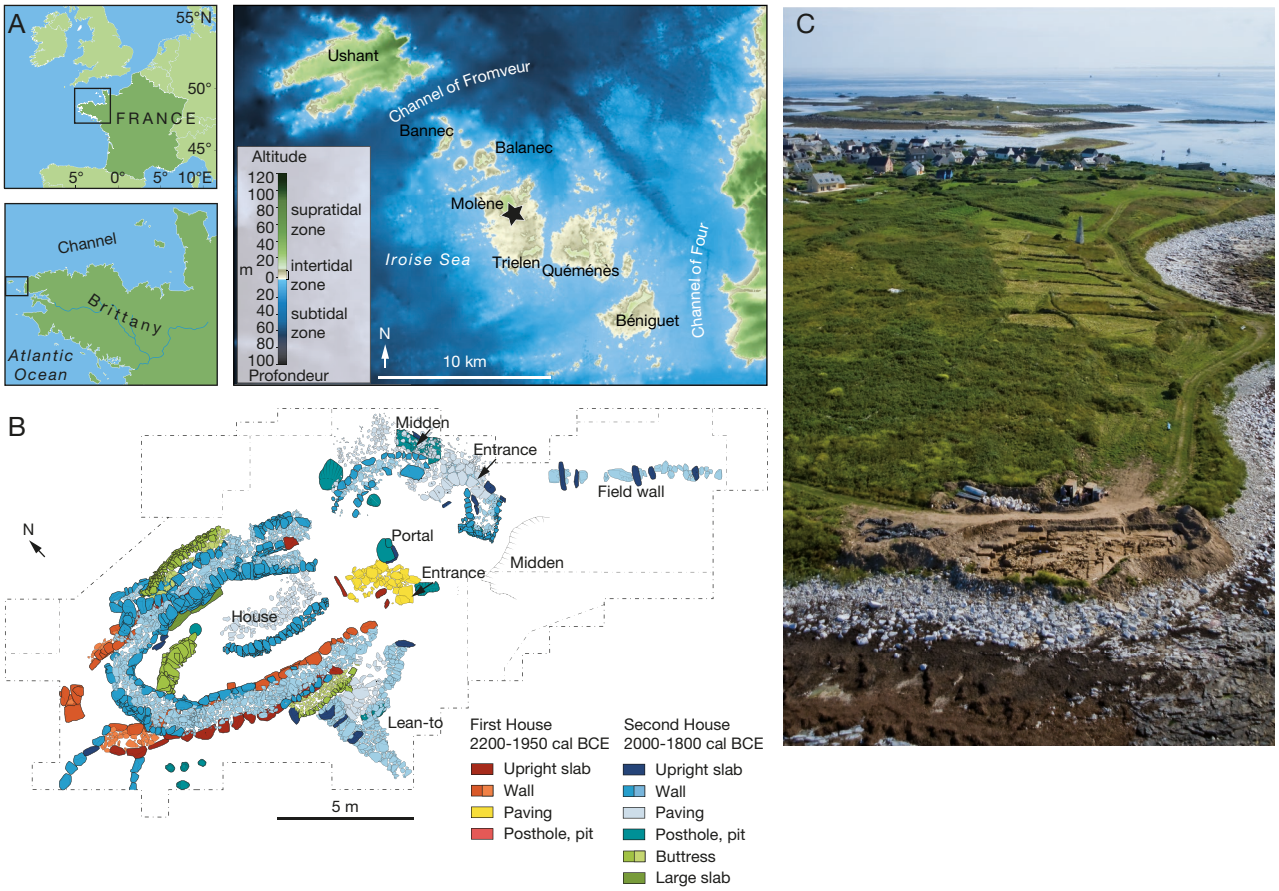


Fig. 1. — Site of Beg ar Loued: **A**, location on maps (approximate DMS coordinates 48°23'25.724"N, 4°57'33.583"W) and reconstructed Early Bronze Age shorelines; **B**, detailed plan of the superimposed houses; **C**, aerial photograph of the settlement. Credits: Marine Nationale; CAD, C. Nicolas.

Ulmus L.) replaced by various riparian taxa such as ash, elder, hazel, hawthorn and beech (Gaudin 2004; Martín-Chivelet *et al.* 2011; David 2014). The causes of these changes are still debated (Stéphan *et al.* 2019a, b).

Beg ar Loued is located on the island of Molène (Finistère) which belongs to the Molène archipelago that was attached to the mainland during the Last Glacial Maximum (Billard *et al.* 2020). With the gradual rise of sea level, the archipelago was transformed into a variety of smaller islands and islets by the early Neolithic; 4800 cal. BCE (for a better overview, see Dréano *et al.* 2013; Gandois *et al.* 2013; Stéphan *et al.* 2013; Pailler *et al.* 2014; Bernard *et al.* 2020; Billard *et al.* 2020). During the occupation of the two dry-stone buildings of Beg ar Loued, depending on the tide, the sea was between 0.1 and 2 km from the site (Pailler *et al.* 2014; Stéphan *et al.* 2019a).

Marine and terrestrial food strategies at Beg ar Loued

An abundance of faunal remains, both marine and terrestrial, have been discovered at Beg ar Loued (Pailler *et al.* 2004; Hanot & Tresset 2019; Pailler & Nicolas 2019a). Nearly 40 species of marine fish, particularly sea bass and gilthead bream, have been identified, as well as cuttlefish, crabs, and sea urchins (Pailler & Nicolas 2019a). The range of species, particularly migratory species such as gilthead bream, sea bass, garfish, eel and conger eel, demonstrates that fishing was carried out throughout the year, perhaps more intensively during the summer, since the majority of the remains identified are of gilthead bream (Dréano 2019). Based on a study of the burns and traces on the fishbones, as well as their spatial distribution and deposition across the site, it has been suggested that a system of procurement and processing (preparation, drying, smoking and/or salting) of fish was carried out on site, perhaps to transport or trade the fish (Dréano 2019).

Additionally, no less than 21 species of shellfish have been found, including limpets, mussels, thick top shell, and in lower proportions carpet clams, abalone, and scallops (Dupont 2019; Mougne 2019). Around 1% of the limpets show charring marks. A variety of bird remains, both freshwater, shorebirds and marine, are also attested at the site (Hanot & Tresset 2019), suggesting that they could have contributed to the diet of the island's inhabitants.

Terrestrial mammals, such as cattle, pigs, sheep, and perhaps goats, were also found at the site (Pailler *et al.* 2014; Hanot & Tresset 2019). The presence of all age categories and a homogeneous anatomical distribution of the faunal remains suggests that at least ovicaprines were raised at the site for meat production, and also for secondary product use such as dairying (Hanot & Tresset 2019). The faunal remains

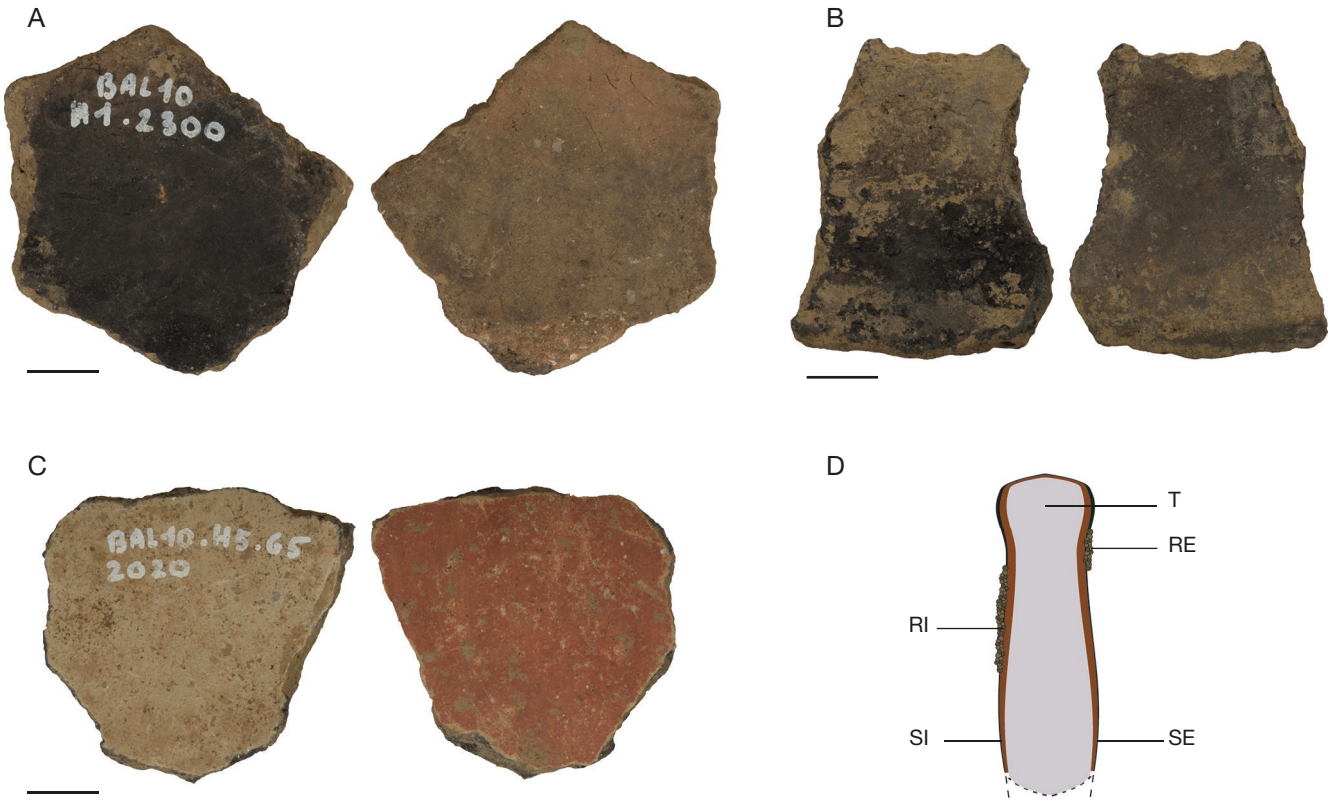


Fig. 2. — **A**, **B**, **C**, Examples of pottery fragments analysed in the framework of this study (internal face on left); **D**, diagram showing different sampling options and abbreviations used. Abbreviations: **RE**, external visible charred residues; **RI**, internal visible charred residues; **SE**, external surface/coating layer; **SI**, internal surface/coating layer. Scale bars: 1 cm. Credits: photos, J.-D.Strich; CAD, C. Prévost.

from Beg ar Loued provide some of the first evidence of animal husbandry in Atlantic island contexts during the Final Neolithic-Early Bronze Age (Hanot & Tresset 2019).

Archaeobotanical remains of 11 species of plant taxa were also recovered from the site, which included hulled and naked barley, emmer, and wheat, as well as fava bean and pea (Pailler *et al.* 2014; Pailler & Nicolas 2019a). A crop weed, wild radish, is also present on the site, as well as three wild fruit trees (hazel, common hawthorn, and common dogwood), possibly linked to a gathering activity (Pailler *et al.* 2014). These remains suggest that coastal fishing was associated with the use of crops and livestock, thus linking prehistoric human groups to the mainland (Pailler *et al.* 2014).

Ceramic and lithic remains

Many lithic tools and ceramics have been discovered at Beg ar Loued (Pailler & Nicolas 2019a), although the ceramic assemblage is highly fragmentary, and many sherds do not have a precise chronological or typological classification. Despite its limitations, the ceramic corpus is important as it provides one of the few examples of ceramics from a settlement dating through the 3rd and 2nd millennium BCE in the region (Salanova 2019). Final Neolithic *Conguel* style, Bell-Beaker pottery (Salanova 2000) as well as cordoned urns and handled jugs, which are typical of the Early Bronze Age, have been identified in the assemblage. All the usual forms are present. However, glossy red coating surfaces (*engobe*) have been found on fine vessels, which is unique to the Beg ar Loued corpus, as the use of coating techniques is relatively unknown in the northern French Atlantic zone (Hamon 2008; Salanova *et al.* 2011). The red colour, reminiscent of the red-brick *Campaniforme*/Bell Beaker of Europe (Lemercier 2002; Salanova 2004; Favrel 2020), has a brilliant lustre and very fine porosity contrasting with the brown-black ceramics and coatings observed for Middle and Final Neolithic period ceramics (Fig. 2). The site also provides a good example of the dynamic between ceramics and lithics from the mainland versus the islands. While the lithics and pottery were produced locally, ceramic styles follow the same trends observed on the mainland.

Abbreviations

Molecules

AL	*n*-alkane(s);
APAA(s)	ω-(o-alkylphenyl) alkanoic acid(s);
Ch	cholesterol derivative(s);
CT	contamination;
D/DAG	diacylglycerol(s);
E	ester(s);
FA	fatty acid(s);
H	hydroxy ester(s);
IFA	isoprenoid fatty acids (PRI+PHY+TMTD);
M/MAG	monoacylglycerol(s);
N	diterpene(s);
OH	*n*-alcohol(s);
P	phthalates;

TABLE 1. — Number of ceramic fragments, visible charred residues and surface/coating layers studied from Beg ar Loued according to chronological periods. These include samples studied by Regert & Mazuy (2019). In **bold**, total values for the whole sample-set. Abbreviations: **EBA**, Early Bronze Age; **DCM**, dichloromethane; **FN**, Final Neolithic; **H_2SO_4**, sulphuric acid; **MeOH**, methanol; **RE**, external visible charred residues; **RI**, internal visible charred residues; **SE**, external surface/coating layer; **SI**, internal surface/coating layer.

Chronological period	Key dates (cal. BCE)	Rim	Body	Base	Unsp.	Total sherds	Visible residues		Coating surfaces		Total of extracted samples	
							RI	RE	SI	SE	DCM/MeOH	MeOH/H_2SO_4
FN	3300-2800	2	1	0	3	**6**	1	0	0	0	**7**	**1**
EBA1 (1st house)	2200-1950	8	17	6	10	**41**	9	5	2	5	**62**	**12**
EBA (1 and 2)	2200-1800	6	1	0	1	**8**	0	1	0	0	**9**	**2**
EBA2 (2nd house)	2000-1800	7	5	4	3	**19**	5	0	1	1	**26**	**4**
EBA (3)	1660-1300	11	18	5	8	**42**	3	3	3	3	**54**	**8**
Unsp. (FN to EBA3)	3000/1660-1300	2	8	2	1	**13**	2	1	0	0	**16**	**4**
Total	–	**36**	**50**	**17**	**26**	**129**	**20**	**10**	**6**	**9**	**174**	**31**

PHY phytanic acid;
PRI pristanic acid;
RRR 3R,7R,11R,15-phytanic acid;
S sterol(s);
SRR 3S,7R,11R,15-phytanic acid;
T/TAG triacylglycerol(s);
TMTD 4,8,12-trimethyltridecanoic acid;
γ gamma lactone(s).

Analyses
CSIA compound-specific isotopic analysis;
GC gas chromatography;
GC-c-IRMS gas chromatography-combustion-isotope ratio mass spectrometry;
GC-FID gas chromatography-flame ionization detection;
GC-MS gas chromatography-mass spectrometry.

Reactive chemicals
BF3 boron trifluoride;
DCM dichloromethane;
H_2SO_4 sulphuric acid;
MeOH methanol;
NaOH sodium hydroxide.

Quantification
TLE total lipid extract;
TR trace amount.

Various
BAL Beg ar Loued;
US stratigraphic unit.

Chronological periods
BA Bronze Age;
EBA Early Bronze Age;
EBA1 Early Bronze Age 1;
EBA2 Early Bronze Age 2;
EBA3 Early Bronze Age 3;
FN Final Neolithic;
MBA Middle Bronze Age;
U unspecified.

Samples
RE external visible charred residues;
RI internal visible charred residues;
SE external surface/coating layer;
SI internal surface/coating layer;
T sherd.

SAMPLE-SET AND ANALYTICAL STRATEGY

A preliminary investigation of 23 samples (20 sherds, three carbonised residues) was carried out by solvent extraction (DCM/MeOH) and analysed via chromatographic techniques in 2016 (GC-FID, GC-MS) (Regert & Mazuy 2019). Our study expanded the sample set to include a larger number of sherds, visible encrusted residues and surface/coating layers. We also sampled topsoil as a control for lipid residue analysis from a beach scarp north of the site in 2022, several years after excavations and the initial sampling of vessel fragments.

Combining the samples from the Regert & Mazuy 2019 study, the sample-set for lipid residue analysis includes 129 sherds and 30 visible carbonised residues adhering to the external ('RE'; n = 10/30) or the internal surfaces ('RI'; n = 20/30), which are most often found on body fragments from pots with medium thickness walls. Among the 129 sherds, some had abraded surface layers and discolorations, with a few examples of lustrous red coating on the exterior and white coating on the interior part of some sherds (e.g. MR6516, Fig. 2). Where feasible, the surface/coating layers were sampled separately for lipid analysis. Table 1 provides details of the sherds ('T'), visible charred residues ('RI'/'RE') and surface/coating layers ('SI'/'SE') samples analysed in the study grouped by chronological period.

Despite the fragmentary nature of the ceramic assemblage of Beg ar Loued, several unique elements of the vessel form (rims, bases, decoration) allow a typological attribution and, more rarely, reconstructions of vessel profiles. Most of the pottery is attributed stylistically, as they have been rarely found in well-dated layers (Pailler & Nicolas 2019b). A large number of the vessel fragments (n = 37/129; 29%) came from a sandy colluvium unit, which includes Early Bronze Age objects *in situ,* and rolled sherds from a scree of an area uphill of the EBA houses and dated to the Final Neolithic, in secondary deposition. When the sherds from this layer are not typical of any cultural group, they are classified as 'Unspecified (FN/EBA3)' as a precautionary measure.

Prior to lipid extraction, the external layer of each potsherd was removed, and when present, surface/coating layers were gently cleaned with a modelling drill in order to reduce the potential of contamination from the burial environment or during post-excavation (Stern *et al.* 2000). In total, 174 solvent

TABLE 2. — Details of the total lipid extract (**TLE**) obtained by solvent extraction from absorbed lipids in sherd fragments 'T', charred surface residues adhering to either the internal 'RI' or external surface of the sherd 'RE', and surface/coating layers (either internal 'SI' or external 'SE'), and individual vessels (combining different samples linked to one vessel) analysed in this study. In **bold**, total values for the whole sample-set.

	TLE ≥5 µg.g^{-1}			TLE <5 µg.g^{-1}	
Sample Type	**Number of samples**	**Average (µg.g^{-1})**	**Median (µg.g^{-1})**	**Number of samples**	**Total samples**
Total samples	126	–	–	48	**174**
Sherd (T)	95	140	54	34	**129**
Visible charred residues	17	–	–	13	**30**
Internal residues (RI)	11	148	47	9	**–**
External residues (RE)	6	302	146	4	**–**
Surface/Coating layers	14	–	–	1	**15**
Internal surface layer (SI)	6	892	460	0	**–**
External surface layer (SE)	8	814	258	1	**–**
Individual vessels (combining T, SE/SE, RI/RE)	100	285	69	29	**129**

extractions were carried out on the 129 sherds collected (sherd, visible charred residues and surface/coating layers). A selection of 24 solvent-extracted samples were prepared for compound-specific isotopic analysis (CSIA) of palmitic and stearic acid in the extracts to refine the characterisation of ruminant and dairy fats, and poorly preserved molecular assemblages (Dudd & Evershed 1998; Copley *et al.* 2003). As the question about the use of aquatic resources at the coastal site of Beg ar Loued was important, we looked for specific evidence of these substances in the solvent-extracted compounds. But due to the challenges for detecting aquatic markers through solvent extraction alone (Garnier *et al.* 2018), we further conducted acidified methanol extractions on 31 samples (including 3 external surface/coating layers). These samples were selected on the basis of high lipid concentrations and the presence of long-chain fatty acids, phytanic acid or ketones. They were analysed via GC-MS using SIM mode for detecting characteristic ions of aquatic biomarkers (e.g. *m/z* 74, 87, 213, 270 for TMTD; *m/z* 101, 312, 171, 326 for phytanic acid (PHY) and pristanic acid (PRI); *m/z* 105, 262, 290, 318, 346 for $APAAs_{C16\text{-}C22}$; Cramp & Evershed 2014; Lucquin *et al.* 2016a).

In summary, we prepared samples (DCM/MeOH; n = 174) for chromatographic (GC-FID, GC-MS) and Compound-Specific Isotopic Analysis (CSIA; n = 24). We also conducted acidified methanol extractions on selected samples (MeOH/H_2SO_4; n = 31) and analysed them via GC-MS in SIM mode. The methods for lipid extractions (Evershed *et al.* 1990; Correa-Ascencio & Evershed 2014), GC-MS analysis (Cramp & Evershed 2014; Garnier *et al.* 2018) and CSIA (Dudd & Evershed 1998; Copley *et al.* 2003) are based on previously published protocols. See Appendices 1-4 for full details of our analytical strategy, methods, control analysis and data.

RESULTS

Lipid concentration from solvent-extracted samples

When considered as individual vessels (i.e., combining different sample types linked to a vessel: 'T', 'RE'/'RI', 'SE'/'SI'), one-hundred samples (n = 100/129; 78%) had total lipid extracts (TLEs) greater than 5 µg.g^{-1}. The average lipid yield of this group was 285 µg.g^{-1}, and median lipid yield was 69 µg.g^{-1}. Sixty-five vessels (n = 65/129; 50%) contained intact triacylglycerols (TAGs – the main lipid constituents of fresh animal fats and plant oils), and half of this group had TAG concentrations above 5% of the TLE (n = 33/65; 51%). These results suggest that despite excellent preservation of TAGs, lipid concentrations across analysed fragments were relatively low, with a few samples driving up the average.

In Table 2, we provide the details of lipid yields for each sample category: 'Individuals vessels'; 'T'; 'RI'; 'RE'; 'SI'; 'SE'. Comparisons of the lipid yields from the absorbed sherd residues and visible charred residues revealed that they were broadly similar (Wilcoxon Signed-Rank Test, Z = 2252.5, p = 0.16; Table 2; see also Appendix 5), and external charred residues had higher lipid yields than internal charred residues (Wilcoxon Signed-Rank Test, Z = 118.5, p = 0.4; Table 2; see also Appendix 6). This pattern is contrary to other studies, were lipid yields from visible charred surface residues are significantly higher than those from absorbed residues (Mukherjee *et al.* 2008; Courel *et al.* 2020a, b). Visible residues may have been exposed to various diagenesis processes during burial, while absorbed residues are protected from microbial attack by the inorganic ceramic matrix. This could explain the poor conservation of visible residues in this context. We also noticed that external visible residues and external coating layers have higher amounts of lipids than the internal visible residues and internal surfaces/coating layers (see Appendix 6). This could be related to the use of pottery or specific coating treatment. The comparison of the TLE and the percentage of TAGs preserved against the year the potsherd was excavated, or the chronological period of the pottery did not reveal any patterns (see Appendices 7-9).

Molecular characterisation of solvent-extracted samples

The obtained lipid profiles of all the solvent-extracted samples in this study are dominated by even-chain free fatty acids ($C_{10:0}$-$C_{30:0}$), half of them including long carboxyl chains ($\geq C_{20:0}$). Other fatty acids are present in low amounts, such

as: 1) odd-chain fatty acids ($C_{11:0}$-$C_{25:0}$) including samples with long carboxyl chains (≥$C_{17:0}$); and 2) even (C_{14Br}-C_{20Br}) and odd (C_{15Br}-C_{21Br}) branched-chain fatty acids with C_{15Br} and C_{17Br} being the most frequent. A third of the samples contain unsaturated fatty acids ($C_{18:1}$, $C_{16:1}$, and some with $C_{18:2}$, $C_{20:1}$, or $C_{23:1}$). Full details are provided in Appendix 4.

Cholesterol derivatives constitute the major part of the preserved sterols in comparison to plant sterols. Many sherds 'T' contained monoacylglycerols (MAGs), diacylglycerols (DAGs), and triacylglycerols (TAGs), with a third of them containing well-preserved TAGs.

The most frequent profiles have saturated even- (majority $C_{16:0}$ and $C_{18:0}$) and branched-chain saturated fatty acids (C_{15Br}-C_{17Br}) with rarer unsaturated fatty acids, cholesterol derivatives, and TAGs. The TAG profiles are typical of the presence of ruminant fats: T_{42}-T_{54}, dominated by T_{52} (Dudd & Evershed 1998; Dudd 1999), and degraded dairy fats with a broader distribution, T_{40}-T_{54} (Fig. 3A; Mirabaud *et al.* 2007; Regert 2011).

Odd, asymmetrical long-chain ketones (K_{29}-K_{35}), formed by condensation of fatty acids ($C_{16:0}$, $C_{18:0}$ and $C_{20:0}$), and indicating heating of the containers and their contents to high temperatures or for an extended period (Evershed *et al.* 1995; Raven *et al.* 1997), were observed in a total of 8 vessels (e.g. MR6553 in Fig. 3C; Appendices 5-7).

Wax esters (E_{40}-E_{52}) were detected in a few samples, as well as *n*-alkanes (often AL_{25}-AL_{33}) and *n*-alcohols (OH_{20}-OH_{32}), the latter, potentially derived from ester hydrolysis (Regert *et al.* 2001) were detected in several samples (full details in Appendices 3; 10-12). Hydroxy esters (H_{46}-H_{52}) were present in a single sample (MR6558T, Fig. 3F).

The 30 visible charred residues ('RI'/'RE') did not have high lipid survival, and most had lipid profiles consisting of free fatty acids ($C_{16:0}$-$C_{18:0}$). A single 'RE' demonstrated the preservation of MAGs, DAGs and TAGs (MR6521RE), while another (MR6506RE) had odd-chain *n*-alkanes, *n*-alcohols and wax esters ranging between C_{42}-C_{50} with a higher proportion of palmitic over stearic, indicating a possible mixture of different waxes (Fig. 3D, E).

Extracts of surface coatings/layers ('SE'/'SI') were dominated by even-chain free-fatty acids ($C_{14:0}$-$C_{26:0}$), but some samples also contained odd-chain fatty acids ($C_{13:0}$-$C_{23:0}$), branched-chain fatty acids (C_{15Br}-C_{21Br}), and unsaturated fatty acids ($C_{18:1}$ or $C_{20:1}$ and $C_{23:1}$). Phytanic acid, MAGs, DAGs and TAGs, as well as *n*-alkanes, alcohols and wax esters were detected in a smaller subset of samples. TAGs are present in both SIs and SEs and represent between 1.0-26.4% of the TLE. Esters were detected in 3 samples, representing between 3.1-8.6% of the TLE. At least five SEs and some SIs show saturated TAG profiles which are typical of the presence of ruminant dairy fats (T_{42}-T_{54}, dominated by T_{50} (Dudd & Evershed 1998; Dudd *et al.* 1999), as well as *n*-alkanes, *n*-alcohols and wax esters (E_{40}-E_{48}, majority composed of $C_{16:0}$), suggesting that the organic component of the layer was a mixture of animal fats (possibly dairy products) and waxes (see Appendices 4; 10-12). For three vessels (MR6516T/SE/SI, MR6517T/SE/SI and MR6520T/SE/SI), the external and internal coating layers were more lipid enriched and better preserved than the central part of the sherd (see Vessel use and technology and Fig. 6).

Evidence of plant products and contamination from sediment

Eight samples (MR6506T, MR6516SE, MR6517SE, MR6534T, MR6538T, MR6541T, MR6544T, MR6558T) had sufficient concentrations to characterise the composition of the ester profile. Most of the esters are derived from palmitic and stearic acids. The distribution of these wax esters, even-numbered compounds between 40 and 52 carbon atoms, may be consistent with those of beeswax. However, unlike beeswax, which contains palmitic ester moieties almost exclusively (Regert *et al.* 2001; Roffet-Salque *et al.* 2015; Tarifa-Mateo *et al.* 2021), detailed examination of their mass spectrum shows that their composition is more complex than that of beeswax, with peaks at *m/z* 201 ($C_{12:0}$), 229 ($C_{14:0}$), 257 ($C_{16:0}$), 285 ($C_{18:0}$), 313 ($C_{20:0}$), 341 ($C_{22:0}$), 369 ($C_{24:0}$) and 397 ($C_{26:0}$) (see Appendices 10-12). Here, this complex composition of wax esters indicates either vegetable epicuticular waxes or mixtures with beeswax, as discussed in other Neolithic contexts (Drieu *et al.* 2018).

In parallel, plant sterols (β-sitosterol and undetermined phytosterol; four vessels), different kinds of betulin derivatives, lupeol derivatives (three samples; two vessels) and terpenoids were also occasionally identified. The presence of unusual compounds in 20% of the sherds and some amides made it necessary to carry out a control analysis on sediments well after the sherds had been excavated and analysed (see Appendices 1-4; 12). The results obtained show that these unusual compounds are indeed present in the sediment (see Appendix 12). Both compounds are not commonly found in archaeological contexts and were randomly detected in the assemblage, sometimes in an unexplained high quantity. Consequently, they have been excluded from our interpretations and considered as contaminants. In contrast, wax esters, *n*-alkanes and *n*-alcohols, were mainly found within the absorbed lipids in the clay matrix and rarely in surface/coating layers or visible charred residues. Thus, they are not considered as contaminants. It's assumed that if they came from the surrounding sediment they would have been detected in the visible charred residues and in the other surface layers analysed, in higher concentrations and systematically associated with other soil contaminants. Furthermore, the ester distribution in the archaeological samples was more often restricted than the one in the sediment (Appendix 12).

Measurements of pH, electrical conductivity (CE), redox potential (Eh) and total dissolved solids (TDS) were performed on the same sediment. These measurements reveal that the sedimentary context is weakly acidic (pH = 6.8) with a tendency for water saturation or soil compaction (Eh = –46 mV). The conductivity is too low to effectively support vegetation growth (CE = 192 µS). The soil is low in salts and dissolved solids (TDS = 88.9 ppm) (see Appendix 2 for details on the method). These values are typical of a leached soil, a condition that could explain the infiltration of certain molecules into the matrix of the ceramics.

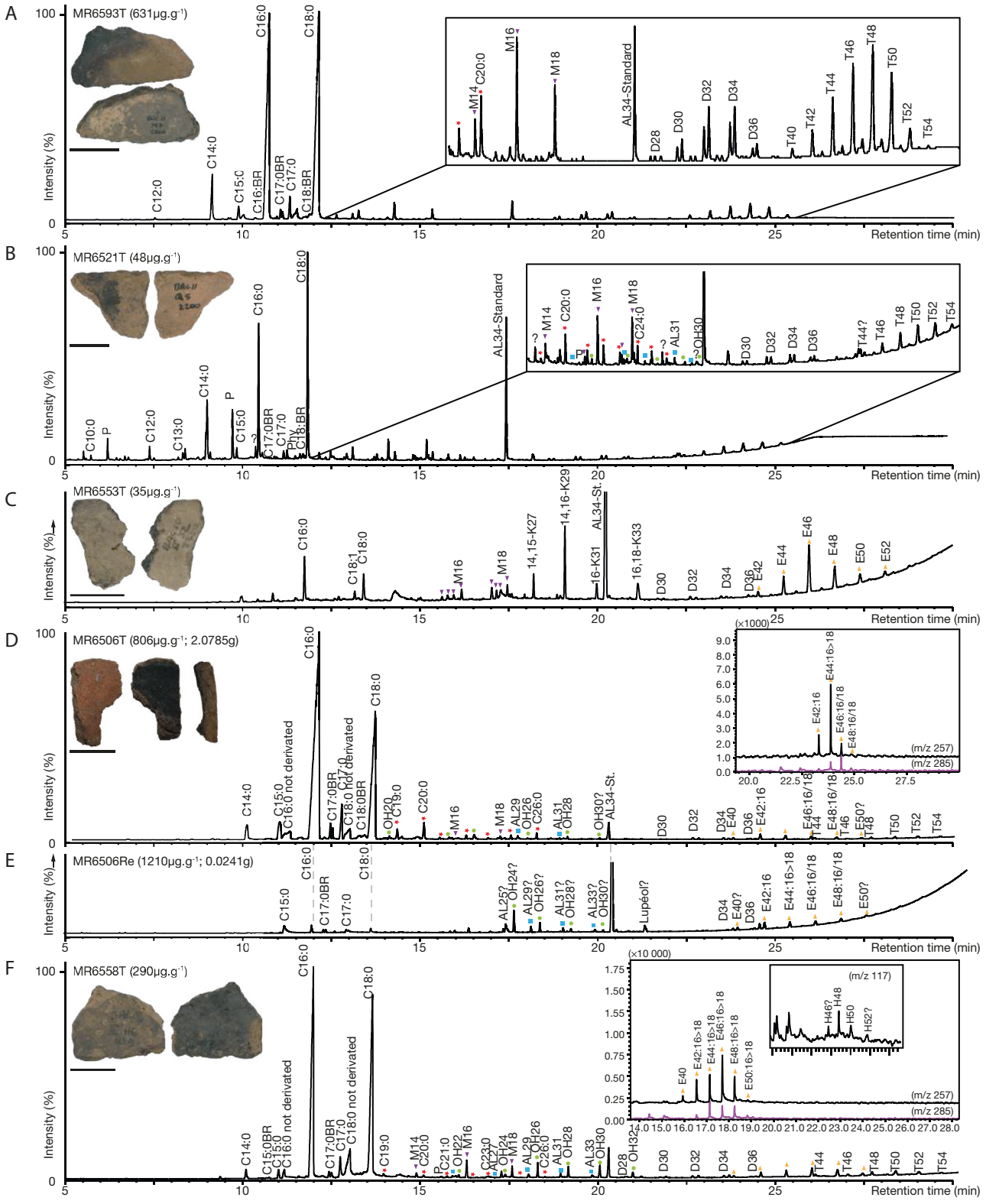


FIG. 3. — Chromatograms of TMS lipid extracts (obtained by conventional solvent extraction) of vessels from Beg ar Loued that demonstrate: **A**, the presence of animal fats with the survival of TAGs indicating the possible presence of dairy products; **B**, mixtures of substances including animal fats (possibly ruminant carcass) and degraded unspecific wax; **C**, the presence of an unspecific wax with evidence of heat treatment (mid-chain ketones); **D**, **E**, are chromatograms of samples MR6506T and MR6506Re, an absorbed residue and external visible surface residue that indicate the presence of wax (possible mixture of plant wax and beeswax) on the external surface: **D** includes (inset) a partial fragmentogram for the *m/z* 257 and *m/z* 285 ions showing the relative proportion of palmitate vs stearate esters characterising the wax; **F**, is a compilation of the chromatogram and the partial fragmentograms for the *m/z* 257, *m/z* 285 and *m/z* 117 ions of sample MR6558T, which shows the most significant evidence concerning potential beeswax. Abbreviations: **AL**, *n*-alkanes (blue squares); **Cn:x**, fatty acids with n carbon atoms and x double bonds (**red star**); **D**, diacylglycerols; **E**, wax esters (**yellow triangles**); **K**, ketones; **M**, monoacylglycerols (inverted purple triangles); **OH**, *n*-alcohols (**green dots**); **T**, triacylglycerols. Scale bars: 1 cm. Credits: sherds photographed by C. Prévost; CAD, C. Prévost.

Detection of aquatic products

Some solvent-extracted samples had traces of phytanic acid, found in aquatic products and ruminant fats, as well as long chains of saturated and unsaturated fatty acids, found in aquatic products and plant material (Cramp & Evershed 2014; Bondetti *et al.* 2021). Thus, none of the samples provided clear aquatic markers.

To better detect aquatic biomarkers, we conducted 31 acidified methanol extractions and analysed via SIM mode to search for isoprenoid fatty acids and APAAs (see Sample-set and analytical strategy; Appendix 2). The combination of pristanic (PRI), phytanic (PHY) and 4,8,12-trimethyltridecanoic acid (TMTD) indicates the presence of aquatic products, of which TMTD is considered to be a specific aquatic biomarker (Cramp & Evershed 2014; Lucquin *et al.* 2016b). The ratios of diastereomers of phytanic acid, 3S,7R,11R,15-phytanic (SRR) and 3R,7R,11R,15-phytanic acid (RRR), also provide an indication of the origin of phytanic acid, distinguishing ruminant fats from aquatic products via SRR% >70% (Lucquin *et al.* 2016a). Additionally, ω-(o-alkylphenyl) alkanoic acids (APAAs) ranging from C_{16} to C_{22}, attest to the heating of aquatic resources above 270°C (Cramp & Evershed 2014). $APAAs_{C18}$ can also occur in ruminant products (Cramp & Evershed 2014; Lucquin *et al.* 2016a).

The 31 samples extracted with acidified methanol contained saturated fatty acids ($C_{8:0}$/$C_{10:0}$-$C_{24:0}$/$C_{26:0}$), branched-chain fatty acids ($C_{14:0Br}$/$C_{15:0Br}$-$C_{17:0Br}$) and PHY, while 20 of them also contained PRI and 8 possibly contained TMTD (MR6506TH, MR6532TH, MR6534TH, MR6541TH, MR6545TH, MR6573TH, MR6586TH, MR6593TH) (Fig. 4A, B). The identification of TMTD in the samples was challenging due to coelution with another compound.

Four samples (MR6506TH, MR6532TH, MR6534TH and MR6586TH) had SRR% >70% (Fig. 4C; Appendix 4).

Five samples (MR6560TH, MR6573TH, MR6583, MR6593TH, MR6599TH) had enough concentrations of ($APAAs_{C18}$), to calculate a part of the distribution of the profile, out of which only one (MR6560TH) presented a complete distribution (Fig. 4D).

One sample (MR6534TH) contained the most of the criteria mentioned (PRI, PHY (SRR%: 75%), TMTD and traces of $APAA_{C18}$), without complete evidence for the heating markers $APAA_{C18\text{-}C20}$, and three other samples had SRR ratio >70% also indicative of the putative presence of aquatic products.

Identification of dairy products and ruminant meat

Several solvent-extracted samples suggested the presence of degraded ruminant fats, and particularly dairy products, as evidenced by saturated fatty acids, cholesterol derivatives, MAGs, DAGs and TAG profiles ($T_{40/42}$-T_{54}; Regert 2011).

To refine the characterisation of ruminant products and expand the information available for less well-preserved samples, we measured the stable carbon isotope ($\delta^{13}C$) values of two main saturated fatty acids ($C_{16:0}$ and $C_{18:0}$) in 24 samples (Craig *et al.* 2012). The differential routing of dietary carbon and fatty acids during the synthesis of adipose and dairy fats in ruminant animals enables ruminant milk fats to be distinguished from carcass fats by calculating $\Delta^{13}C$ values ($\delta^{13}C_{18:0} - \delta^{13}C_{16:0}$) and plotting them against $\delta^{13}C_{16:0}$ (Dudd & Evershed 1998; Copley *et al.* 2003).

Of the 24 solvent-extracted samples prepared for CSIA, eight samples had lower lipid concentrations with only fatty acids, while sixteen had between 1.1 – 14.7 TAG% of the TLE, with profiles indicative of ruminant carcass/dairy products. Of all samples analysed, 17 had $\Delta^{13}C$ values ranging from –6.4‰ to –3.1‰, indicative of dairy products (e.g. Dudd & Evershed 1998; Copley *et al.* 2003), and two (MR6605T and MR6618T) had $\Delta^{13}C$ values ranging from –1.8‰ to –2‰, suggestive of ruminant carcass fats (Fig. 5; see also Appendix 4). Four samples have values that do not fall within established references and may be the product of mixtures (MR6507T, MR6516SI, MR6527T, and MR6575T).

A single sample had enriched ^{13}C values for $C_{18:0}$, which may indicate marine input, but had a $\Delta^{13}C$ value of –2.9‰, which falls within the range of ruminant fats (MR6545T). This sample has the possible presence of TMTD, and traces of PRI, PHY (SRR%: 61%) and $APAAs_{C18}$, which may explain the stearic acid enrichment of the ^{13}C. As the sample only has traces of aquatic markers and SRR% indicates ruminant products, it might be a mixture of mainly ruminant meat and marine products.

DISCUSSION

The preservation of lipids in potsherds from the Beg ar Loued, particularly TAGs, is exceptional, although lipid yields are low and lipids from external residues appear to have been leached. The content of ceramic vessels from Beg ar Loued is often the result of mixtures of several products but dominated by terrestrial animal fats (Table 3). The combination of molecular and isotopic data clearly indicates that most of these fats come from ruminant carcass, and/or dairy products. Despite the island context of the site, the incorporation of aquatic products in pottery is debatable. Plant input, possibly mixed with beeswax in a few samples, is also suspected, usually associated with animal fats (details in Appendix 4).

Combined with zooarchaeological information available at the site, we now discuss the food systems and cooking practices at Beg ar Loued. The use of pottery and the role of organic products for coating layers in the manufacture of pottery is also hypothesised.

Pottery used for processing ruminant carcass and dairy products

Domestic cattle, pig and sheep/goat are first reported on the islands of the Iroise Sea off the coast of Brittany in the 4th millennium BC (Hanot & Tresset 2019). At Beg ar Loued, zooarchaeological data suggest breeding of sheep/goat at the site (Hanot & Tresset 2019). Slaughter curves show that sheep/goat were likely exploited for their milk;

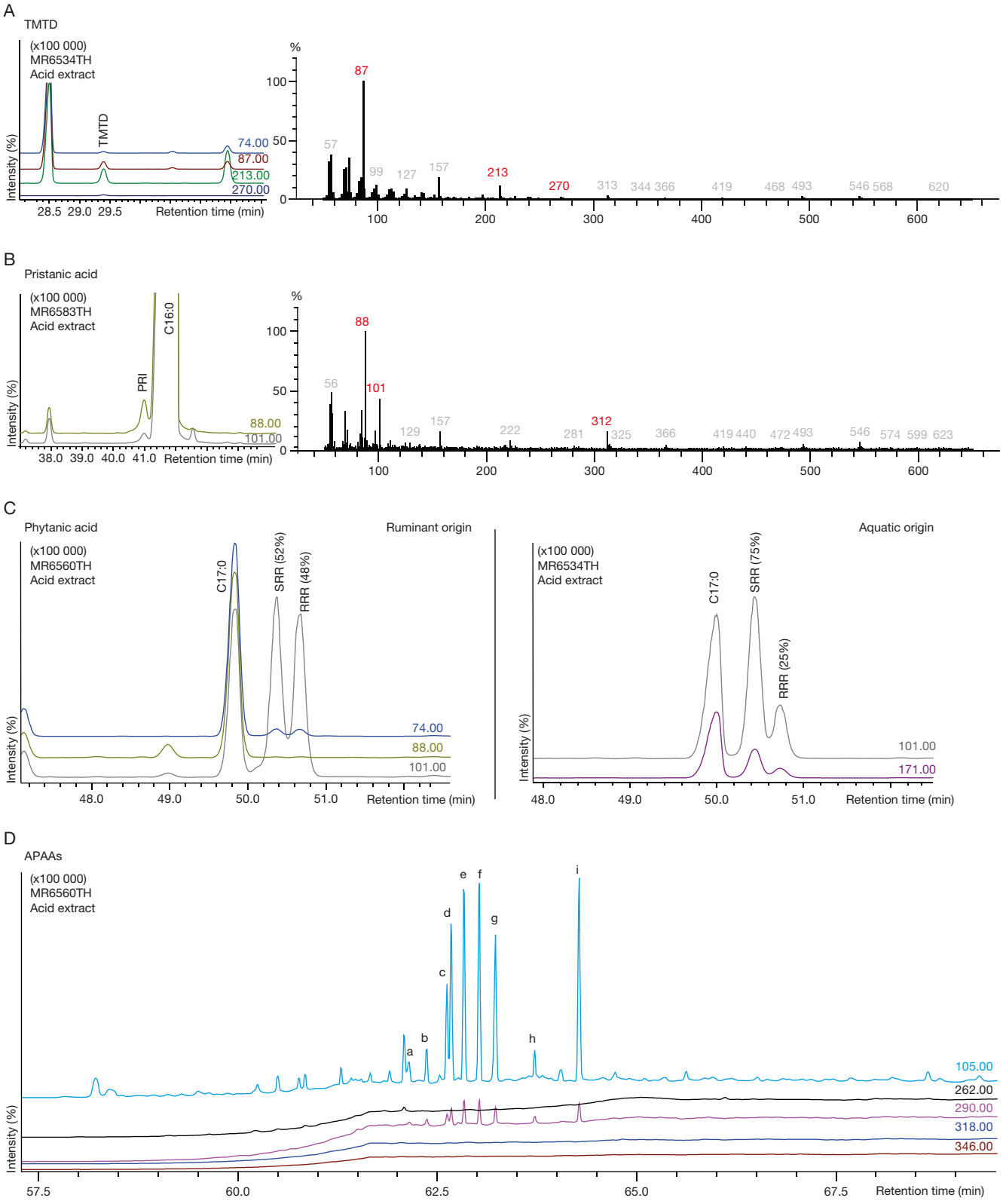


FIG. 4. — Partial chromatograms of acid-extracted samples of vessels from Beg ar Loued that demonstrate the presence of: **A**, 4,8,12-trimethyltridecanoic acid (TMTD) (mass spectra on the right); **B**, pristanic acid (mass spectra on the right); **C**, phytanic acid showing both ruminant (**left**) and aquatic (**right**) origin based on SRR%; **D**, ω-(o-alkylphenyl) alkanoic acids (APAAs), with only $APAAs_{C18}$ (*m/z* 290) detected. Credits: CAD, C. Prévost and A. Suryanarayan.

TABLE 3. — Overview of the molecular characterisation and products/biomarkers identified in vessels from Beg ar Loued.

Products and biomarkers identified	No.	%
Ketones (heat markers)	8	–
Commodities		
Animal (ruminant)	19	–
Animal (ruminant); plant (plant wax)	3	–
Animal (ruminant); plant (resin); Wax (undet.)	1	–
Animal (ruminant); wax (undet.)	5	–
Animal (ruminant)	28	27.2
Animal (dairy)	27	–
Animal (dairy); wax (undet.)	7	–
Animal (dairy)	34	33.0
Animal (undet.)	13	–
Animal (undet.); wax (undet.)	2	–
Animal (undet.); wax (undet.); plant (resin)	1	–
Animal (undet.)	16	15.5
Animal	**78**	**75.7**
Plant (resin); animal (undet.); wax (undet.)	1	–
Plant (resin); animal (ruminant); wax (undet.)	1	–
Plant (resin)	2	1.9
Plant (plant wax); animal (ruminant)	3	–
Plant (plant wax)	2	–
Plant (plant wax)	5	4.9
Plant	**7**	**6.8**
Wax (undet.)	2	–
Wax (undet.); animal (undet.)	2	–
Wax (undet.); animal (dairy)	7	–
Wax (undet.); animal (ruminant)	5	–
Wax (undet.); animal (ruminant); plant (resin)	1	–
Wax (undet.); animal (undet.); plant (resin)	1	–
Wax (undet.)	18	17.5
Wax (undet.)	**18**	**17.5**
Products and mixtures identified in 174 samples (excluding heat markers)	**103**	**100.0**

unlike cattle and pigs, which would have been exploited for their meat (Hanot & Tresset 2019).

The content of ceramic vessels on the site of Beg ar Loued was dominated by the processing or use of animal fats (n = 79/174; 46%; which comes to 70/129 vessels, 54%). The combination of molecular and isotopic data clearly indicates that most of these fats came from ruminants (n = 28/174; 16%; 28/129 vessels; 21%) and that dairy (n = 34/174; 19%; 30/129 vessels; 23%) made up a significant proportion of these animal products. Mid-chain ketones (K_{31} to K_{35}; Raven *et al.* 1997) are occasionally found in vessels with ruminant carcass and dairy products, suggesting the continuous heating of these products in vessels (Evershed *et al.* 1995).

Despite the site's close relationship with the sea, this data confirms the importance of dairying as a subsistence strategy on the site (Table 3; Appendix 4). This suggests that the site was part of a cultural landscape of pastoral societies on the Atlantic coast, established at least by the Early Neolithic (Cubas *et al.* 2020; Evershed *et al.* 2022) and continuing into the Bronze Age, as evident from sites across the channel (Copley *et al.* 2003; Šoberl 2011).

WAS POTTERY USED FOR PROCESSING AQUATIC PRODUCTS?

Zooarchaeological analyses have demonstrated that the populations of Beg ar Loued exploited a wide range of coastal resources, especially during summer for fishing gilthead bream, as well as the collection of limpets close to the site (Pailler & Nicolas 2019a). It has been suggested that fish was stored for long-term consumption by drying, smoking and/or salting (Dréano 2019).

However, lipid and isotopic data do not provide a clear indication of the exploitation of aquatic resources in ceramics. Among the 31 acid-extracted samples, some of the vessels have partial biomarkers for aquatic products. TMTD, the isoprenoid marker specific for aquatic products when associated with PRI and PHY, co-eluted with another compound, but may be present in low amounts in only eight vessels. Fatty acid isotopic values for only one of these (MR6545T) are available, which demonstrates slightly enriched ^{13}C values for stearic acid, suggesting a mixture of mainly ruminant meat and marine products (as suggested in Identification of dairy products and ruminant meat). The SRR%>70% for 4 vessels may indicate the aquatic origin of phytanic acid and therefore an input of fish into the ceramics. None of the samples contained APAAs in significant quantities and they were restricted to the non-specific $APAAs_{C18}$ (see Evidence of plant products and contamination from sediment; Cramp & Evershed 2014; Bondetti *et al.* 2021), which suggests that aquatic products were not heated in vessels.

Pending further detection of aquatic products, the results of this study suggest minimal use of ceramics to process aquatic products at Beg ar Loued. These findings support that the settlement at Beg ar Loued was linked to seasonal fishing activities, for the stocking of dried/smoked/salted fish (aceramic process), while shellfish could be eaten fresh or cooked on stone on-site (Dupont 2019; Dréano 2019; Mougne 2019; Pailler & Nicolas 2019a). The pattern observed at Beg ar Loued appears similar to those studied at Bronze Age sites in Britain and Scotland, where fishing did not play a central role in the subsistence economy (Craig *et al.* 2000; Copley *et al.* 2003; Richards *et al.* 2003; Šoberl 2011; Baeten *et al.* 2013). Such a subsistence strategy is unique when compared to other areas of the Baltic coast, where lipids studied in ceramics reveal the integration of pottery in the processing and consumption of aquatic resources at sub-Neolithic and Bronze Age sites (Craig *et al.* 2011; Cramp & Evershed 2014; Cramp *et al.* 2014a, b; Heron *et al.* 2015; Robson *et al.* 2019).

VESSEL USE AND TECHNOLOGY

Given the fragmentary nature of the ceramic assemblage at Beg ar Loued, the reconstruction of the forms of vessels has been limited. Available evidence from the location of the sherd on the vessel (rim, base, body; see Appendix 7) did not allow for a reconstruction of the gradient of lipid concentration in order to make inferences about specific culinary activities such as boiling or roasting (Drieu *et al.* 2022).

The presence of mid-chain ketones (K_{29}-K_{35}) formed by the condensation of free fatty acids provides an indication of vessel use for heating (Raven *et al.* 1997). Mid-chain

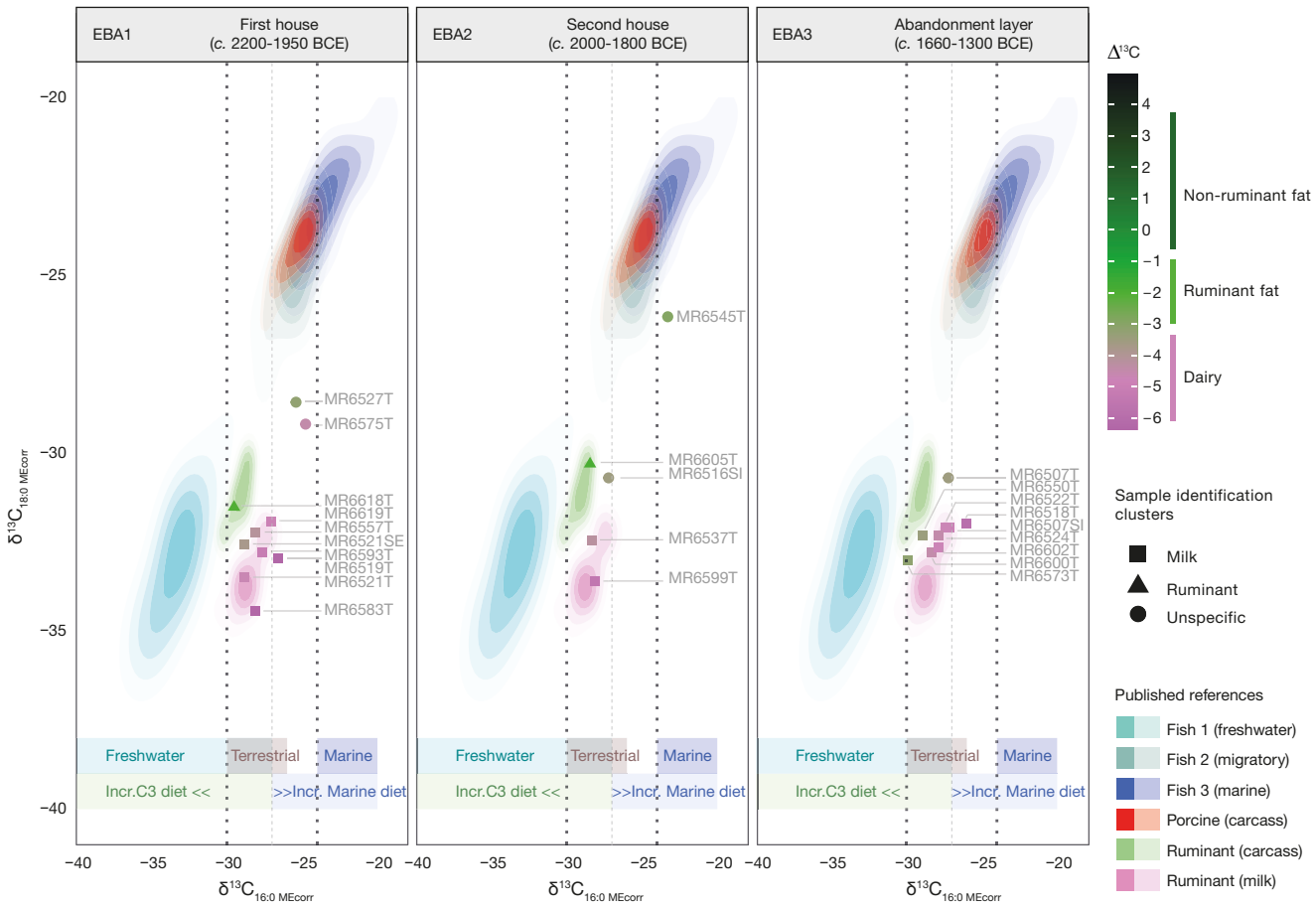


FIG. 5. — $\delta^{13}C$ values (**squares**, **circles** and **triangles**) for methylated fatty acids ($C_{16:0}$ and $C_{18:0}$) from Beg ar Loued compared with established modern references (gradient ellipses with centroid). The **colour scale** provides an indication of the $\Delta^{13}C$ value: we use accepted threshold values to distinguish dairy (–3.3‰<$\Delta^{13}C$), ruminant (3.3‰<$\Delta^{13}C$<–1.0‰) and non-ruminant fats ($\Delta^{13}C$>–1.0‰) (Dudd 1999; Outram *et al.* 2009; Craig *et al.* 2011; Cramp *et al.* 2014b; Taché & Craig 2015; Colonese *et al.* 2015; Horiuchi *et al.* 2015; Choy *et al.* 2016; Lucquin *et al.* 2016b; Liu *et al.* 2017; Courel *et al.* 2020b; Pääkkönen *et al.* 2020). R Markdown file in Appendix 13. Credits: R code and CAD, C. Prévost.

ketones were mostly found absorbed in ceramic matrices (see Appendices 5; 6), and for some of the samples, as the sherds do not have charred visible encrustations, they probably result from repeated use of vessels rather than long heating periods (*ibid.*). Moreover, they are found rather at the level of the body and absent from the bases (see Appendix 7). Unlike other studies on cooking pots, where mid-chain ketones are generally found at the bottom or on the rims of containers (Duplaix-Rata 1997; Evershed 2008); here the pattern is slightly more consistent with the boiling of half-filled containers. Visible charred residues are more often found on body fragments of pots (21 of 27 samples; 78%) with walls with medium thickness (median of the average thickness *c.* 7 cm; Appendices 5; 6). These observations suggest that vessels may have been heated laterally rather than centrally or suspended over a fire, as it was reported in a recent study of ethnological vessels used for indirect/steam cooking (Drieu *et al.* 2022).

Investigations into surface treatments of pottery on the Atlantic coast of France are mostly limited to the examination of ceramic thin sections or vessel surface traces (Convertini & Querré 1998; Convertini 2019). Very little literature exists on the chemical determination of surface treatments in general for European prehistoric contexts (Drieu *et al.* 2020). Our preliminary results on six potsherds present one of the first investigations of the organic component of vessels coating layers (*engobe*) in the region that we know of.

The surface/coating layers ('SE'/'SI') had higher concentrations of lipids than those absorbed in the potsherd ('T') or the visible carbonised residues ('RE'/'RI') (e.g. MR6507, MR6520).

The gradient of lipid accumulation and composition indicates that organic components in the external layers are often not related to the contents of the vessel, and they are not analogous to the surrounding sediments (see Fig. 6; Appendices 4C-6; 12).

One can note that coating layers are mainly identified on vessels with thin walls (*c.* 5 cm; see Appendix 5). Heat markers (charring, visible residues and ketones) are not detected on these fragments, indicating that these might constitute an assemblage distinct from cooking pots. In most of the surface/coating layers investigated, wax/plant biomarkers (esters, *n*-alkanes, *n*-alcohols) were identified together with the typical composition of animal fats (see Appendix 4).

As an example, the external layer, internal layer and absorbed residue of the sample (MR6517) had similar lipid concentrations (*c.* 200 µg.g^{-1}; see Fig. 6). Degraded wax

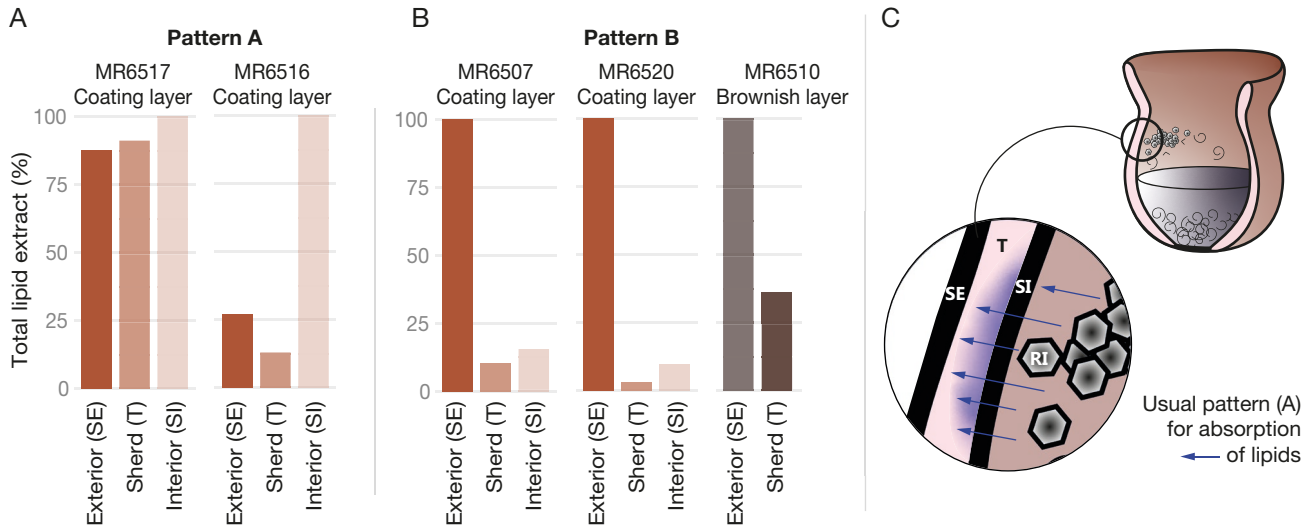


Fig. 6. — **A**, **B**, Histograms of the total lipid extract (%) from selected samples with separated brownish surface layer (MR6510) and red/white coating layers (MR6507, MR6520, MR6516, MR6517); **C**, scheme presenting the usual pattern of absorption of lipids in vessels. **A**, Pattern A fits the distribution of lipid impregnation through the vessel profile generally observed in archaeological pottery; **B**, pattern B shows a unique distribution of lipid impregnation supporting the use of fatty substances in ceramic finishing processes for samples from Beg at Loued. R Markdown file available in Appendix 13. Abbreviations: **SE**, external surface/coating layer; **SI**, internal surface/coating layer; **T**, sherd. Credits: R code and CAD, C. Prévost and A. Suryanarayan.

and potential dairy products were found in the external red lustrous layer (MR6517SE), while the internal layer (MR6517SI) only supports evidence for potential dairy products. The absorbed residue (MR6517T) showed free fatty acids, presumably from animal fats. This suggests that the products in the outer layer are not related to the contents of the vessel. Other examples suggest the presence of ruminant carcass fats and waxy products in red lustrous external coating layers (e.g. MR6507SE, MR6520SE).

Overall, as these pottery fragments have no heat markers (visible residues or ketones); show particular investment in the finishing treatment; and a specific gradient of lipid impregnation, this evidence suggests that waxes and/or animal grease were used for vessel finishing treatment, or constituted the organic component of vessel coating layers, particularly for those with red lustrous external layers. In these cases, the adding of wax may have been used to obtain the lustrous finish rather than to act as a vessel sealant or as culinary input. The esters detected do not make it possible to clearly determine wax sources. Plant waxes were probably used, but still need further investigation. Meanwhile, knowledge about the use of beehive products in Europe was known long before the occupation of Beg ar Loued (Regert *et al.* 2001; Evershed *et al.* 2008; Salque *et al.* 2013; Roffet-Salque *et al.* 2015, 2016; Drieu *et al.* 2021; Tarifa-Mateo *et al.* 2021). The use of beeswax is well attested in southern England in vessels from the Collared Urns (*c.* 2200-1780 BCE) at the site of Gayhurst Barro (Šoberl 2011), but as far as we know, there are no contemporary published examples on the Atlantic coast. It is possible that inhabitants of Molène may have used beeswax mixed with other plant waxes while finishing vessels so as to improve the mechanical strength of the induction, or to make up for a lack of sufficient quantities during the manufacture of pottery.

CONCLUSION AND OUTLOOK: FROM POTTERY CONTENT TO COOKING AND CULINARY PRACTICES

The lipid residue results presented here facilitate a new understanding of resource exploitation, vessel use and culinary practices at Beg ar Loued, a unique island settlement off the northwest French Atlantic coast. Our study investigates lipids in ceramics from the Final Neolithic and Early Bronze Age, filling a gap in lipid residue research in this region, which has mostly focused on the Early Neolithic period.

The exceptional lipid preservation in the ceramics, evidenced by the presence of TAGs and esters in several samples, indicates that burial environment conditions (leached and fairly acidic soils) in Brittany likely enable the survival of lipids in pottery.

Domestic ruminants make up an essential component of the products being processed in vessels. The strong evidence of dairy products in vessels highlights that pastoral lifeways were adopted by the inhabitants of the island, much like other populations along the Atlantic coast (Cubas *et al.* 2020; Evershed *et al.* 2022). Plant biomarkers are also occasionally identified but their origin is often difficult to establish with certainty due to their low amount in ceramic vessels and their presence in the sediment control sample.

Despite the site's island context and close relationship with the sea, minimal evidence for the processing of aquatic products in the ceramics is detected, and the extent of their use in vessels remains an open question. This pattern fits with the general lack of evidence of fish in vessels observed along the Atlantic coast in the Neolithic period (Cubas *et al.* 2020). This perhaps indicates the continuity of specific culinary preferences over time and specific local choices for the processing of fish without the use of ceramics, as demonstrated by the

diversity of marine fauna present at the site. Together with the material evidence and the ceramic lipid residue results, it is clear that populations at Beg ar Loued maintained an integrated land- and coastal-based subsistence economy, connecting both the mainland and the ocean.

Finally, the analyses reveal at least two categories of vessels: cooking pots with molecular evidence of heating, and vessels with coating layers or special finishing treatment, possibly linked to consumption or service of food commodities without any heating operation. The latter category has evidence of organic components such as mixtures of waxes and animal fats used for surface treatments.

Our study combines the data obtained from lipid residue results, characteristics of the ceramics, environmental data and faunal remains to contribute new aspects about culinary practices of prehistoric Atlantic coastal communities. Developing other studies using such multiproxy approaches in the future will be useful for studying natural resource management and culinary practices in greater depth. A focus on ceramics and their use will enable a better understanding of the role of pottery in food production and variabilities or links between different settlements across time.

Author contributions

Camielsa Prévost and Akshyeta Suryanarayan have contributed equally to this work.
Camielsa Prévost: conceptualization, formal analysis - lab prep and GC-FID and GC-MS and statistics, funding acquisition – GC-c-IRMS acquisitions, writing – original draft, visualization (Excel, R, and Illustrator).
Akshyeta Suryanarayan: formal analysis – lab prep and GC-FID and GC-MS and statistics, funding acquisition – GC-c-IRMS acquisitions, writing – original draft, visualization (Excel and R).
Thierry Blasco: formal analysis – GC-c-IRMS acquisitions.
Martine Regert: investigation – sampling, funding acquisition – GC-c-IRMS acquisitions, validation, review and editing.
Arnaud Mazuy: resources.
Clement Nicolas: investigation – archaeological field, review and editing.
Yvan Pailler: investigation – archaeological field, review and editing.
Pauline Hanot: investigation – zooarchaeological expertise, review and editing.
Catherine Dupont: investigation – zooarchaeological expertise, review and editing.
Yvon Dréano: investigation – zooarchaeological expertise, review and editing.

Acknowledgements

Financial support for analytical and isotopic analyses was obtained from the Université Côte d'Azur EUR ODYSSE CSI grant (project ISONEO). The authors sincerely thank the reviewers for their precise and constructive comments and suggestions.

REFERENCES

Anonymous 2015. — *On-site Testing of Growing Media and Irrigation Water*. Greenhouse, Factsheet, 7 p.

Anonymous 2018. — Saline Water and Salinity. Available from: https://www.usgs.gov/special-topics/water-science-school/science/saline-water-and-salinity?qt-science_center_objects=0#qt-science_center_objects (last accessed on 21 March 2023).

Baeten J., Jervis B., de Vos D. & Waelkens M. 2013. — Molecular evidence for the mixing of meat, fish and vegetables in Anglo-Saxon coarseware from Hamwic, UK. *Archaeometry* 55 (6): 1150-1174. https://doi.org/10.1111/j.1475-4754.2012.00731.x

Baize D. 2000. — *Guide des analyses en pédologie: choix, expression, présentation, interprétation. Techniques et pratiques*. Institut national de la recherche agronomique, Paris, 257 p.

Bernard V., Billard C., Clavel B., Ganne A., Guillon M., Herrscher E., Jaouen G., Oberlin C. & Werthe E. 2020. — Approches globales et synthèse sur les pêcheries de la fin du Néolithique et du début de l'âge du Bronze, *in* Billard C. & Bernard V. (eds), *Pêcheries de Normandie : archéologie et histoire des pêcheries littorales du département de la Manche*. Presses universitaires de Rennes: 357-404.

Billard C., Daire M. Y. & Martin C. 2020. — France: submerged prehistory on Atlantic and Mediterranean coasts, *in* Bailey G., Galanidou N., Peeters H., Jöns H. & Mennenga M. (eds) *The Archaeology of Europe's Drowned Landscapes. Coastal Research Library*. Vol. 35. Springer, Cham: 249-280. https://doi.org/10.1007/978-3-030-37367-2_12

Blanchet S., Levan M., Sicard S. & Toron S. 2018. — Les formes de l'habitat et l'occupation du sol à l'âge du Bronze en Bretagne, *in* Lemercier O., Sénépart I., Besse M. & Mordant C. (eds), *Habitations et habitat du Néolithique à l'âge du Bronze en France et ses marges. Actes des IIe Rencontres Nord/Sud de Préhistoire récente, Dijon, 19-21 novembre 2015*. Archives d'Écologie Préhistorique, Toulouse: 623-636.

Blanchet S., Favrel Q., Fily M., Nicolas C., Nicolas T., Pailler Y. & Ripoche J. 2019. — Le Campaniforme et la genèse de l'âge du Bronze ancien en Bretagne : vers une nouvelle donne ? *in* Montoya C., Fagnart J.-P. & Locht J.-L. (eds), *Préhistoire de l'Europe du Nord-Ouest. Mobilités, climats et identités culturelles. XXVII Congrès préhistorique de France (Amiens, 30 mai - 4 juin 2016)*. Vol. 3. Société Préhistorique Française, Paris: 269-288. https://hal-univ-rennes1.archives-ouvertes.fr/hal-02868518

Bond G. C., Showers W., Elliot M., Evans M. N., Lotti R., Hajdas I., Bonani G. & Johnsen S. 1999. — The North Atlantic's 1–2 kyr climate rhythm: relation to Heinrich events, Dansgaard/Oeschger cycles and the little ice age, *in* Clark P. U., Webb R. S. & Keigwin L. D. (eds), *Mechanisms of Global Climate Change at Millennial Time Scales*. Advancing Earth and Space Sciences, Washington, DC, : 35-68.

Bondetti M., Scott E., Courel B., Lucquin A., Shoda S., Lundy J., Labra-Odde C., Drieu L. & Craig O. E. 2021.— Investigating the formation and diagnostic value of ω-(o-alkylphenyl)alkanoic acids in ancient pottery. *Archaeometry* 63 (3): 594-608. https://doi.org/10.1111/arcm.12631

Braguier S. 2000. — *Économie alimentaire et gestion des troupeaux au Néolithique récent/final dans le centre-ouest de la France*. Thesis, Université Toulouse 2, 539 p.

Choy K., Potter B. A., Mckinney H. J., Reuther J. D., Wang S. W. & Wooller M. J. 2016. — Chemical profiling of ancient hearths reveals recurrent salmon use in ice age Beringia. *Proceedings of the National Academy of Sciences of the United States of America* 113 (35): 9757-9762. https://doi.org/10.1073/pnas.1606219113

Colonese A. C., Farrell T. F. G., Lucquin A., Firth D., Charlton S., Robson H. K., Alexander M. & Craig O. E. 2015. — Archaeological bone lipids as palaeodietary markers. *Rapid Communications in Mass Spectrometry* 29 (7): 611-618. https://doi.org/10.1002/rcm.7144

Convertini F. 2019. — Étude pétrographique de la céramique de Beg ar Loued, *in* Pailler Y. & Nicolas C. (eds), *Une maison sous les dunes : Beg ar Loued, île Molène, Finistère. Identité et adaptation des groupes humains en mer d'Iroise à la transition des IIIe et IIe millénaires avant notre ère*. Sidestone Press, Leiden: 353-366.

Convertini F. & Querré G. 1998. — Apports des études céramologiques en laboratoire à la connaissance du Campaniforme : résultats, bilan et perspectives. *Bulletin de la Société Préhistorique Française* 95 (3): 333-342. https://doi.org/10.3406/bspf.1998.10808

Copley M. S., Berstan R., Dudd S. N., Docherty G., Mukherjee A. J., Straker V., Payne S. & Evershed R. P. 2003. — Direct chemical evidence for widespread dairying in prehistoric Britain. *Proceedings of the National Academy of Sciences of the United States of America* 100 (4): 1524-1529. https://doi.org/10.1073/pnas.0335955100

Correa-Ascencio M. & Evershed R. P. 2014. — High throughput screening of organic residues in archaeological potsherds using direct acidified methanol extraction. *Analytical Methods* 6: 1330. https://doi.org/10.1039/c3ay41678j

Courel B., Robson H. K., Lucquin A., Dolbunova E., Oras E., Adamczak K., Andersen S. H., Astrup P. M., Charniauski M., Czekaj-Zastawny A., Ezepenko I., Hartz S., Kabaciński J., Kotula A., Kukawka S., Loze I., Mazurkevich A., Piezonka H., Piličiauskas G., Sørensen S. A., Talbot H. M., Tkachou A., Tkachova M., Wawrusiewicz A., Meadows J., Heron C. P. & Craig O. E. 2020a. — Organic residue analysis shows sub-regional patterns in the use of pottery by Northern European hunter-gatherers. *Royal Society Open Science* 7 (4): 192016. https://doi.org/10.1098/rsos.192016

Courel B., Robson H. K., Lucquin A., Dolbunova E., Oras E., Adamczak K., Andersen S. H., Astrup P. M., Charniauski M., Czekaj-Zastawny A., Ezepenko I., Hartz S., Kabaciński J., Kotula A., Kukawka S., Loze I., Mazurkevich A., Piezonka H., Piličiauskas G., Sørensen S. A., Talbot H. M., Tkachou A., Tkachova M., Wawrusiewicz A., Meadows J., Heron C. P. & Craig O. E. 2020b. — Erratum: Organic residue analysis shows sub-regional patterns in the use of pottery by Northern European hunter-gatherers. *Royal Society Open Science* 7 (7): 201168. https://doi.org/10.1098/rsos.201168

Craig O. E., Mulville J., Pearson P. M., Sokol R., Gelsthorpe K., Staceyll R. & Collins M. J. 2000. — Detecting milk proteins in ancient pots. *Nature* 408 (1): 312. https://doi.org/10.1038/35042684

Craig O. E., Chapman J., Heron C. P., Willis L. H., Bartosiewicz L., Taylor G., Whittle A. & Collins M. J. 2005a. — Did the first farmers of central and eastern Europe produce dairy foods? *Antiquity* 79 (306): 882-894. https://doi.org/10.1017/s0003598x00115017

Craig O. E., Taylor G., Mulville J., Collins M. J. & Parker Pearson M. 2005b. — The identification of prehistoric dairying activities in the Western Isles of Scotland: an integrated biomolecular approach. *Journal of Archaeological Science* 32 (1): 91-103 https://doi.org/10.1016/j.jas.2004.06.009

Craig O. E., Forster M., Andersen S. H., Koch E., Crombé P., Milner N. J., Stern B., Bailey G. N. & Heron C. P. 2007. — Molecular and isotopic demonstration of the processing of acquatic products in Northern European prehistoric pottery. *Archaeometry* 49 (1): 135-152. https://doi.org/10.1111/j.1475-4754.2007.00292.x

Craig O. E., Steele V. J., Fischer A., Hartz S., Andersen S. H., Donohoe P., Glykou A., Saul H., Jones D. M., Koch E. & Heron C. P. 2011. — Ancient lipids reveal continuity in culinary practices across the transition to agriculture in Northern Europe. *Proceedings of the National Academy of Sciences* 108 (44): 17910-17915. https://doi.org/10.1073/pnas.1107202108

Craig O. E., Allen R. B., Thompson A., Stevens R. E., Steele V. J. & Heron C. P. 2012. — Distinguishing wild ruminant lipids by gas chromatography/combustion/isotope ratio mass spectrometry. *Rapid Communications in Mass Spectrometry* 26 (19): 2359-2364. https://doi.org/10.1002/rcm.6349

Craig O. E., Saul H., Lucquin A., Nishida Y., Taché K., Clarke L., Thompson A., Altoft D. T., Uchiyama J., Ajimoto M., Gibbs K., Isaksson S., Heron C. P. & Jordan P. 2013. — Earliest evidence for the use of pottery. *Nature* 496: 351-354. https://doi.org/10.1038/nature12109

Cramp L. J. E. & Evershed R. P. 2014. — Reconstructing aquatic resource exploitation in human prehistory using lipid biomarkers and stable isotopes, *in* Holland H. D. & Turekian K. K. (eds), *Treatise on Geochemistry*. Second Edition. Vol. 14. Elsevier, Oxford: 319-339. https://doi.org/10.1016/B978-0-08-095975-7.01225-0

Cramp L. J. E., Evershed R. P., Lavento M., Halinen P., Mannermaa K., Oinonen M., Kettunen J., Perola M., Onkamo P. & Heyd V. 2014a. — Neolithic dairy farming at the extreme of agriculture in northern Europe. *Proceedings of the Royal Society B: Biological Sciences* 281 (1791): 1-9. https://doi.org/10.1098/rspb.2014.0819

Cramp L. J. E., Jones J., Sheridan A., Smyth J., Whelton H. L., Mulville J., Sharples N. & Evershed R. P. 2014b. — Immediate replacement of fishing with dairying by the earliest farmers of the northeast Atlantic archipelagos. *Proceedings of the Royal Society B: Biological Sciences* 281 (1780): 1-8. https://doi.org/10.1098/rspb.2013.2372

Cramp L. J. E., Ethier J., Urem-Kotsou D., Bonsall C., Borić D., Boroneant A., Evershed R. P., Perić S., Roffet-Salque M., Whelton H. L. & Ivanova M. 2019. — Regional diversity in subsistence among early farmers in Southeast Europe revealed by archaeological organic residues. *Proceedings of the Royal Society B: Biological Sciences* 286 (1894): 20182347. https://doi.org/10.1098/rspb.2018.2347

Cubas M., Lucquin A., Robson H. K., Colonese A. C., Arias P., Aubry B., Billard C., Jan D., Diniz M. & Fernandes R. 2020. — Latitudinal gradient in dairy production with the introduction of farming in Atlantic Europe. *Nature Communications* 11: 2036. https://doi.org/10.1038/s41467-020-15907-4

David R. 2014. — *Modélisation de la végétation holocène du Nord-Ouest de la France : Reconstruction de la chronologie et de l'évolution du couvert végétal du Bassin parisien et du Massif armoricain.* Thesis, Université de Rennes 1, 279 p.

Debono Spiteri C. 2012. — *Pottery Use at the Transition to Agriculture in the Western Mediterranean: Evidence from Biomolecular and Isotopic Characterisation of Organic Residues in Impressed/Cardial Ware vessels*. Thesis, University of York, 326 p.

Debono Spiteri C., Gillis R. É., Roffet-Salque M., Castells Navarro L., Guilaine J., Manen C., Muntoni I. M., Saña Segui M., Urem-Kotsou D., Whelton H. L., Craig O. E., Vigne J.-D. & Evershed R. P. 2016. — Regional asynchronicity in dairy production and processing in early farming communities of the northern Mediterranean. *Proceedings of the National Academy of Sciences* 113 (48): 13594-13599. https://doi.org/10.1073/pnas.1607810113

Delaune R. D., Reddy C. N. & Patrick Jr W. H. 1981. — Organic matter decomposition in soil as influenced by pH and redox conditions. *Soil Biologie Biochemistry* 13: 533-534.

Dréano Y. 2019. — La pêche des poissons, *in* Pailler Y. & Nicolas C. (eds), *Une maison sous les dunes : Beg ar Loued, île Molène, Finistère. Identité et adaptation des groupes humains en mer d'Iroise à la transition des IIIe et IIe millénaires avant notre ère*. Sidestone Press, Leiden: 488-514.

Dréano Y., Gandois H. & Pailler Y. 2013. — L'exploitation des poissons dans l'archipel de Molène (Finistère, France) du Néolithique récent à l'Âge du Bronze ancien, *in* Daire M.-Y., Dupont C., Baudry A., Billard C., Large J.-M., Lespez L., Normand E. & Scarre C. (eds), *Anciens peuplements littoraux*

et relations Homme/Milieu sur les côtes de l'Europe atlantique/ Ancient maritime communities and the relationship between people and environment along the European Atlantic coasts. Proceedings of the HOMER 2011 Conference, Vannes (France), 28/09-1/10/2011. Archaeopress, British Archaeological Reports (coll. International Series; 2570), Oxford: 447-458.

Driard C., Dreano Y. & Garnier N. 2017. — Les sauces de poisson produites sur la côte atlantique des Gaules : sources archéologiques et productions diversifiées des ateliers de salaison. *Gallia* 74 (2): 183-205.

Drieu L., Peche-Quilichini K., Lachenal T. & Regert M. 2018. — Domestic activities and pottery use in the Iron Age Corsican settlement of Cuciurpula revealed by organic residue analysis. *Journal of Archaeological Science: Reports* 19: 213-223. https://doi.org/10.1016/j.jasrep.2018.02.032

Drieu L., Lepère C. & Regert M. 2020. — The missing step of pottery *chaîne opératoire*: considering post-firing treatments on ceramic vessels using macro- and microscopic observation and molecular analysis. *Journal of Archaeological Method and Theory* 27 (2): 302-326. https://doi.org/10.1007/s10816-019-09428-8

Drieu L., Lucquin A., Cassard L., Sorin S., Craig O. E., Binder D. & Regert M. 2021. — A Neolithic without dairy? Chemical evidence from the content of ceramics from the Pendimoun rockshelter (Castellar, France, 5750-5150 BCE). *Journal of Archaeological Science: Reports* 35: 1-26. https://doi.org/10.1016/j.jasrep.2020.102682

Drieu L., Regert M., Mazuy A., Vieugué J., Bocoum H. & Mayor A. 2022. — Relationships between lipid profiles and use of ethnographic pottery: an exploratory study. *Journal of Archaeological Method and Theory* 29 (4): 1294-1322. https://doi.org/10.1007/s10816-021-09547-1

Dudd S. N. 1999. — *Molecular and Isotopic Characterisation of Animal Fats in Archaeological Pottery.* Thesis, University of Bristol, 448 p.

Dudd S. N. & Evershed R. P. 1998. — Direct demonstration of milk as an element of archaeological economies. *Science* 282 (5393): 1478-1481. https://doi.org/10.1126/science.282.5393.1478

Dudd S. N., Evershed R. P. & Gibson A. M. 1999. — Evidence for varying patterns of exploitation of animal products in different prehistoric pottery traditions based on lipids preserved in surface and absorbed residues. *Journal of Archaeological Science* 26 (12): 1473-1482. https://doi.org/10.1006/jasc.1998.0434

Duplaix-Rata A. 1997. — Les dépôts alimentaires carbonisés, *in* Petrequin P. (eds), *Les sites littoraux néolithiques de Clairvaux-les-Lacs et de Chalain (Jura). III Chalain station 3, 3200-2900 av. J.-C.* Vol. 2. Édition de la Maison des Sciences de l'Homme, Paris: 733-745.

Dupont C. 2019. — Exploitation des invertébrés marins du Néolithique au Campaniforme, *in* Pailler Y. & Nicolas C. (eds.), *Une maison sous les dunes : Beg ar Loued, île Molène, Finistère. Identité et adaptation des groupes humains en mer d'Iroise à la transition des III^e^ et II^e^ millénaires avant notre ère*. Sidestone Press, Leiden: 621-646.

Durand J. H. 1983. — *Les sols irrigables. Étude pédologique*. ACCT-PUF, Alger, 339 p.

Evershed R. P. 2008. — Experimental approaches to the interpretation of absorbed organic residues in archaeological ceramics. *World Archaeology* 40 (1): 26. https://doi.org/10.1080/00438240801889373

Evershed R. P., Heron C. & John Goad L. 1990. — Analysis of organic residues of archaeological origin by high-temperature gas chromatography and gas chromatography-mass spectrometry. *The Analyst* 115 (10): 1339-1342. https://doi.org/10.1039/an9901501339

Evershed R. P., Stott A. W., Raven A., Dudd S. N., Charters S. & Leyden A. 1995. — Formation of long-chain ketones in ancient-pottery vessels by pyrolysis of acyl lipids. *Tetrahedron Letters* 36 (48): 8875-8878. https://doi.org/10.1016/0040-4039(95)01844-8

Evershed R. P., Payne S., Sherratt A. G., Copley M. S., Coolidge J., Urem-Kotsou D., Kotsakis K., Özdoğan M., Özdoğan A. E., Nieuwenhuyse O. P., Akkermans P. M. M. G., Bailey D., Andeescu R.-R., Campbell S., Farid S., Hodder I., Yalman N., Özbaşaran M., Biçakci E., Garfinkel Y., Levy T. & Burton M. M. 2008. — Earliest date for milk use in the Near East and southeastern Europe linked to cattle herding. Nature 455 (25): 528-531. https://doi.org/10.1038/nature07180

Evershed R. P., Davey Smith G., Roffet-Salque M., Timpson A., Diekmann Y., Lyon M. S., Cramp L. J. E., Casanova E., Smyth J., Whelton H. L., Dunne J., Brychova V., Šoberl L., Gerbault P., Gillis R. E., Heyd V., Johnson E., Kendall I., Manning K., Marciniak A., Outram A. K., Vigne J.-D., Shennan S., Bevan A., Colledge S., Allason-Jones L., Amkreutz L., Anders A., Arbogast R.-M., Bălăşescu A., Bánffy E., Barclay A., Behrens A., Bogucki P., Carrancho Alonso Á., Carretero J. M., Cavanagh N., Classen E., Collado Giraldo H., Conrad M., Csengeri P., Czerniak L., Dębiec M., Denaire A., Domboróczki L., Donald C., Ebert J., Evans C., Francés-Negro M., Gronenborn D., Haack F., Halle M., Hamon C., Hülshoff R., Ilett M., Iriarte E., Jakucs J., Jeunesse C., Johnson M., Jones A. M., Karul N., Kiosak D., Kotova N., Krause R., Kretschmer S., Krüger M., Lefranc P., Lelong O., Lenneis E., Logvin A., Lüth F., Marton T., Marley J., Mortimer R., Oosterbeek L., Oross K., Pavúk J., Pechtl J., Pétrequin P., Pollard J., Pollard R., Powlesland D., Pyzel J., Raczky P., Richardson A., Rowe P., Rowland S., Rowlandson I., Saile T., Sebők K., Schier W., Schmalfuss G., Sharapova S., Sharp H., Sheridan A., Shevnina I., Sobkowiak-Tabaka I., Stadler P., Stäuble H., Stobbe A., Stojanovski D., Tasić N., Van Wijk I., Vostrovská I., Vuković J., Wolfram S., Zeeb-Lanz A. & Thomas M. G. 2022. — Dairying, diseases and the evolution of lactase persistence in Europe. *Nature* 608 (7922): 336-345. https://doi.org/10.1038/s41586-022-05010-7

Favrel Q. 2020. — Autour du gobelet maritime : les vases à cordon, gobelets linéaires et productions épicampaniformes du nord-ouest de la France. *Préhistoires méditerranéennes* 8. https://doi.org/10.4000/pm.2091

Gandois H., Pailler Y., Stephan P. & Nicolas C. 2013. — L'érosion marine et ses effets sur les vestiges archéologiques en mer d'Iroise : exemple de l'impact de la tempête de mars 2008 sur l'île de Kemenez et ses Ledenez (Le Conquet, Finistère, France), *in* Daire M.-Y., Dupont C., Baudry A., Billard C., arge J.-M., Lespez L., Normand E. & Scarre C. (eds), *Anciens peuplements littoraux et relations Homme/Milieu sur les côtes de l'Europe Atlanique/ Ancient maritime communities and the relationship between people and environment along the European Atlantic coasts. Proceedings of the HOMER 2011 Conference, Vannes (France), 28/09-1/10/2011.* Archeopress, British Archaeological Reports (coll. International Series; S2570), Oxford: 99-110.

Garnier N., Bernal-Casasola D., Driard C. & Pinto I. V. 2018. — Looking for ancient fish products through invisible biomolecular residues in the Roman production vats from the Atlantic Coast. *Journal of Maritime Archaeology* 13: 285-328. https://doi.org/10.1007/s11457-018-9219-x

Gaudin L. 2004. — *Les transformations spatio-temporelles de la végétation du nord-ouest de la France depuis la fin de la dernière glaciation. Reconstitutions paléo-paysagères*. Thesis, Université de Rennes 1, 757 p.

Giesen E. 2015. — Quality management for robust and reliable research. *International Journal of Metrology and Quality Engineering* 6 (4), 10 p. https://doi.org/10.1051/ijmqe/2015029

Hamon G. 2008. — Productions céramiques du néolithique armoricain. *Bulletin de la Société d'archéologie et d'histoire du Pays de Lorient* 37: 21-32.

HANOT P. & TRESSET A. 2019. — Les restes de grands vertébrés du Néolithique final aux âges du Bronze ancien et moyen, *in* PAILLER Y. & NICOLAS C. (eds), *Une maison sous les dunes : Beg ar Loued, île Molène, Finistère. Identité et adaptation des groupes humains en mer d'Iroise à la transition des IIIe et IIe millénaires avant notre ère*. Sidestone Press, Leiden: 461-480.

HERON C. P., CRAIG O. E., LUCQUIN A., STEELE V. J., THOMPSON A. & PILIČIAUSKAS G. 2015. — Cooking fish and drinking milk? Patterns in pottery use in the southeastern Baltic, 3300-2400 cal BC. *Journal of Archaeological Science* 63: 33-43. https://doi.org/10.1016/j.jas.2015.08.002

HORIUCHI A., MIYATA Y., KAMIJO N., CRAMP L. & EVERSHED R. P. 2015. — A dietary study of the Kamegaoka culture population during the final Jomon period, Japan, using stable isotope and lipid analyses of ceramic residues. *Radiocarbon* 57 (4): 721-736. https://doi.org/10.2458/azu_rc.57.18455

HUSSON O. 2013. — Redox potential (Eh) and pH as drivers of soil/plant/microorganism systems: a transdisciplinary overview pointing to integrative opportunities for agronomy. *Plant and Soil* 362 (1-2): 389-417. https://doi.org/10.1007/s11104-012-1429-7

JOLY C. & VISSET L. 2005. — Nouveaux éléments d'anthropisation sur le littoral vendéen dès la fin du Mésolithique. *Comptes Rendus Palevol* 4 (3): 285-293. https://doi.org/10.1016/j.crpv.2004.12.006

JOLY D., BROSSARD T., CARDOT H., CAVAILHES J., HILAL M. & WAVRESKY P. 2010. — Les types de climats en France, une construction spatiale – Types of climates on continental France, a spatial construction. *Cybergéo* (501): 1-23

LEMERCIER O. 2002. — Le Campaniforme dans le sud-est de la France. De l'Archéologie à l'Histoire du troisième millénaire avant notre ère. *Préhistoires méditerranéennes* 10-11: 223-227. https://doi.org/10.4000/pm.279

LIU L., WANG J. & LEVIN M. J. 2017. — Usewear and residue analyses of experimental harvesting stone tools for archaeological research. *Journal of Archaeological Science: Reports* 14: 439-453. https://doi.org/10.1016/j.jasrep.2017.06.018

LUCQUIN A., COLONESE A. C., FARRELL T. F. G. & CRAIG O. E. 2016a. — Utilising phytanic acid diastereomers for the characterisation of archaeological lipid residues in pottery samples. *Tetrahedron Letters* 57 (6): 703-707. https://doi.org/10.1016/j.tetlet.2016.01.011

LUCQUIN A., GIBBS K., UCHIYAMA J., SAUL H., AJIMOTO M., ELEY Y., RADINI A., HERON C. P., SHODA S., NISHIDA Y., LUNDY J., JORDAN P., ISAKSSON S. & CRAIG O. E. 2016b. — Ancient lipids document continuity in the use of early hunter–gatherer pottery through 9,000 years of Japanese prehistory. *Proceedings of the National Academy of Sciences* 113 (15): 3991-3996. https://doi.org/10.1073/pnas.1522908113

MAGNY M. 2004. — Holocene climate variability as reflected by mid-European lake-level fluctuations and its probable impact on prehistoric human settlements. *Quaternary International* 113 (1): 65-79. https://doi.org/10.1016/S1040-6182(03)00080-6

MARTÍN-CHIVELET J., MUÑOZ-GARCIA M. B., EDWARDS R. L., TURRERO M. J. & ORTEGA A. I. 2011. — Land surface temperature changes in Northern Iberia since 4000yrBP, based on δ13C of speleothems. Global and Planetary Change 77 (1-2): 1-12. https://doi.org/10.1016/j.gloplacha.2011.02.002

MATHIEU C. & PIELTAIN F. 2003. — *Analyse chimique des sols méthodes choisies*. Éditions Lavoisier (coll. TEC&DOC), Paris, 388 p.

MIRABAUD S., ROLANDO C. & REGERT M. 2007. — Molecular criteria for discriminating adipose fat and milk from different species by NanoESI MS and MS/MS of their triacylglycerols: application to archaeological remains. *Analytical Chemistry* 79 (16): 6182-6192. https://doi.org/10.1021/ac070594p

MOUGNE C. 2019. — Exploitation et utilisation des invertébrés marins durant l'âge du Bronze ancien, *in* PAILLER Y. & NICOLAS C. (eds), *Une maison sous les dunes : Beg ar Loued, île Molène, Finistère. Identité et adaptation des groupes humains en mer d'Iroise à la transition des IIIe et IIe millénaires avant notre ère*. Sidestone Press, Leiden: 647-654.

MUKHERJEE A. J., GIBSON A. M. & EVERSHED R. P. 2008. — Trends in pig product processing at British Neolithic Grooved Ware sites traced through organic residues in potsherds. *Journal of Archaeological Science* 35 (7): 2059-2073. https://doi.org/10.1016/j.jas.2008.01.010

NICOLAS C., FAVREL Q., ROUSSEAU L., ARD V., BLANCHET S., DONNART K., FROMONT N., MANCEAU L., MARCIGNY C., MARTICORENA P., NICOLAS T., PAILLER Y. & RIPOCHE J. 2019. — The introduction of the Bell Beaker culture in Atlantic France: an overview of settlements, *in* GIBSON A. (eds), *Bell Beaker Settlements in Europe. The Bell Beaker phenomenon from a domestic perspective*. Oxbow Books, Oxford: 329-352.

OUTRAM A. K., STEAR N. A., BENDREY R., OLSEN S., KASPAROV A., ZAIBERT V., THORPE N. & EVERSHED R. P. 2009. — The earliest horse harnessing and milking. *Science* 323 (5919): 1332-1335. https://doi.org/10.1126/science.1168594

PÄÄKKÖNEN M., EVERSHED R. P. & ASPLUND H. 2020. — Compound-specific stable carbon isotope values of fatty acids in modern aquatic and terrestrial animals from the Baltic Sea and Finland as an aid to interpretations of the origins of organic residues preserved in archaeological pottery. *Journal of Nordic Archaeological Science (JONAS)* 19: 3-19. https://www.archaeology.su.se/english/publications/publication-series/jonas/jonas-19

PAILLER Y. & NICOLAS C. 2019a. — *Une maison sous les dunes : Beg ar Loued, île Molène, Finistère. Identité et adaptation des groupes humains en mer d'Iroise à la transition des IIIe et IIe millénaires avant notre ère*. Sidestone Press, Leiden, 621 p.

PAILLER Y. & NICOLAS C. 2019b. — Insularité et singularité. Bilan et éléments de synthèse, *in* PAILLER Y. & NICOLAS C. (eds), *Une maison sous les dunes : Beg ar Loued, île Molène, Finistère. Identité et adaptation des groupes humains en mer d'Iroise à la transition des IIIe et IIe millénaires avant notre ère*. Sidestone Press, Leiden: 603-621.

PAILLER Y., SPARFEL Y., TRESSET A., BALASSE M., DUPONT C., GIOVANNACCI S., HALLEGOUËT B., JOSSELIN J. & MARCHAND G. 2004. — Fouille d'un dépotoir du Néolithique final à Beg ar Loued (île Molène, Finistère) : premiers résultats. *Bulletin de la Société Préhistorique Française* 101 (4): 881-886 https://doi.org/10.3406/bspf.2004.13076

PAILLER Y., STEPHAN P., GANDOIS H., NICOLAS C., SPARFEL Y., TRESSET A., DONNART K., FICHAUT B., SUANEZ S., DUPONT C., CLEZIO L. LE, MARCOUX N., PINEAU A., SALANOVA L., SELLAMI F., DEBUE K., JOSSELIN J. & DIETSCH-SELLAMI M.-F. 2011. — Évolution des paysages et occupation humaine en mer d'Iroise (Finistère, Bretagne) du Néolithique à l'Âge du Bronze. *Norois* 220: 39-68. https://doi.org/10.4000/norois.3662

PAILLER Y., STEPHAN P., GANDOIS H., NICOLAS C., SPARFEL Y., TRESSET A., DONNART K., DRÉANO Y., FICHAUT B., SUANEZ S., DUPONT C., AUDOUARD L., MARCOUX N., MOUGNE C., SALANOVA L., SELLAMI F. & DIETSCH-SELLAMI M. F. 2014. — *Landscape Evolution and Human Settlement in the Iroise Sea (Brittany, France) during the Neolithic and Bronze AgeIn*. Cambridge University Press, Cambridge: 105-139. https://doi.org/10.1017/ppr.2014.9

PAUL D., SKRZYPEK G. & FORIZS I. 2007. — Normalization of measured stable isotopic compositions to isotope reference scales – a review. *Rapid Communications in Mass Spectrometry* 21 (18): 3006-3014. https://doi.org/10.1002/rcm.3185

RAVEN A., VAN BERGEN P. F., STOTT A. W., HARDEN S. & EVERSHED R. P. 1997. — Formation of long-chain ketones in archaeological pottery vessels by pyrolysis of acyl lipids. *Journal of Analytical and Applied Pyrolysis* 40-41: 267-285. https://doi.org/10.1016/S0165-2370(97)00036-3

REGERT M. 2011. — Analytical strategies for discriminating archaeological fatty substances from animal origin. *Mass Spectrometry Reviews* 30 (2): 177-220. https://doi.org/10.1002/mas.20271

REGERT M. & MAZUY A. 2019. — Des graisses sous-cutanées de ruminants et des produits laitiers dans les céramiques, *in* PAILLER Y. & NICOLAS C. (eds), *Une maison sous les dunes : Beg ar Loued, Île de Molène, Finistère. Identité et adaptation des groupes humains entre les IIIe et IIe millénaires avant notre ère*. Sidestone Press, Leiden: 569-576.

REGERT M., COLINART S., DEGRAND L. & DECAVALLAS O. 2001. — Chemical alteration and use of beeswax through time: accelerated ageing test and analysis of archaeological samples from various environemental contexts. *Archaeometry* 43 (4): 549-569 https://doi.org/10.1111/1475-4754.00036

RICHARDS M. P., SCHULTING R. J. & HEDGES R. E. M. 2003. — Sharp shift in diet at onset of Neolithic. *Nature* 425: 366. https://doi.org/10.1038/425366a

RIPOCHE J. 2017. — La transition du Néolithique à l'âge du Bronze dans le Nord-Ouest de la façade atlantique : entre révolutions techniques et transformations sociales, *in* FILET C., HÖLTKEMEIER S., PERRIOT C. & ROLLAND J. (dirs), *Révolutions : L'archéologie face aux renouvellements des sociétés*. Éditions de la Sorbonne, Paris. https://doi.org/10.4000/books.psorbonne.6748

RIPOCHE J. 2022. — *Traditions céramiques et dynamiques culturelles à la transition des IIIème et IIème millénaires en France Atlantique : genèse de l'âge du bronze et interactions en Europe Atlantique (2250-1600 av. JC)*. Thesis, Université de Paris 1, 800 p.

ROBSON H. K., SKIPITYTĖ R., PILIČIAUSKIENĖ G., LUCQUIN A., HERON C. P., CRAIG O. E. & PILIČIAUSKAS G. 2019. — Diet, cuisine and consumption practices of the first farmers in the south-eastern Baltic. *Archaeological and Anthropological Sciences* 11: 4011-4024. https://doi.org/10.1007/s12520-019-00804-9

ROFFET-SALQUE M., DUNNE J., ALTOFT D. T., CASANOVA E., CRAMP L. J. E., SMYTH J., WHELTON H. L. & EVERSHED R. P. 2016. — From the inside out: upscaling organic residue analyses of archaeological ceramics. *Journal of Archaeological Science: Reports* 30: 1-13. https://doi.org/10.1016/j.jasrep.2016.04.005

ROFFET-SALQUE M., REGERT M., EVERSHED R. P., OUTRAM A. K., CRAMP L. J. E., DECAVALLAS O., DUNNE J., GERBAULT P., MILETO S., MIRABAUD S., PÄÄKKÖNEN M., SMYTH J., ŠOBERL L., WHELTON H. L., ALDAY-RUIZ A., ASPLUND H., BARTKOWIAK M., BAYER-NIEMEIER E., BELHOUCHET L., BERNARDINI F., BUDJA M., COONEY G., CUBAS M., DANAHER E. M., DINIZ M., DOMBORÓCZKI L., FABBRI C., GONZÁLEZ-URQUIJO J. E., GUILAINE J., HACHI S., HARTWELL B. N., HOFMANN D., HOHLE I., IBÁÑEZ J. J., KARUL N., KHERBOUCHE F., KIELY J., KOTSAKIS K., LUETH F., MALLORY J. P., MANEN C., MARCINIAK A., MAURICE-CHABARD B., MC GONIGLE M. A., MULAZZANI S., ÖZDOĞAN M., PERIĆ O. S., PERIĆ S. R., PETRASCH J., PÉTREQUIN A.-M., PÉTREQUIN P., POENSGEN U., JOSHUA POLLARD C., POPLIN F., RADI G., STADLER P., STÄUBLE H., TASIĆ N., UREM-KOTSOU D., VUKOVIĆ J. B., WALSH F., WHITTLE A., WOLFRAM S., ZAPATA-PEÑA L. & ZOUGHLAMI J. 2015. — Widespread exploitation of the honeybee by early Neolithic farmers. *Nature* 527 (7577): 226-230. https://doi.org/10.1038/nature15757

SALANOVA L. 2000. — *La question du campaniforme en France et dans les îles anglo-normandes : productions, chronologie et rôles d'un standard céramique*. CTHS et Société Préhistorique Française, Paris, 391 p.

SALANOVA L. 2004. — Le rôle de la façade atlantique dans la genèse du Campaniforme en Europe. *Bulletin de la Société préhistorique française* 101 (2): 223-226. https://doi.org/10.3406/bspf.2004.12989

SALANOVA L., BRUNET P., COTTIAUX R., HAMON T., LANGRY-FRANÇOIS F., MARTINEAU R., POLLONI A., RENARD C. & SOHN M. 2011. — Du Néolithique récent à l'âge du Bronze dans le Centre Nord de la France : les étapes de l'évolution chrono-culturelle. *Revue archéologique de Picardie* 28: 77-101. https://doi.org/10.3406/pica.2011.3323

SALANOVA L. 2019. — Les productions céramiques des IIIe et IIe millénaires av. J.-C., *in* PAILLER Y. & NICOLAS C. (eds), *Une maison sous les dunes : Beg ar Loued, île Molène, Finistère. Identité et adaptation des groupes humains en mer d'Iroise à la transition des IIIe et IIe millénaires avant notre ère*. Sidestone Press, Leiden, 273-290.

SALQUE M., BOGUCKI P. I., PYZEL J., SOBKOWIAK-TABAKA I., GRYGIEL R., SZMYT M. & EVERSHED R. P. 2013. — Earliest evidence for cheese making in the sixth millennium BC in northern Europe. *Nature* 493 (7433): 522-525. https://doi.org/10.1038/nature11698

SCHULTING R. J. 2005. — Comme la mer qui se retire : les changements dans l'exploitation des ressources marines du Mésolithique au Néolithique en Bretagne, *in* MARCHAND G & TRESSET A (eds), *Unité et diversité des processus de néolithisation sur la façade atlantique de l'Europe (6e-4e millénaires avant J.-C.)*. Société préhistorique française, Joué-lès-Tours: 163-171

ŠOBERL L. 2011. — *Pots for the Afterlife: organic residue analysis of British early Bronze age pottery from funerary contexts*. Thesis, University of Bristol, 280 p.

STÉPHAN P., PAILLER Y., TRESSET A. & GANDOIS H. 2013. — Changements paléogéographiques de l'archipel de Molène (Finistère, Bretagne, France) : implications sur les peuplements humains du Néolithique à l'âge du Bronze, *in* DAIRE M.-Y., DUPONT C., BAUDRY A., BILLARD C., LARGE J.-M., LESPEZ L., NORMAND E. & SCARRE C. (eds), *Ancient maritime communities and the relationship between people and environment along the European Atlantic coasts, Proceedings of the HOMER 2011 Conference, Vannes, 28 sept.-1er oct. 2011*. Archaeopress, British Archaeological Reports (coll. International Series; 2570), Oxford: 647-660.

STÉPHAN P., GANDOIS H., EHRHOLD A., LE DANTEC N., FRANZETTI M., PAILLER Y., BALTZER A. & JOUET G. 2019a. — De l'usage de certaines pêcheries à l'âge du Bronze ancien dans l'archipel de Molène, *in* PAILLER Y. & NICOLAS C. (eds), *Une maison sous les dunes : Beg ar Loued, île Molène, Finistère. Identité et adaptation des groupes humains en mer d'Iroise à la transition des IIIe et IIe millénaires avant notre ère*. Sidestone Press, Leiden: 88-100.

STÉPHAN P., VERDIN F., ARNAUD FASSETTA G., BERTRAND F., EYNAUD F., GARCIA ARTOLA A., BOSQ M., CULIOLI C., SUANEZ S., COUTELIER C., BERTRAN P. & COSTA S. 2019b. — Holocene coastal changes along the Gironde estuary (SW France): new insights from the North Médoc peninsula beach/dune system. *Quaternaire* 30 (1): 47-75. https://doi.org/10.4000/quaternaire.11172

STERN B., HERON C. P., SERPICO M. & BOURRIAU J. 2000. — A comparison of methods for establishing fatty acid concentration gradients across potsherds: a case study using Late Bronze Age Canaanite amphorae. *Archaeometry* 42 (2): 399-414. https://doi.org/10.1111/j.1475-4754.2000.tb00890.x

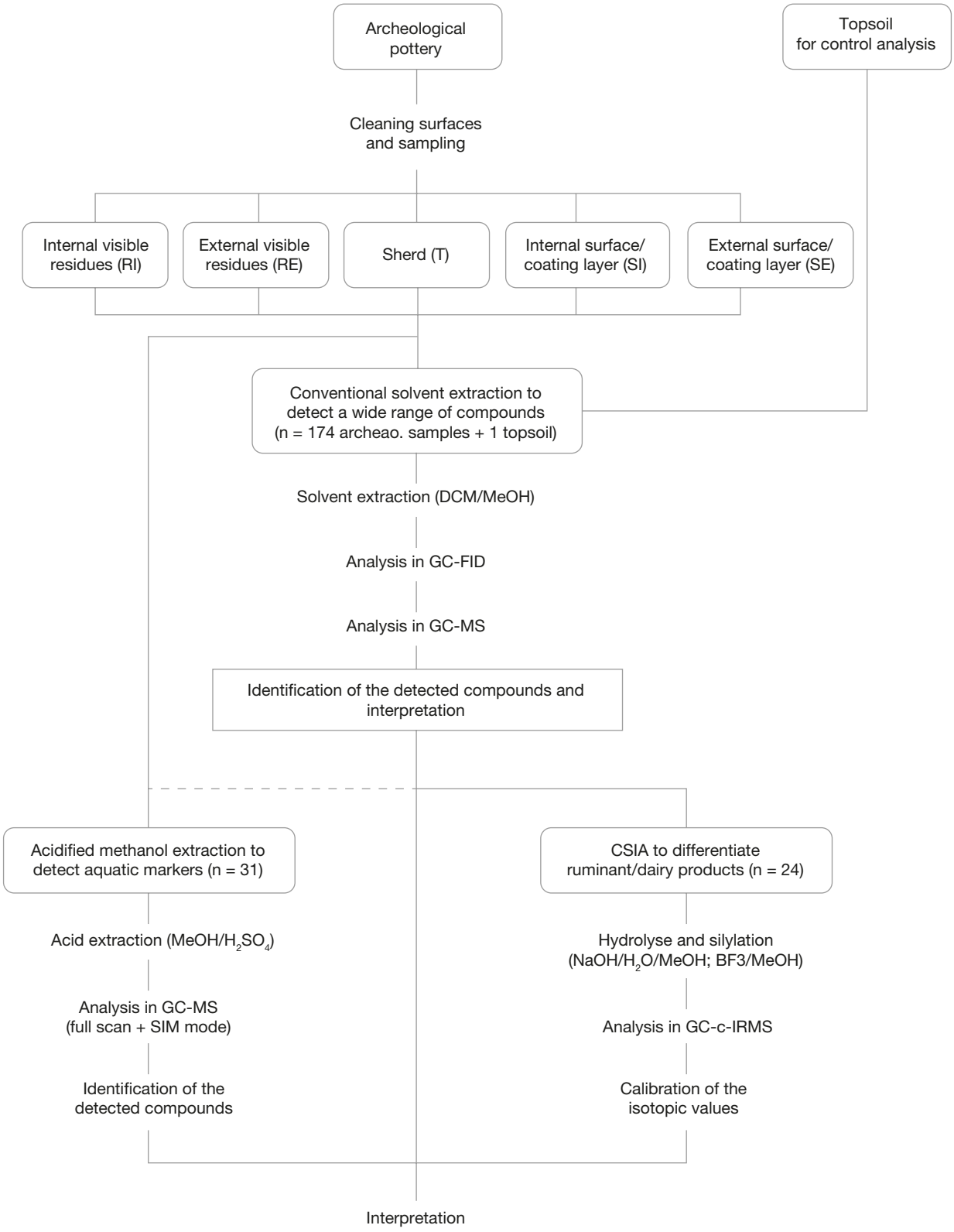
TACHÉ K. & CRAIG O. E. 2015. — Cooperative harvesting of aquatic resources and the beginning of pottery production in north-eastern North America. *Antiquity* 89 (343): 177-190. https://doi.org/10.15184/aqy.2014.36

TARIFA-MATEO N., REGERT M., CRAIG O. E., ROSELL-MELÉ A., CLOP X. & SAÑA M. 2021. — Pottery use in the mining site of variscite Mines de Gavà (Barcelona, Spain) during the 4th millennium BC based on organic residue analysis. *Journal of Archaeological Science: Reports* 38: 103080. https://doi.org/10.1016/j.jasrep.2021.103080

Submitted on 12 May 2023;
accepted on 11 September 2023;
published on 15 January 2024.

APPENDICES

Appendix 1. — Scheme of the analytical strategy carried out on the sample-set of pottery from Beg ar Loued. Abbreviations: **BF3**, boron trifluoride; **CSIA**, compound-specific isotopic analysis; **DCM**, dichloromethane; **GC-c-IRMS**, gas chromatography-combustion-isotope ratio mass spectrometry; **GC-FID**, gas chromatography-flame ionization detection; **GC-MS**, gas chromatography-mass spectrometry; **H_2SO_4**, sulphuric acid; **MeOH** methanol; **NaOH**, sodium hydroxide.



Appendix 2. — Description of lipids extractions, methods, and control analysis.

LIPID EXTRACTION

Solvent extraction (DCM/MeOH)

A total of 174 archaeological samples including sherds, adhering visible charred residues, surface/coating layers and a topsoil sample were extracted with this method in the first place.

Sherd fragments (*c.* 2 g) were cleaned with a modeling drill and ground to a powder in a solvent-cleaned mortar and pestle, while charred internal or external surface visible residues (between 5-50 mg) adhering to pottery were scraped with a solvent-cleaned scalpel and ground in a solvent-cleaned mortar and pestle. External/internal surface/coating layers were gently cleaned with a modeling drill to remove the top-most contaminating surface, and then drilled to obtain a powder. The control topsoil sample (5 g) was sieved (2 mm grid) and ground.

Then, lipids were extracted via conventional solvent extraction (Evershed *et al.* 1990). An internal standard was added in the archaeological samples (20 µg of *n*-C_{34}, 1 mg.mL^{-1} in *n*-hexane) and control topsoil sample (50 µg of *n*-C_{34}, 1 mg.mL^{-1} in *n*-hexane). A solution of dichloromethane/methanol (DCM/MeOH) 2:1 v/v was added to the samples (10 mL for crushed sherds, charred visible residues, and surface/coating layers, and 20 mL for the control topsoil sample) and sonicated (2 × 15 min). After centrifugation (12 min; 3000 rpm), the supernatant was evaporated to dryness and dissolved in 500 µl of DCM/MeOH to obtain the total lipid extract (TLE). Aliquots of the TLE were trimethylsilylated (N,O-is(trimethylsilyl)trifluoroacetamide, 70 µL, 70°C, 60 min), and submitted to analysis by GC and GC-MS. A method blank was extracted alongside each batch of samples to identify any contaminant introduced during the extraction.

A sample-set (n = 24) was selected for compound-specific isotopic analysis. Samples with poor preservation only with fatty acids, or those with traces of triacylglycerols (TAGs) which could be indicative of ruminant carcass/dairy products were chosen.

Aliquots of the total lipid extract by solvent (DCM/MeOH) were hydrolysed (0.5 M NaOH/ H_2O/MeOH (9:1 v/v, 4 mL); 70°C, 1 h). The neutral fraction was removed after acidification to pH 3 using HCl (200 µL; 6M) and deionised water (1 mL), and free fatty acids were extracted (3 × 3 mL *n*-hexane). Fatty acids were methylated using 500 µL BF3/MeOH (14% w/v) and toluene (1 mL, 70°C, 1 h). The extracts were cooled, diluted with deionised water (2 mL), and the toluene phase was transferred to an autosampler vial. The extracts were evaporated to dryness and dissolved in 50-100 µL of *n*-hexane before being submitted to GC-C-IRMS analysis at the Laboratoire d'Océanographie de Villefranche-sur-Mer (LOV – CNRS UMR 7093).

Acid extraction (MeOH/H_2SO_4)

Archaeological samples with good preservation of lipids, or with the presence of polyunsaturated fats (PUFAs), isoprenoid fatty acids (IFAs) and/or ketones were selected for further extraction to detect aquatic resources and improve the characterisation of ruminant fats.

The acid extraction protocol was adapted from Craig *et al.*, 2007, 2013; Correa-Ascencio & Evershed 2014. Samples were ground (*c.* 0.8 g) and an internal standard was added (triacontane, 1 mg.mL^{-1}, 10 µL), after which methanol was added (4 mL). The samples were sonicated (15 min), then after cooling, concentrated sulphuric acid was added (*c.* 800 µL). The tubes were heated for 4 hours at 70°C. Once cooled, they were centrifuged (3000 rpm; 5 min) to facilitate separation of the solid fraction. The liquid fraction was recovered. The acid phase was then extracted by adding 2 mL of hexane and rinsing the solid fraction with 2 mL of hexane, again after centrifugation (3000 rpm; 15 min). The acid phase (6 mL) was evaporated under a stream of nitrogen (40°C), and re-diluted in 500 µL of cyclohexane. Finally, 1 µL was used for each analysis, while the rest was kept in the freezer (–40°C).

ANALYSIS VIA GC-FID AND GC-MS

Archaeological samples

GC-FID analyses (gas chromatography-flame ionization detection) were performed on an Agilent Technologies 7890A device. A volume of 1 µL of sample was introduced via an on-column injector into a 15 m × 0.32 mm i.d. fused silica capillary (DB5-MS, 0.1 µL film thickness, Agilent J&W), with helium used as carrier gas. The GC temperature program was as follows: increased from 50°C to 100°C at 15°C.min^{-1}, then from 100°C to 375°C at 10°C.min^{-1}.

For GC-MS analysis (gas chromatography-mass spectrometry), the instrument used was a Shimadzu GC 2010 PLUS chromatograph coupled to a Shimadzu QP 2010 ULTRA mass spectrometer, fitted with a high temperature non-polar column (DB5-HT, 15 m × 0.322 mm i.d., 0.1 µm film thickness, Agilent J&W). The injection was performed using a splitless injector. The temperature programme consisted of a 1 min isothermal hold at 50°C followed by an increase to 150°C at 20°C.min^{-1}, then to 250°C at 10°C.min^{-1} and to 350°C.min^{-1} and a final isothermal hold for 10 min. The GC-MS interface was maintained at a temperature of 300°C and the mass spectrometer was run in electron ionization mode (EI, 70 eV). Mass spectra were acquired over the range *m/z* 50-950.

The instrument was also used on SIM mode to track specific ions for the detection of aquatic resources and ruminant fat, such as isoprenoid fatty acids (IFAs) and ω-(o-alkylphenyl) alkanoic acids (APAAs).

A first approach used the solvent extracts by adapting the SIM scan for trimelthylsilylated samples (e.g. *m/z* 105, 91, 333, 361, 389; 346, 376); (Driard *et al.* 2017)

A second approach used the methanolic extracts by adapting the SIM scan for screening characteristic ions (e.g. *m/z* 74, 87, 213, 270 for TMTD; *m/z* 101, 312, 171, 326 for phytanic acid (PHY) and pristanic acid (PRI); *m/z* 105, 262, 290, 318, 346 for $APAAs_{C16\text{-}C22}$); (Cramp & Evershed 2014). For these analyses, the same couplage of the Shimadzu GC 2010 PLUS and the Shimadzu QP 2010 ULTRA mass spectrometer was

used and fitted with DB-23 column (60 m × 0.250 mm i.d., 0.25 µm film thickness, Agilent J&W). The injection was performed using a splitless injector (250°C). The temperature programme consisted of a 2 min isothermal hold at 50°C followed by an increase to 100°C at 10°C.min^{-1}, then to 140°C at 4°C.min^{-1}, then to 160°C at 0.5°C.min^{-1}, then to 250°C at 10°C.min^{-1}, and a final isothermal hold for 10 min. The GC-MS interface was maintained at a temperature of 250°C, the mass spectrometer run in electron ionization mode (EI, 70 eV) and the gas flow was set for 2.3 mL.min^{-1}. Mass spectra were acquired over the range *m/z* 50-950 and in SIM mode for selecting characteristic ions.

Control topsoil sample

GC-FID and GC-MS analyses were performed on an Agilent Technologies CN2039A035 coupling of a 8890 FID detector (G3540A) and a 5977B spectrometer via a G3181 splitter and two post columns (DB-5HT, adjustable length × 0.180 µm i.d., 0.1 µm film thickness, 60-400°C, Agilent J&W): 1µL of analyte was split for analysis into two aliquots. The main column was a non-polar column (DB-5HT, 15 m × 0.320 µm i.d., 0.1 µm film thickness, 60-400°C, Agilent J&W). The configuration was used with splitless injection and full scan detection (50-900). The FID was heated to 370°C. The MSD transfer line was maintained at 370°C, the ion source was heated to 280°C and the quad to 150°C. The temperature program consisted of a 1 min isothermal hold at 50°C, followed by an increase to 370°C at 15°C.min^{-1}, and an isothermal hold for 12 min. The gas flow rate was set at 4 mL.m^{-1} for 15 minutes, then reduced to 2.9 mL.m^{-1} at a rate of 0.15 mL.m^{-1}.

ANALYSIS VIA GC-C-IRMS

Stable carbon isotope values of methyl palmitate ($C_{16:0}$) and methyl stearate ($C_{18:0}$), derived from precursor fatty acids, were measured by GC-C-IRMS (GasChromatography Combustion Isotope Ratio Mass Spectrometry) at the Laboratoire d'Océanographie de Villefranche-sur-Mer (LOV – CNRS UMR 7093). The instrumentation consisted of an Agilent 7890 series GC (Agilent Technologies, Santa Clara, CA, United States) coupled to a Agilent 5995 single quadrupole mass spectrometer (Agilent Technologies, Santa Clara, CA, United States – 15% of the output flow) and an Isoprime 100 (Isoprime, Cheadle, United Kingdom – 85% of the output flow). Ion intensities (44, 45 and 46 *m/z*) of eluted products were recorded and the corresponding $^{13}C/^{12}C$ ratios were computed.

Results were calibrated against a standard reference gas (CO_2) of known isotopic composition. Most samples were run in duplicate (19/24). A certified reference material (Indiana F8-3) was injected at the beginning and end of each run, a certified fatty acid methyl ester standard of known $\delta^{13}C$ (FAME $C_{10:0}$) was added to each sample, and in-house standards were used and run in sequence with the samples to check instrument reproductibility and reliability (0.5‰; Giesen 2015). The $\delta^{13}C$ values were derived according to the following expression and are relative to the international standard VPDB: $\delta^{13}C‰ = ((R_{sample} - R_{standard})/R_{standard}) \times 1000$, where $R = {}^{13}C/{}^{12}C$ (Paul *et al.* 2007). The resulting data were corrected to account for methylation through EA-IRMS measurements of the isotopic values of the fatty acids used in the laboratory standards.

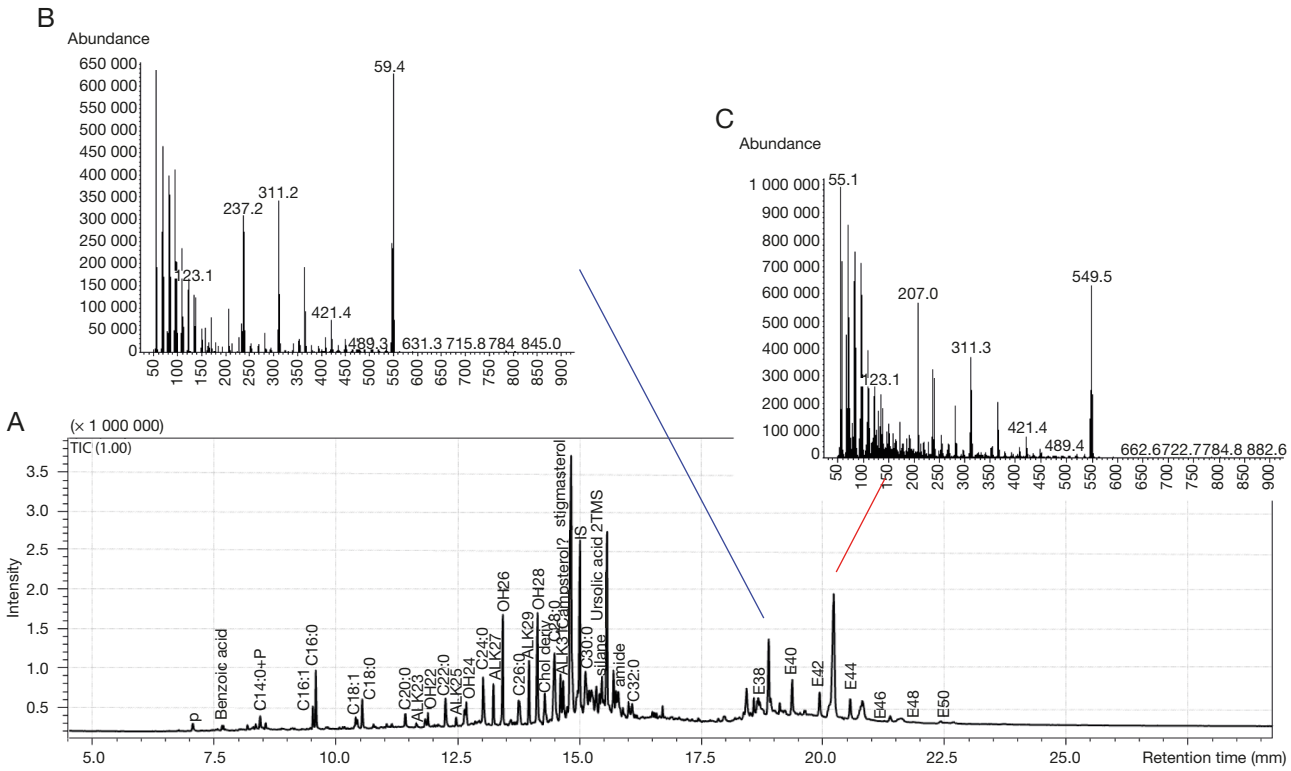
The obtained results were compared with δ13C values of a range of authentic modern European adipose, dairy and marine fats, published elsewhere (Craig *et al.* 2005b, 2007; Debono Spiteri 2012).

CONDUCTOMETRIC ANALYSIS FOR CONTROL TOPSOIL SAMPLE

Measurements on the control topsoil sample were carried out according to the pHwater method on ions dissolved in demineralised water (Baize 2000). Sediments were crushed and dry-sieved with a 2 mm grid and 10 g of powder (<2 mm) was collected for analysis. Deionised water (v/v; 1:2.5) was added to the sediment and mixed with a magnetic stirrer for 2 mm before being left to settle for 1.5 hours. It was mixed again before carrying out the measurements of the hydrogen (pH) and oxidation-reduction potentials (Eh). For this purpose, the probe of the pH-meter (Eutech PC 450) was calibrated beforehand with buffer solutions adapted to pH 4.01 and 7 at 25°C. Interpretation for the pH and Eh value were based on published work and public domain knowledge (DeLaune *et al.* 1981; Joly *et al.* 2010; Husson 2013).

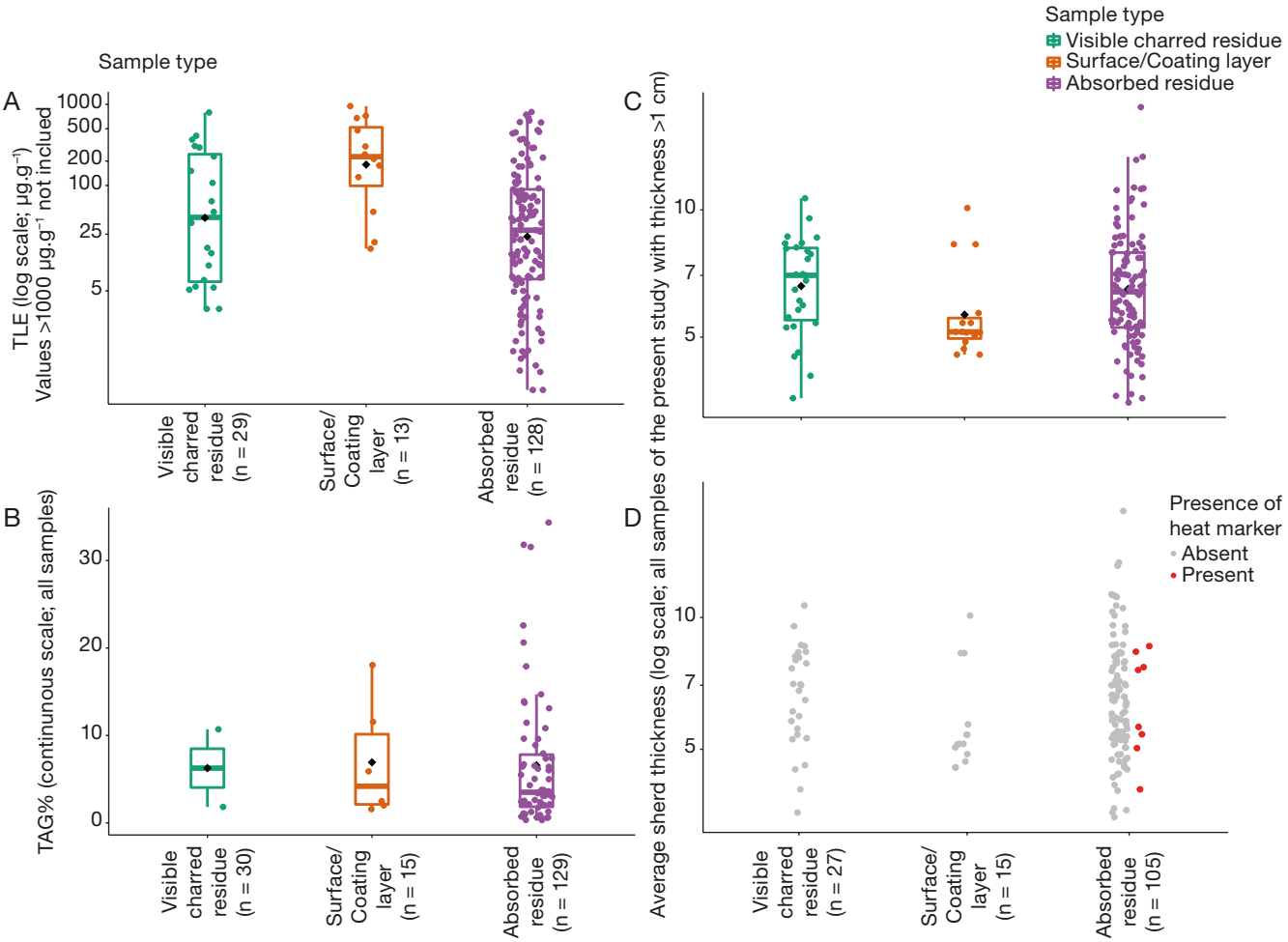
To perform electrical conductivity (EC) and total dissolved solids (TDS) measurements (Mathieu & Pieltain 2003), the aqueous extracts used for pH and Eh measurements were diluted again to the commonly used 1994 NF ISO 11265 standard (v/v; 1:5). The samples were mixed again by a magnetic stirrer for 2 mm, before being left to decant for 30 min, centrifuged (2000 rpm; 10 min) and filtered. The conductivity-meter (Eutech PC 450) equipped with a two-electrode probe was calibrated with an EC buffer solution set at 12 880 µS.cm^{-1} at 25°C. Measurements were then made by immersing the two rings in solution. The rings are sensitive to the environmental temperature, so a correction of the measured values was applied according to the formula, where the cell constant is involved ($K_{cell.}$): $EC_{corr.} = (EC_{mesured} \times CoorT)/K_{cell.}$, where $[K_{cell.} = EC_{buffer\ cal.}/EC_{buffer\ before\ cal.}]$. Interpretation for the EC and TDS values are based on published work and public domain knowledge (Durand 1983; Anonymous 2015, 2018).

Appendix 3. — Chromatogram of lipid extract of control sediment and mass spectrum of unknown compounds (Beg ar Loued): **A**, chromatogram of lipid extract of control sediment; **B**, **C**, mass spectrum of unknown compounds.

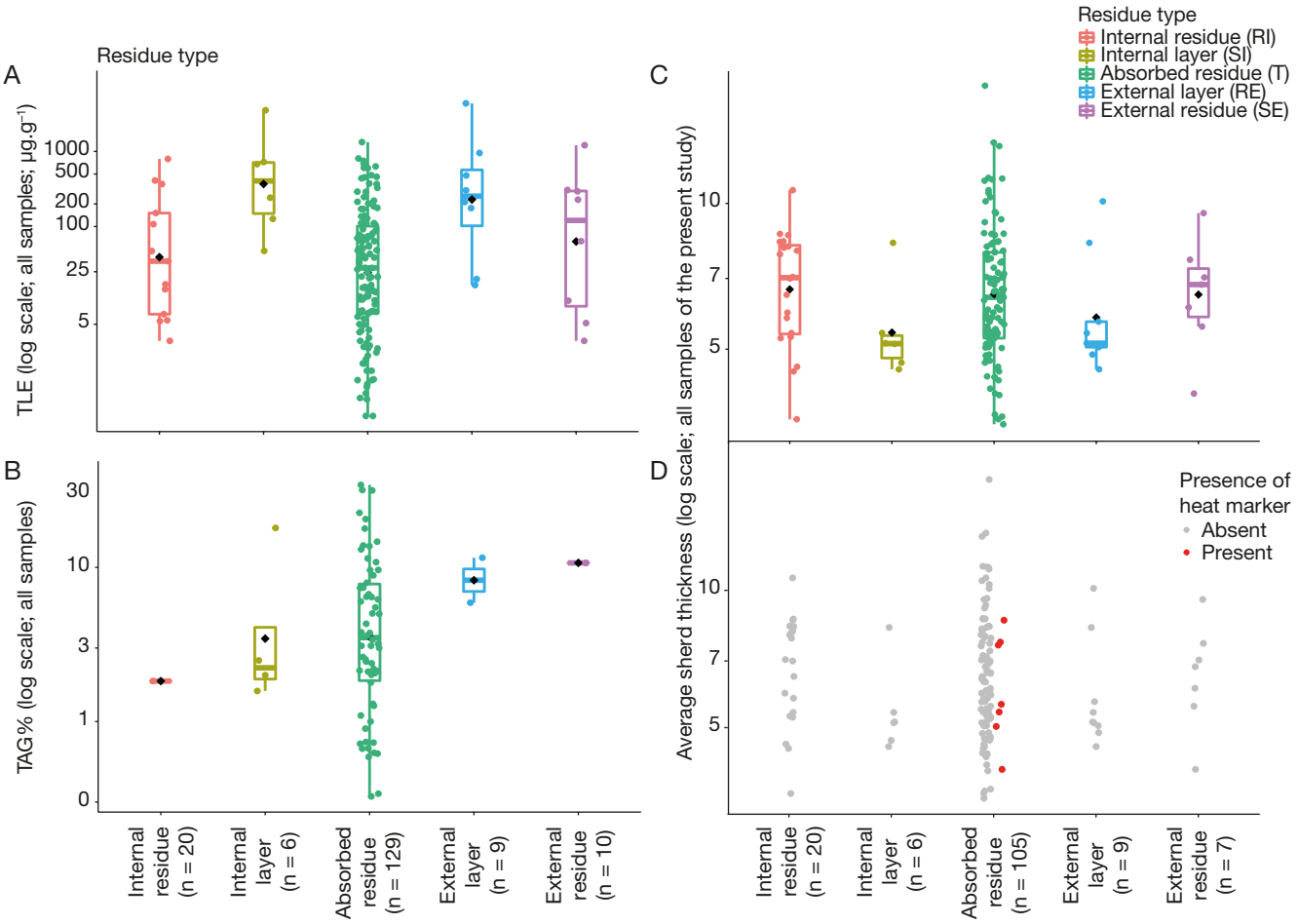


Appendix 4. — Tables of all molecular and isotopic data: **A**, acronyms used; **B**, general archaeological data; **C**, general analysis data; **D**, table comparing the total lipid extract (TLE) of samples that have external and internal charred residues, external and internal coating/surfaces and absorbed residues; **E**, table with esters data: https://doi.org/10.5852/cr-palevol2024v23a1_s1

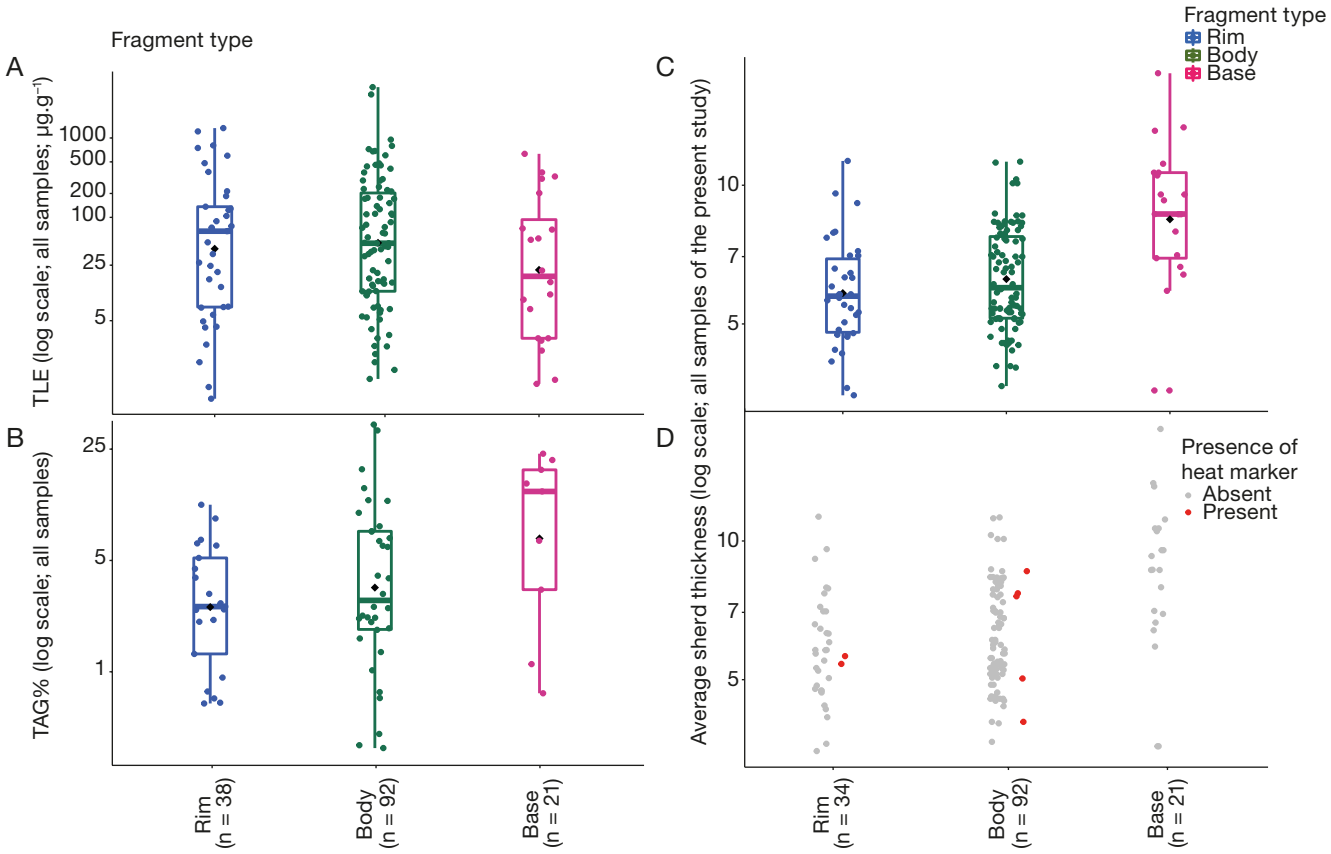
Appendix 5. — Boxplots comparing different sample residue types with: **A**, total lipid extract (TLE; samples with TLE <1000µg.g^{-1}); **B**, triacylglycerol preservation (TAG%); **C**, average pot wall thickness; **D**, identification of molecular heating markers. **C**, **D**, not including samples from 2019 study. **Black diamonds** indicate mean value. R Markdown file for plots available in Appendix 14. Abbreviations: **TAG**, triacylglycerol(s); **TLE**, total lipid extract.



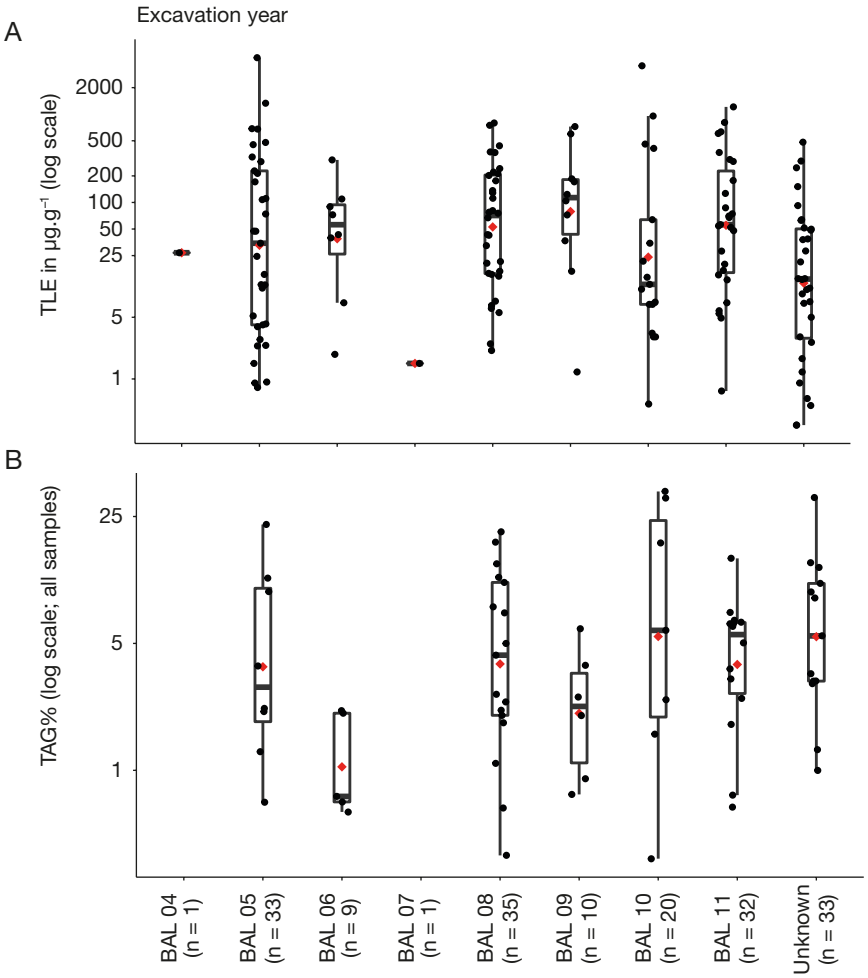
Appendix 6. — Boxplots comparing different sample residue types with: **A**, total lipid extract (TLE); **B**, triacylglycerol preservation (TAG%); **C**, average pot wall thickness; **D**, identification of molecular heating markers. **C**, **D**, do not include samples from 2019 study. **Black diamonds** indicate mean value. R Markdown file for plots available in Appendix 14. Abbreviations: **TAG**, triacylglycerol(s); **TLE**, total lipid extract.



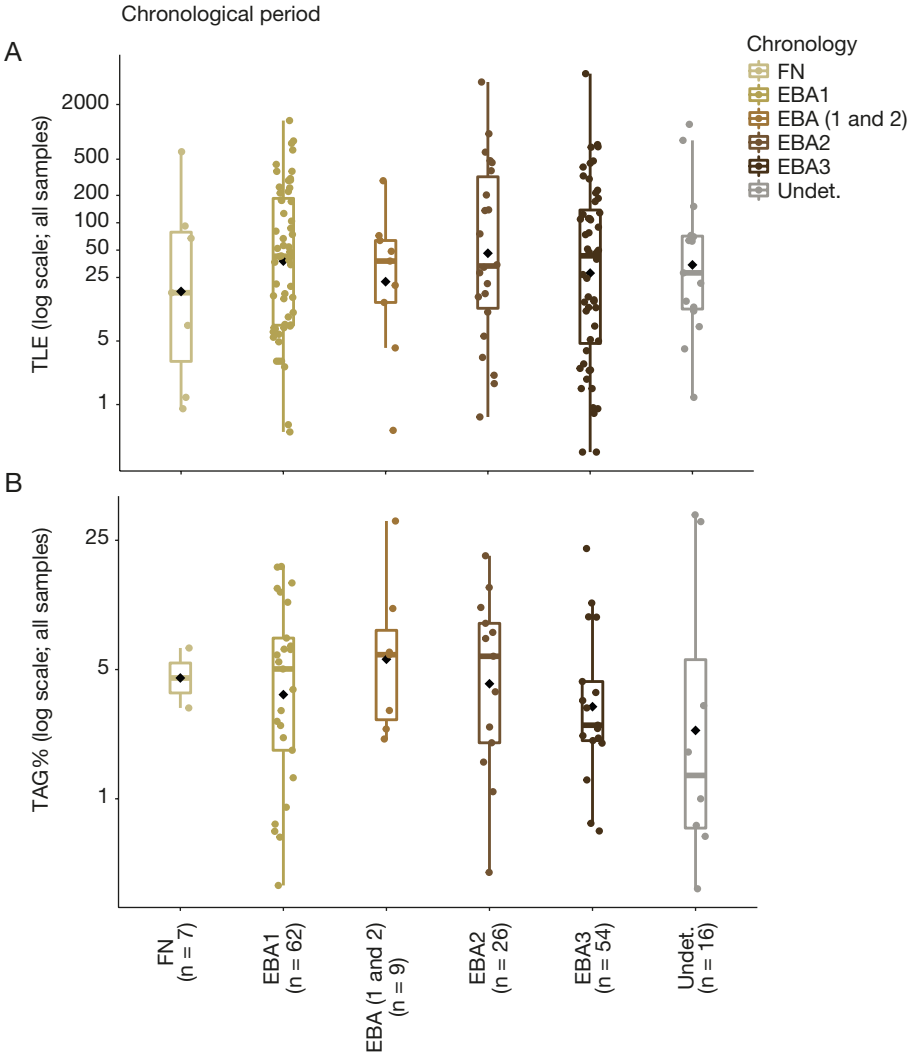
Appendix 7. — Boxplots comparing vessel fragment types with: **A**, total lipid extract (TLE); **B**, triacylglycerol preservation (TAG%); **C**, average pot wall thickness; **D**, identification of molecular heating markers. **C**, **D**, do not include samples from 2019 study. **Black diamonds** indicate mean value. R Markdown file for plots available in Appendix 14. Abbreviations: **TAG**, triacylglycerol(s); **TLE**, total lipid extract.



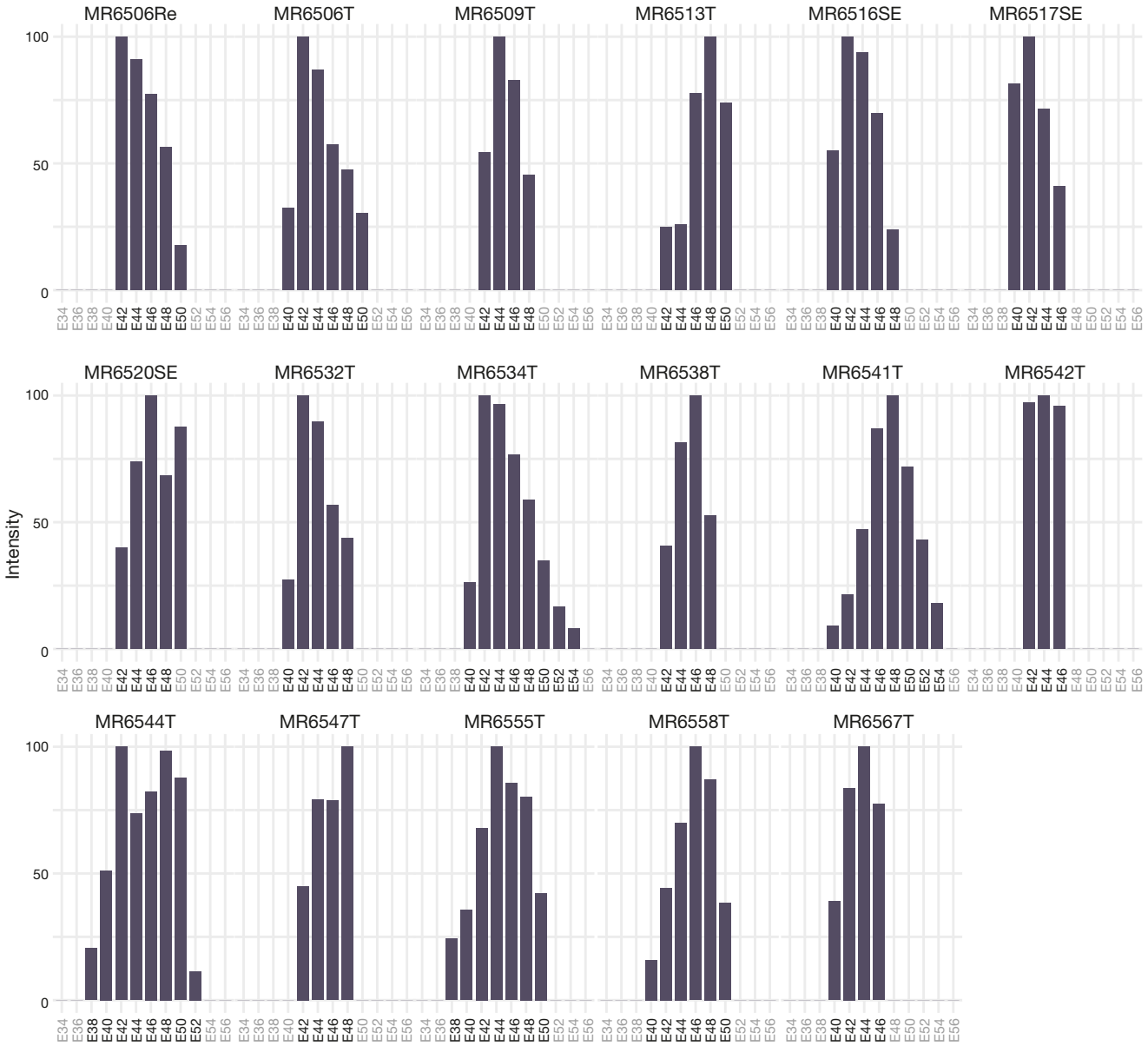
Appendix 8. — Boxplots comparing the year of excavation with: **A**, total lipid extract (TLE); **B**, triacylglycerol preservation (TAG%). **A**, **B**, include samples from 2019 study. **Red diamonds** indicate mean value. R Markdown file for plots available in Appendix 14. Abbreviations: **BAL**, Beg ar Loued; **TAG**, triacylglycerol(s); **TLE**, total lipid extract.



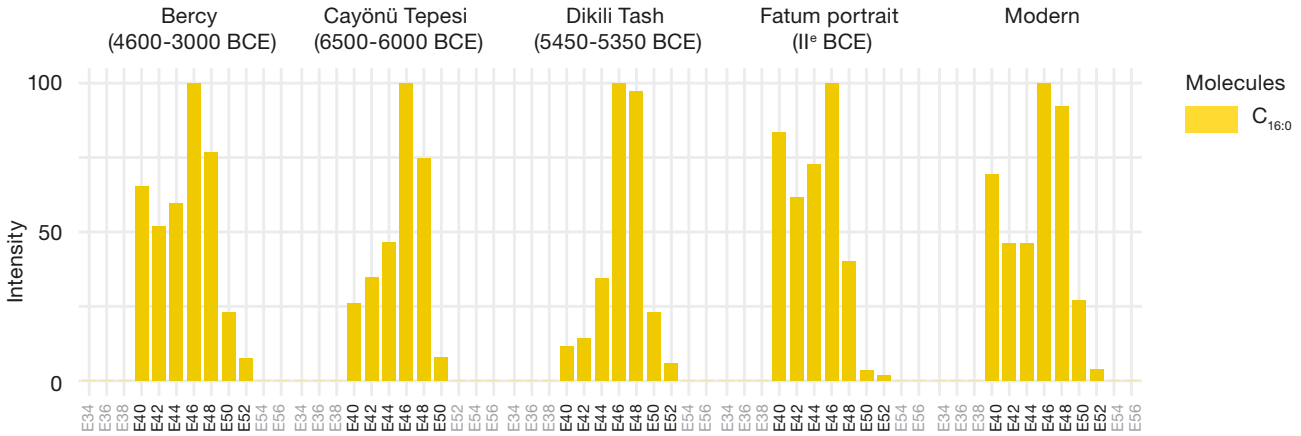
Appendix 9. — Boxplots comparing the prehistoric chronological period with: **A**, total lipid extract (TLE); **B**, triacylglycerol preservation (TAG%). **A**, **B**, include samples from 2019 study. **Black diamonds** indicate mean value. R Markdown file for plots available in Appendix 14. Abbreviations: **EBA**, Early Bronze Age; **FN**, Final Neolithic; **TAG**, triacylglycerol(s); **TLE**, total lipid extract..



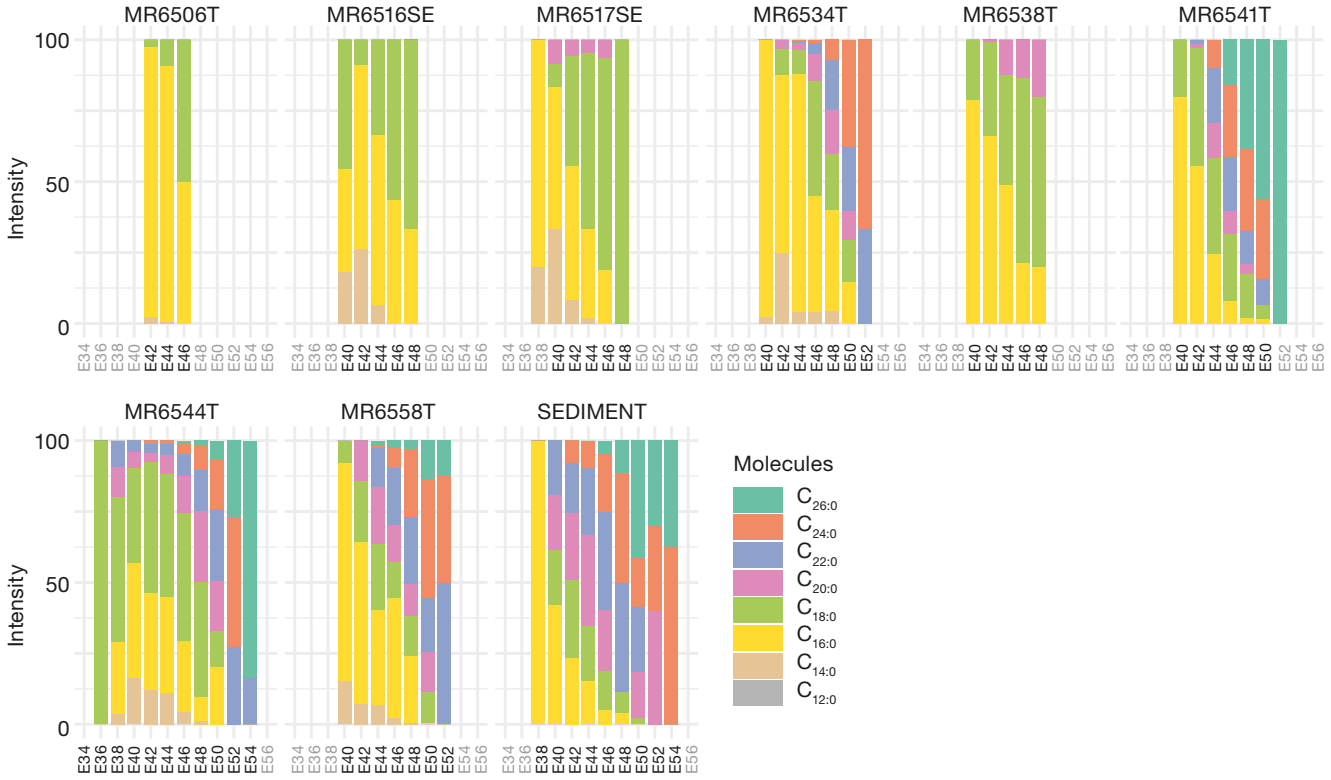
Appendix 10. — Plots with ester profiles (Beg ar Loued). R Markdown file for figure available in Appendix 14.



Appendix 11. — Plots with published examples of ester profiles of beeswax (extracted from Mirabaud *et al.* 2007). R Markdown file for figure available in Appendix 14.



Appendix 12. — Plots with percent composition of wax ester profiles (Beg ar Loued). R Markdown file for figure available in Appendix 14.



Appendix 13. — R Markdown file with code used to make all figures for the article: https://doi.org/10.5852/cr-palevol2024v23a1_s2

Appendix 14. — R Markdown file with code used to make all figures for Appendices 5-12: https://doi.org/10.5852/cr-palevol2024v23a1_s3