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Studying the use of the pots from the Pendimoun rock shelter: considerations about organic residue analysis of Mediterranean Neolithic pottery

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Abstract: The detailed stratigraphy and in-depth study of the pottery *chaîne opératoire* at the Pendimoun rock shelter make it an ideal context for studying the use of the initial pottery in the North-Western Mediterranean. However, the first batch of lipid extraction, carried out using the traditional dichloromethane/methanol method, provided very scarce information. The combination of multiple factors of climatic and sedimentary origin and related to the ceramic matrix is responsible for the almost complete degradation of lipids that are free or weakly bound to the ceramic matrix. These low extraction yields have questioned the interpretation of lipid extracts, by revealing the presence of molecules of uncertain origin and by suggesting specific uses of pottery. Additionally, these data were considered in order to optimise the extraction protocol and release the lipid fraction strongly bound to the ceramic wall. The approach set up for the ceramics of the Pendimoun rock shelter could in the future be a guideline for the analysis of the contents of Mediterranean Neolithic pottery, which has been little explored to date.

Keywords: organic residue analysis, pottery use, Mediterranean, lipid preservation

Résumé : La stratigraphie détaillée et l'étude approfondie de la chaîne opératoire céramique à l'abri Pendimoun en font un contexte idéal pour étudier l'usage des premières poteries de Méditerranée nord-occidentale. Les premières extractions des lipides des parois des céramiques, réalisées avec la méthode traditionnelle d'extraction au dichlorométhane/méthanol n'ont pourtant livré que très peu d'informations. La conjugaison de multiples facteurs d'origine climatique, sédimentaire et liés à la matrice céramique est en effet responsable de la dégradation quasi-complète de la fraction « libre » des lipides ou faiblement liée à la matrice céramique. Ces faibles rendements d'extraction ont permis d'interroger les modes d'interprétation des extraits lipidiques, en révélant la présence de molécules à l'origine incertaine et en questionnant les modes d'utilisation des récipients céramiques. La prise en compte de ces données a, de plus, permis d'optimiser les protocoles d'extraction, afin d'obtenir plus informations, en accédant notamment à la fraction lipidique fortement liée à la paroi céramique. La démarche mise en place pour étudier l'usage des céramiques de l'abri Pendimoun pourra à l'avenir servir de feuille de route pour l'analyse du contenu des céramiques néolithiques méditerranéennes, encore peu exploré.

Mots-clés : résidus organiques amorphes, usage des céramiques, bassin méditerranéen, préservation des lipides

The Mediterranean is one of the two main routes along which the Neolithic economy spread from the Near East to Western Europe, but surprisingly few studies have focused on exploring the content of the earliest pottery that appeared on these coasts during this period (Breu *et al.*, 2021; Debono Spiteri *et al.*, 2016; Tarifa-Mateo *et al.*, 2019). Organic residue analyses have however proved their high informative potential, in particular based on the study of several hundred vessels dating from the Neolithic in Central and Northern Europe. These analyses provided crucial information for understanding the Neolithic phenomenon, revealing, for example, the growing importance of dairy products as farming spreads through Europe (*e.g.* Copley *et al.*, 2005a; Cramp *et al.*, 2014; Salque *et al.*, 2013; Smyth and Evershed, 2016), or the persistence of the exploitation of aquatic products around the Baltic Sea, despite the introduction of farming practices (*e.g.* Courel *et al.*, 2020; Craig *et al.*, 2007 and 2011; Heron *et al.*, 2015). The study of the content of Early Neolithic vessels of the Mediterranean seems to be a topical issue over these last few years (Breu *et al.*, 2021; Debono Spiteri *et al.*, 2016; McClure *et al.*, 2018; Tarifa-Mateo *et al.*, 2019; Whelton *et al.*, 2017), but still very limited data is available. This is particularly true for the earliest Neolithic settlement sites in the Western Mediterranean, located in the Liguro-Provençal region and in Languedoc, which are key sites for understanding the functioning of the initial farming societies in Western Europe.

The Pendimoun rock shelter, through its precise stratigraphy and the detailed ceramic study that was conducted as part of the ANR CIMO (Binder, De Stefanis *et al.*, this volume; Cassard *et al.*, this volume; Gabriele *et al.*, this volume; Gomart, Binder, Blanc-Féraud *et al.*, this volume; Lardeaux *et al.*, this volume; Mouralis *et al.*, this volume), is a unique opportunity to explore the content of pottery within such contexts even though the analysis of these samples is quite challenging, particularly due to the poor preservation of organic matter. Nevertheless, this matter of fact proved to be an excellent opportunity to address the modes of degradation of organic matter, to refine the interpretations and to optimise the extraction and analysis protocols.

This paper presents the scientific approach implemented during the study of the *Impresso-Cardial* ceramic corpus of the Pendimoun rock shelter and the questions that have gradually been raised during this work, and finally proposes a guideline for the analysis of organic residues in the Mediterranean context.

PRESERVATION OF LIPIDS IN THE POTTERY OF THE PENDIMOUN ROCK-SHELTER

Low lipid extraction yields

A first sampling of thirty-eight potsherds and four contextual sedimentary samples was performed at the site of the Pendimoun rock shelter (see materials and methods, table 1). Lipid extraction was carried out using the conventional dichloromethane/methanol extraction method (DCM/MeOH, see materials and methods; Evershed *et al.*, 1990). The extraction yields for all potsherds were very low: only two vessels provided enough lipids (> 5 µg/g lipids; Dudd *et al.*, 1999) to be interpretable. In addition to low concentrations of fatty acids ($C_{16:0}$ and $C_{18:0}$) and a linear alcohol ($C_{18}OH$), a large proportion of these samples yielded large amounts of modern contaminants, probably from plastic bag packaging (various phthalates and benzoic acid esters; fig. 1a and 1b).

The two samples that yielded lipid related to their use (AP_0001, 10.46 µg/g and AP_0022, 34.09 µg/g), are mainly composed of fatty acids ($C_{10:0}$ to $C_{18:0}$), dominated by palmitic acid ($C_{16:0}$; fig. 1a). The molecular profile of the AP_0001 sample only indicates the presence of an undetermined fat. The presence in the AP_0022 sample of unsaturated fatty acids ($C_{18:1}$ and $C_{16:1}$) in relatively large amounts, the two isomers (*threo* and *erythro* isomers) of 9,10dihydroxyoctadecanoic acid as a trace, and the prevalence of $C_{16:0}$ versus $C_{18:0}$, ($C_{16:0}/C_{18:0} = 2.4$) suggest the presence of a substance of vegetable or aquatic origin (Copley *et al.*, 2005b; Hansel and Evershed, 2009).

Lipid extracts from sediments are dominated by wax esters (W_{44} to W_{60}) and linear alcohols (fig. 1c). The alcohols identified in free form and within esters ($C_{22}OH$ to $C_{30}OH$, dominated by $C_{26}OH$), correspond to a typical plant wax profile (Gülz, 1994; Kolattukudy, 1970). $C_{18}OH$ is also present in large amounts, mainly in free form (not in esters). This compound is not ubiquitous among vegetable waxes, but has been identified in some plants (Kolattukudy, 1970). Odd-carbon-numbered linear alkanes are also present, with a maximum of C_{31}, a typical profile of epicuticular plant waxes origin (Gülz, 1994; Kolattukudy, 1970). Some compounds form an unresolved massif difficult to interpret which could be due to the presence of $C_{18:0}$ and $C_{18:1}$ amides (predominance of *m/z* 59 and 72 ions) resulting from contamination via packaging in plastic bags (Cooper and Tice, 1995). The much higher lipid yields in sediments (13.4 to 49.7 µg/g) compared to potsherds confirm that contamination from sediments in the ceramics is limited (Heron *et al.*, 1991). The main compounds identified in the sediments (long-chain alcohols, alkanes, and wax esters) are poorly soluble in water, which limits their diffusion via runoff water. However, it cannot be ruled out that the shortest compounds ($C_{16:0}$ and $C_{18:1}$ fatty acids and $C_{18}OH$ linear alcohol), which are much more soluble in water and present in small amounts in most potsherds (less than 2 µg/g each), are due to sediment contamination.

Lipid preservation in the vessels at the site (5.5% of interpretable vessels, with a maximum yield of 34.1 µg/g lipid) is very poor compared to most studies on Neolithic pottery (Craig *et al.*, 2007; Cramp *et al.*, 2014; Salque *et al.*, 2013). The time period of approximately six years between the end of the excavation and the first analyses could partly explain the degradation of lipids within the potsherds, as observed on the site of Clairvaux XIV (Drieu, 2020). However, this duration is not exceptional in organic residue analysis, suggesting that other param-

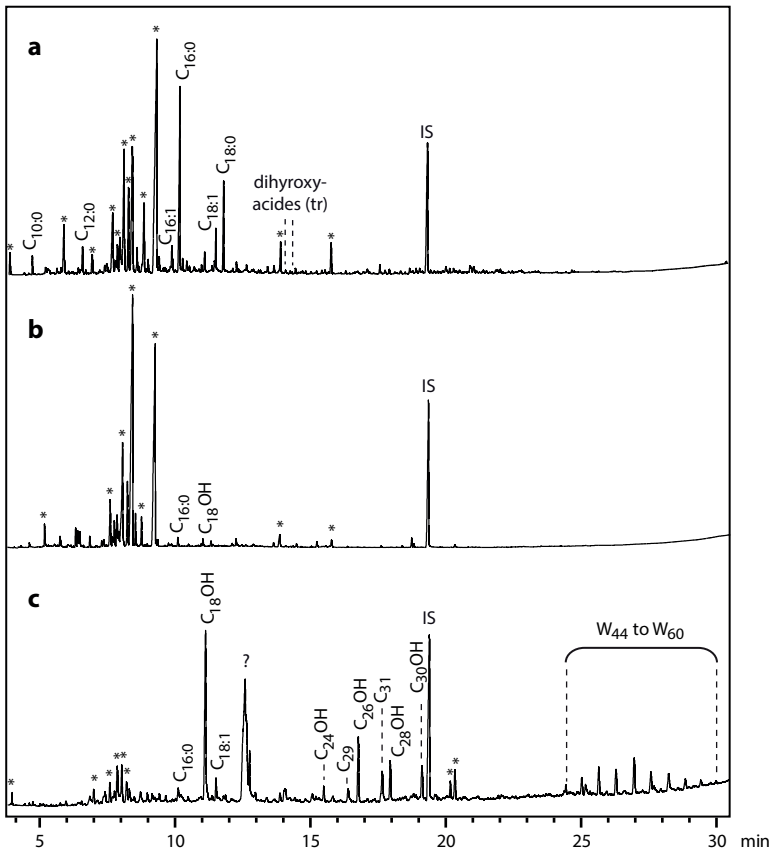

Fig. 1 – Chromatograms obtained on samples from Pendimoun, after extraction with DCM/MeOH; a: pot with preserved interpretable lipids (AP_0022); b: pot with a very low amount of interpretable lipids (AP_0001); c: sediments (sample 48045). $C_{xx:x}$: fatty acids; $C_{xx}OH$: linear alcohols; C_{xx}: linear alkanes; W_{xx}: wax esters; ?: unidentified compound; IS: internal standard; *: modern contamination.

Fig. 1 – *Chromatogrammes obtenus sur des échantillons de Pendimoun, après extraction au DCM/MeOH ; a : récipient avec lipides interprétables préservés (AP_0022) ; b : récipient avec une très faible quantité de lipides interprétables (AP_0001) ; c : sédiments (sample 48045). $C_{xx:x}$: acides gras ; $C_{xx}OH$: alcools linéaires ; C_{xx} : alcanes linéaires ; W_{xx} : esters de cire ; ? : composé non identifié ; IS : standard interne ; * : contamination moderne.*

eters, such as climatic and sedimentary conditions at the site, may have had an adverse role in preserving lipids.

Is the geographical location of the site responsible for the poor preservation of lipids at Pendimoun?

Many Mediterranean Neolithic pottery published in the literature yielded low amounts of lipids (Debono Spiteri *et al.*, 2016; Decavallas, 2011; Evershed *et al.*, 2008b; Salque *et al.*, 2012; Šoberl *et al.*, 2014). This observation has led some authors to suggest that the Mediterranean climate, characterized by hot and dry periods alternating with phases of heavy rainfall, is very unfavourable to the preservation of lipids, since it promotes both hydrolysis and oxidation mechanisms (Cramp, 2008; Evershed *et al.*, 2008a). However, some Mediterranean sites are characterised by a very good preservation of lipids in pottery, for example at Fondo Azzollini in Italy (Spiteri *et al.*, 2017) or at Cuciurpula on Corsica (Drieu *et al.*, 2018). This suggests that the Mediterranean climate may not be the main cause of lipid degradation at the Pendimoun rock shelter.

The geographical location of the Pendimoun rock shelter could be a factor in the degradation of lipids, since its location at the base of a cliff favours water circulation (Binder *et al.*, 1993), and therefore the elimination of organic matter by leaching. Calcium carbonate deposits on the potsherds testify to successive steps of humidification and drying during burial, which could also promote the degradation of lipids. These calcified deposits are distributed indifferently over the internal and external surfaces and fracture edges, in contrast to calcified deposits related to use, located only on the internal surface and which seem to favour the preservation of organic matter (Hendy *et al.*, 2018).

Soil properties may also have an influence on the preservation of lipids. The sediment samples from the site were measured for pH and total calcareous content to investigate this hypothesis by assessing their acidity (see materials and methods). These measurements reveal that the sediments of the site have a pH ranging between 8 and 9, and therefore correspond to a basic soil, typical of limestone substrates, which buffer the pH above 6.5 by a high concentration of Ca^{2+} ions (Fabian *et al.*, 2014). These results are supported by total calcareous content

measurements: sediment samples do have a high to very high total calcareous content (37 to 81% calcareous content as a percentage of dry matter). Basic conditions, such as those in Pendimoun, are slightly favourable to the development of microbial activity (DeLaune *et al.*, 1981; Moucawi *et al.*, 1981). This type of context therefore leads to a greater degradation of lipids than in the acidic contexts typical of northern Europe, but are not optimal conditions for microbial degradation (neutral pH; Hita *et al.*, 1996; Moucawi *et al.*, 1981). In addition, in a basic soil, fatty acids exist in the form of soluble salts and are therefore more easily eliminated by leaching (Evershed *et al.*, 1997).

The climate and soil properties, as a result of the geographical location of the Pendimoun rock shelter, explain, at least in part, the low lipid extraction yields from pottery. Although the degradation mechanisms are different, it is worth mentioning that bone collagen is not consistently well preserved in faunal remains from the Pendimoun rock shelter either (Le Bras-Goude *et al.*, 2006).

What influence do the properties of Pendimoun vessels have on the preservation of lipids?

The porosity of the vessels also seems to have an impact on the preservation of lipids. First analyses have been performed using scanning electron microscopy (SEM), but they did not allow to study the smallest pores (<10 µm) and their distribution. Mercury intrusion porosimetry measurements, however, provided sufficiently accurate data to be compared with lipid extraction yields for a dozen vessels from the Pendimoun rock-shelter. The methodology, results and interpretations are presented elsewhere (Drieu *et al.*, 2019), and are briefly reported here. These measurements revealed that some pots made of granitic paste have both a very small amount of small diameter pores (less than 40% of pores with diameter < 1 µm) and very little lipids preserved (less than 1 µg/g of lipids). A small pore diameter limits the access of microorganisms and water, which are largely responsible for the degradation of the lipids in archaeological pottery (Drieu *et al.*, 2019). Some of the granitic pots at the site therefore seem to have a limited capacity to preserve lipids. No conclusion can be drawn on the function of these pots, since, even if they absorbed lipids at the time of use, they were likely to be rapidly consumed by the microorganisms and degraded by hydrolysis, in the absence of effective protection of the ceramic matrix. Concerning the rest of the pots, the porosity ranges (more than 40% of pores with diameter < 1 µm), although showing various profiles, are all favourable to the preservation of lipids (Drieu *et al.*, 2019).

The mineralogical composition of the ceramic pastes of the Pendimoun rock shelter does not seem to have an influence on the preservation of lipids, since the comparison of extraction yields with the types of paste did not reveal any correlation (fig. 2).

POOR PRESERVATION OF LIPIDS: AN OPPORTUNITY TO EXAMINE MORE GLOBALLY THE USE OF ORGANIC PRODUCTS BY ANCIENT SOCIETIES

Molecular series opening up new avenues for exploration

The poor preservation of molecules related to the use of the vessels (*e.g.* fatty acids) has made it possible to identify very clearly a homologous series of odd- and even-carbon-numbered alkanes, dominated by C_{22} or C_{23} (between 1 and 6 µg/g in total, fig. 3), in some samples. Similar series of alkanes have been identified many times in the literature, for example in other *Impresso-Cardial* sites, in Southern Italy and Spain (Debono Spiteri, 2012), but their interpretation remains a matter of debate. In the case of the pottery recovered from the Pendimoun rock shelter, several hypotheses may be considered to explain their presence: use of bituminous products during the Neolithic, modern contamination with petroleum derivatives (glue, varnish, marker solvent, etc.), sediment contamination, organic material degraded at high temperature, etc. Further experiments and analyses are ongoing to investigate the origin of these compounds.

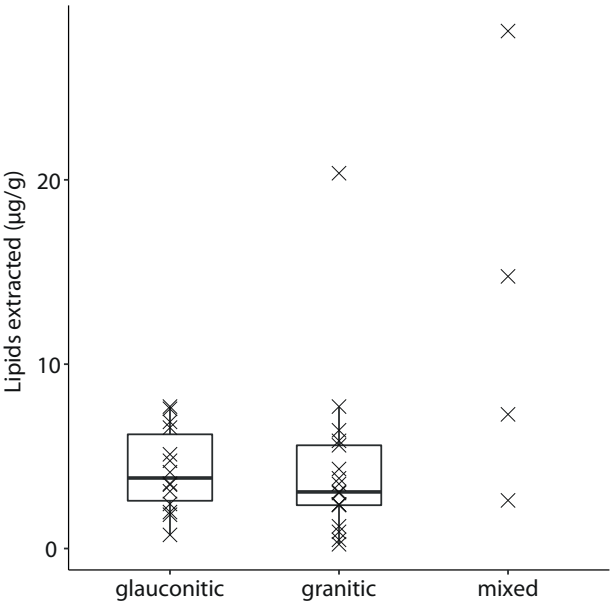

Fig. 2 – Comparative extraction yields by type of ceramic paste from the Pendimoun rock shelter. The box plot represents the minimum and maximum, the median and the first and third quartiles of the dataset. Each sample is represented by a "×". Due to the small number of samples of mixed paste, the data are not represented as a box plot.

***Fig. 2** – Rendements d'extraction comparés par type de pâte céramique de l'abri Pendimoun. Les diagrammes à moustache représentent les minimum et maximum, la médiane et les premier et troisième quartiles du jeu de données. Chaque échantillon est représenté par un « × ». Du fait du faible nombre d'échantillons de pâte mixte, les données ne sont pas représentées sous la forme d'un diagramme à moustache.*

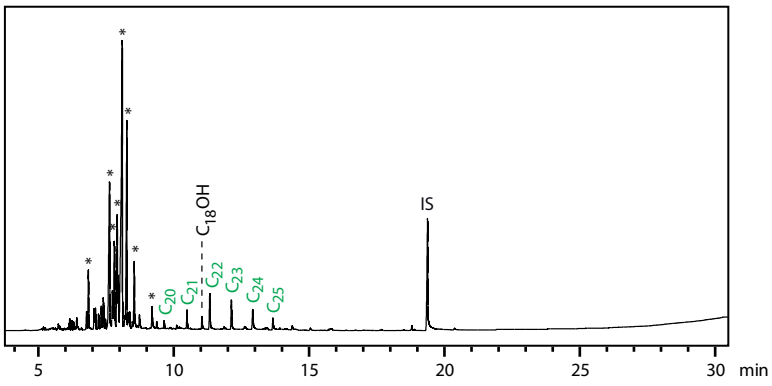

Fig. 3 – Chromatogram of a lipid extract from sample AP_48021, showing the distribution of alkanes (in green). $C_{xx}OH$: linear alcohols; C_{xx}: linear alkanes; IS: internal standard; *: modern contamination.

Fig. 3 – *Chromatogramme d'un extrait lipidique de l'échantillon AP_48021, montrant la distribution des alcanes (en vert). $C_{xx}OH$: alcools linéaires ; C_{xx} : alcanes linéaires ; IS : standard interne ; * : contamination moderne.*

Questioning the absence of preserved lipids in terms of use

It is important to note that the absence of lipids in the pottery from the Pendimoun rock shelter could be explained, not by the degradation of lipids, but by the way pottery was used during the Neolithic. First, some of the pots may have been used to store or process low-fat substances, such as water, cereals or wild fruits, for example, which are well documented on site (Binder *et al.*, 2020; Delhon *et al.*, 2020). Such a hypothesis has, for example, been raised for other sites within the *Impresso-Cardial* complex in Southern Italy (Spiteri *et al.*, 2017). The pots of the Pendimoun rock shelter could also have been used to contain or transform fatty or waxy materials, but to a very low intensity. The status of the site, which seems to be, at the beginning of the 6th millennium BC, a temporary occupation related to specialised activities (Binder *et al.*, 2020), could be correlated to a low intensity of use of ceramic vessels. A parallel can be drawn with the Neolithic site of Mala Triglavca in Slovenia, a temporarily occupied site whose pottery has preserved less lipids (48% of the pots with interpretable content) than a contemporary site, occupied all year round (Moverna vas, 66% of containers with interpretable content; Šoberl *et al.*, 2014). As one of the main activities represented at Pendimoun is the manufacturing of pottery, it can be suggested that one part of this production, made of locally produced glauconitic earth (Gabriele *et al.*, this volume; Gabriele, 2014), has not or only rarely been used. Other types of containers, made of perishable material, may also have been used at the site. Use-wear analysis on lithic tools suggests, for example, plant treatments that may be related to basketry activities (Binder *et al.*, 2020; De Stefanis, 2018).

ADAPTING THE ANALYTICAL PROTOCOL

To complete the first data obtained by DCM/MeOH extraction, a second sampling of fifty-two potsherds (materials and methods, table 1) was carried out to perform an extraction with acidified methanol, more recently developed for the analysis of archaeological samples (H_2SO_4/MeOH; Correa-Ascencio and Evershed, 2014; Craig *et al.*, 2013). Interpretations regarding the contents of the pots, proposed based on these extracts, are presented elsewhere (Drieu *et al.*, 2021), but here we will discuss the results in terms of extraction yields.

The acidified methanol method has proven to be much more effective (88% of the pots yielded enough lipids to be interpretable in terms of use, and delivered up to 547 µg/g (fig. 4), compared to 5% of interpretable potsherds, maximum 34 µg/g with DCM/MeOH) and has led to isotopic measurements on fatty acids, in order to work on the origin of these fats (Drieu *et al.*, 2021). This method is very effective because acid conditions break the chemical bonds that bind some of the organic molecules to the ceramic matrix. In addition, sulphuric acid dissolves part of the ceramic matrix, as well as carbonate deposits (Correa-Ascencio and Evershed, 2014), present on many potsherds of the Pendimoun rock shelter.

It is noteworthy that after heating the samples in acidified methanol, the solvent sometimes had become basic or neutral, indicating that all the acid had been consumed

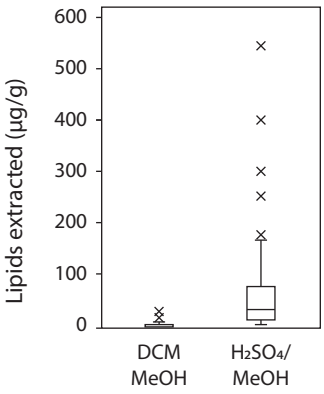

Fig. 4 – Comparative extraction yields on samples from the Pendimoun rock shelter. The box plot represents the minimum and maximum, the median and the first and third quartiles of the dataset. Outliers are represented by a "×".

Fig. 4 – *Rendements d'extraction comparés sur des échantillons de l'abri Pendimoun. Les diagrammes à moustache représentent les minimum et maximum, la médiane et les premier et troisième quartiles du jeu de données. Les valeurs extrêmes sont représentées par un « × ».*

during the reaction and that additional reactant is required to complete the trans-methylation. Post-depositional carbonate deposits and carbonate inclusions of potsherds are probably the cause of the unintentional consumption of sulphuric acid, although it was introduced in large amounts (about 20% v/v). When extracting lipids from archaeological pots, it is therefore important, first, to consider the nature of the ceramic paste with petrographic or mineralogical analyses in order to adapt the amount of acid added before the reaction. In addition, it is crucial to check the pH of the solvent at the end of the reaction, in order to avoid hydrolysing and methylating only a small part of the lipids preserved in the pots.

The difference in yield between the two extraction methods is probably due to the parameters mentioned above: climate, leaching, soil pH, and specific porosity of some vessels. Their combination has certainly led to an almost total disappearance of the "free" lipids within the vessel wall, *i.e.* simply trapped within the pores, or bound to the ceramic matrix via weak chemical bonds, which could explain the low extraction yields with DCM/MeOH. In contrast, lipids strongly bound to the ceramic walls (and extracted only using acidified methanol extraction) have resisted, at least partially, these degradations. In contexts that favour the degradation of "free" lipids, such as the Pendimoun rock shelter, it should be noted that the chemical signal obtained by extraction under acidic conditions may therefore be biased, since only part of the signal related to use has been preserved. The binding of the lipids to the ceramic matrix could have been catalysed by heating, *e.g.* during successive cooking episodes when the lipids were already absorbed into the pottery wall (Copley *et al.*, 2005b; Correa-Ascencio and Evershed, 2014; Regert *et al.*, 1998). This would mean that at the Pendimoun rock shelter, it would not be possible to study the uses such as storage or non-thermal transformation, unless subsequent heating episodes (*e.g.* reuses) had strengthened the bonds to the ceramic matrix.

It is important to note that, while extraction under acidic conditions is particularly effective, it is always interesting to extract samples with DCM/MeOH, at least the best preserved ones, in order to identify specific compounds, such asalkylresorcinols (cereals markers; Colonese *et al.*, 2017; Hammann and Cramp, 2018), and to precise the natural origin of fat and wax using triacylglycerols and wax esters potentially preserved in small amounts. This extraction step has, for example, helped to identify beeswax from wax esters in a pot from the Pendimoun rock shelter (Drieu *et al.*, 2021).

CONCLUSION: A GUIDELINE FOR THE ANALYSIS OF THE CONTENT OF NEOLITHIC POTTERY IN THE MEDITERRANEAN

The approaches implemented for the analysis of the content of the pottery from the Pendimoun rock shelter led to the refinement of the organic residue analysis process and the opening of new fields of investigation. The results obtained, both positive and negative, can guide future lipid studies in Mediterranean Neolithic pots:

– Contexts with acidic soils (*e.g.* Corsica, North-East Sardinia and the Tuscan archipelago, South Calabria, Central Croatia) are probably the most favourable to the preservation of lipids in the Mediterranean.
– Cave sites are also to be favoured, as they are less exposed to seasonal variations in humidity and temperature (Debono Spiteri, 2012; Tarifa-Mateo *et al.*, 2019). For example, preliminary analyses carried out on pots from the Arene Candide cave (data not presented) have shown good lipid preservation, including well-preserved triacylglycerols and wax esters.
– As a general rule, and all the more so for open-air sites with neutral or slightly basic soils, extraction with acidified methanol is to be preferred, as proposed for the contexts of the Iberian Peninsula (Breu *et al.*, 2021; Breu Barcons, 2016; Tarifa-Mateo *et al.*, 2019). As many Mediterranean clays are rich in carbonate inclusions, the extraction solvent must be highly concentrated in sulphuric acid (according to the protocol developed by O. E. Craig and his team; 2013) and the pH must be tested at the end of the reaction. DCM/MeOH extractions can be performed in a second step on an additional sub-sample to refine the interpretations.
– Whenever possible, sampling should include contextual sediment samples associated with the pottery to verify potential contamination.

Based on this framework, the analysis of the content of Mediterranean Neolithic vessels has great potential, still largely unexplored, to understand the mechanisms of the neolithisation process along the Mediterranean route.

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MATERIALS AND METHODS

Study of soil acidity

A Bernard calcimeter was used for the total calcareous content measurement, after a calibration with a known mass of pure $CaCO_3$. The raw sediment samples (weighed, between 300 mg and 1 g) were introduced into a filter paper papillote in the flask containing hydrochloric acid. After stirring to achieve a total reaction, the position of the glass ampoule was adjusted to match its water level with that of the measuring cylinder. The water level, corresponding to the volume of CO_2 released, was measured on the graduated cylinder ($V_{measured}$). The percentage of total calcareous content was calculated from the following formula:

For the pH measurement, 2 g of sediment was sieved to 2 mm. The sample was poured into a 50 ml beaker and 25 ml of distilled water was added. After a magnetic stirring for a few minutes, the solution was left to stand for half an hour. The pH meter (Eutech PC 450, Thermo Scientific) was calibrated with three buffer solutions: a neutral value (pH 7.00) and two values that surround the measurements made (pH 4.00 and 9.81). The pH of the sediment solutions was then measured under magnetic stirring.

Study of organic matter

List of samples

The list of sherds and contextual sediments analysed is presented in table 1.

The contextual sediment samples were selected from the samples taken at the time of the excavation for bioarchaeological analysis (phytoliths).

Table. 1 – List of samples analysed at the Pendimoun rock shelter.
***Tab. 1** – Liste des échantillons analysés à l'abri Pendimoun.*

Type	Vase	Culture	Sample	m²	US (sampling)	Area	Extraction method	
							DCM/ MeOH	H2SO4/ MeOH
Pottery	Unattributed potsherd	Impressa	AP 26816	O13	5711 (26717)	South	X	
Pottery	Unattributed potsherd	Impressa	AP 26618	O13	5711 (26550)	South	X	
Pottery	Unattributed potsherd	Impressa	AP 26702	O12	5711 (26551)	South	X	
Pottery	Unattributed potsherd	Impressa	AP 26938	O14	5711 (26827)	South	X	
Pottery	Unattributed potsherd	Impressa	AP 26855	O13	5711 (26716)	South	X	
Pottery	Unattributed potsherd	Impressa	AP 27246	N12	27226 (27243)	South	X	
Pottery	Unattributed potsherd	Impressa	AP 27452	N12	27354 (27371)	South	X	
Pottery	Unattributed potsherd	Impressa	AP 27450	N12	27354 (27371)	South	X	
Pottery	Unattributed potsherd	Impressa	AP 26650	O13	5711 (26548)	South	X	
Pottery	Unattributed potsherd	Impressa	AP 26986	O13	5711 (26900)	South	X	
Pottery	Unattributed potsherd	Impressa	AP 48123	L22	47801 (48120)	North	X	
Pottery	Unattributed potsherd	Impressa	AP 48174	L22	48159 (48142)	North	X	
Pottery	Unattributed potsherd	Impressa	AP 47548	M22	47647 (47724)	North	X	
Pottery	Unattributed potsherd	Impressa	AP 48084	M22	47801 (48046)	North	X	
Pottery	Unattributed potsherd	Impressa	AP 48095	M22	47801 (48046)	North	X	
Pottery	Unattributed potsherd	Impressa	AP 47544	M22	47647 (47724)	North	X	

Type	Vase	Culture	Sample	m²	US (sampling)	Area	Extraction method	
							DCM/ MeOH	H2SO4/ MeOH
Pottery	Unattributed potsherd	Impressa	AP 47987	M23	47801 (48150)	North	X	
Pottery	Unattributed potsherd	Impressa	AP 48026	L22	47801 (48120)	North	X	
Pottery	Unattributed potsherd	Impressa	AP 48233	L21	47803 (48229)	North	X	
Pottery	Unattributed potsherd	Impressa	AP 47986	M23	47801 (48150)	North	X	
Pottery	Unattributed potsherd	Impressa	AP 47383	L22	47801 (48120)	North	X	
Pottery	Unattributed potsherd	Impressa	AP 48021	M22	47801 (48045)	North	X	
Pottery	Unattributed potsherd	Impressa	AP 47855	L23	47799 (47958)	North	X	
Pottery	Unattributed potsherd	Impressa	AP 47930	M22	47800 (47915)	North	X	
Pottery	AP_0001	Impressa	AP 58949	O16	VI-VII	South	X	
			AP 57 O16	O16	VI (R7)	South		X
Pottery	AP_0002	Impressa	AP 59393	P15	IX	South	X	
			AP 36 P15	P15	IX (R5)	South		X
Pottery	AP_0003	Impressa	AP 48122	L22	47801 (48120)	North		X
Pottery	AP_0004	Impressa	AP 48066	L20	30 (48069)	North		X
Pottery	AP_0007	Impressa	AP 28869	O13	28535 (28691)	South		X
Pottery	AP_0008	Impressa	AP 27778	N12	27743 (27751)	South	X	
			AP NC Q15	Q15	IX	South		X
Pottery	AP_0011	Impressa	AP 30061	M11	30016	South	X	X
Pottery	AP_0014	Impressa	AP 359 O15	O15	(RC)	South		X
Pottery	AP_0015	Impressa	AP 145 O16	O16	IX (R1)	South	X	X
Pottery	AP_0016	Impressa	AP 28461	O13	5711 (28382)	South	X	
			AP 28451	O14	5711 (28381)	South		X
Pottery	AP_0018	Impressa	AP 244 M19	M19	(RC)	North	X	
			AP 29120	O12	28890 (28939)	South		X
Pottery	AP_0019	Impressa	AP 5767	O14	5711 (5723)	South		X
Pottery	AP_0020	Impressa	AP 26806	O13	5711 (26717)	South		X
Pottery	AP_0022	Impressa	AP 58367	O15	IX	South	X	
			AP 154 Q17	Q17	TGC	South		X
Pottery	AP_0027	Impressa	AP 290 O15	O15	NIV 12 (R3)	South		X
Pottery	AP_0028	Impressa	AP 26880	M11	26884 (26885)	South	X	X
Pottery	AP_0029	Impressa	AP 59391	P15	IX	South	X	
			AP 130 O15	O15	IXB	South		X
Pottery	AP_0030	Impressa	AP 42311	L21	40549 (42274)	North		X
Pottery	AP_0036	Impressa	AP 26155	P13	26040 (26055)	South		X
Pottery	AP_0037	Impressa	AP 223 O15	O15	NIV 10 (R1)	South		X
Pottery	AP_0038	Impressa	AP 198 O15	O15	NIV 9-10 (R1)	South		X
Pottery	AP_0039	Impressa	AP 47974	M22	47647 (47915)	North	X	
			AP 47976	N22	47647 (47915)	North		X
Pottery	AP_0040	Impressa	AP 485 K19	K19	SEP2 (R9)	South		X
Pottery	AP_0042	Impressa	AP 47107	L21	45972 (47106)	North		X
Pottery	AP_0045	Impressa	AP 50002	I19	out of stratigra-phy	North	X	X

Type	Vase	Culture	Sample	m²	US (sampling)	Area	Extraction method	
							DCM/ MeOH	H2SO4/ MeOH
Pottery	AP_0046	Impressa	AP 46111	K20	43829 (46066)	North		X
Pottery	AP_0052	Impressa	AP 46245	J20	45437 (46245)	North		X
Pottery	AP_0053	Impressa	AP NC O15	O15	NIV 11-12	South		X
Pottery	AP_0081	Impressa	AP 348 O15	O15	(RC)	South		X
Pottery	AP_0137	Impressa	AP 47004	L21	46832 (46997)	North		X
Pottery	AP_0146	Cardial	AP 392 N16	N16	14'C	South		X
Pottery	AP_0148	Cardial	AP 40042	L20	19011 (19459)	North		X
Pottery	AP_0152	Cardial	AP 32D Q16	Q16	IX	South		X
Pottery	AP_0157	Cardial	out of stratigraphy	_	_	_		X
Pottery	AP_0158	Cardial	AP 24964	O12	24087 (24955)	South		X
Pottery	AP_0163	Cardial	AP 23590	O11	23588 (23589)	South		X
Pottery	AP_0164	Cardial	AP 5562	P14	5557 (5559)	South		X
Pottery	AP_0165	Cardial	AP 21687	N15	21069 (21618)	South		X
Pottery	AP_0166	Cardial	AP 23590	O11	23559 (23589)	South		X
Pottery	AP_0170	Cardial	AP 18802	L20	18390 (18749)	North		X
Pottery	AP_0171	Cardial	AP 23491	M13	22801 (23284)	South		X
Pottery	AP_0174	Cardial	AP 42410	L23	42370 (42405)	North		X
Pottery	AP_0175	Cardial	AP NC N22	N22	(Z 240-245)	North		X
Pottery	AP_0176	Cardial	AP 41845	L22	19000 (41700)	North		X
Pottery	AP_0178	Cardial	AP NC N22	N22	(Z 250-255)	North		X
Pottery	AP_0180	Cardial	AP 19956	K21	19011 (19956)	South		X
Pottery	AP_0181	Cardial	AP NC N22	N22	(Z 220)	North		X
Pottery	AP_0182	Cardial	AP 40326	L21	19908 (40230)	North		X
Pottery	AP_0187	Cardial	AP 19153	M22	17440 (19025)	South		X
Pottery	AP_0206	Cardial	AP 40016	L23	17440 (19498)	North		X
Pottery	AP_0207	Cardial	AP 41024	I22	17440 (40738)	North		X
Pottery	AP_0212	Cardial	AP 21467	N14	21466 (21467)	South		X
Sediment	_	Impressa	AP 48045	M22	47801 (48045)	North	X	
Sediment	_	Impressa	AP 48229	L21	47803 (48229)	North	X	
Sediment	_	Impressa	AP 26717	O13	5711 (26717)	South	X	
Sediment	_	Impressa	AP 27751	N12	27743 (27751)	South	X	

Extraction using DCM/MeOH

The surfaces of the sample were removed with a clean scalpel to eliminate the top layers of ceramics that were potentially contaminated by sediment or handling. The samples were then crushed using a clean mortar and pestle and 20 µl of an internal standard solution (n-C_{34} in hexane at 1 mg/ml) was added to the ceramic powder. The lipids were extracted in 10 ml of DCM/MeOH (2:1, v/v) by two ultrasonications of 15 minutes each. The samples were then centrifuged (15 min at 3000 rpm) and the supernatant liquid was recovered. It was then evaporated under nitrogen flow and dissolved in 500 µl of DCM/MeOH solution to obtain the Total Lipid Extract (TLE). An aliquot of the TLE of each sample (100 µl) was selected and then evaporated to dryness under nitrogen flow. It was then derivatised with 50 µl of BSTFA (N,Obis(trimethylsilyl)trifluroacetamide, containing 1% trimethylchlorosilane) and heated for one hour at 70°C. After cooling, the BSTFA was evaporated under nitrogen flow and the samples were dissolved in cyclohexane, before injection in chromatography.

GC analyses were performed on an Agilent 7890A chromatograph equipped with a flame ionization detector (FID). The injection of 1 µl of sample was carried out via an on-column injector. The compounds were separated in a DB-5MS apolar capillary column (15 m × 0.32 mm, internal diameter: 0.1 µm; Agilent). Hydrogen was used as a carrier gas with a flow maintained at 2 ml/min during 22 min, then increased to 4 ml/min during 4 min and finally increased to 6 ml/min during 16 min. The oven temperature program has been set as follows: temperature increase from 50 to 100°C at 15°C/min, then increase to

375°C at 10°C/min. The detector temperature was set at 375°C.

GC-MS analyses were performed using a Shimadzu GC2010PLUS chromatograph equipped with an injector used in splitless mode at 280°C and a DB-5HT non-polar capillary column (15 m × 0.32 mm, internal diameter: 0.1 µm). The following temperature program was used: 1 min at 50°C, followed by an increase to 100°C at 15°C/min, an increase to 240°C at 10°C/min, an increase to 380°C at 20°C/min, and a temperature hold time of 7 min. The carrier gas (helium) flow rate at the head of the column was set at 3 ml/min. The molecules were detected in a QP2010ULTRa mass spectrometer equipped with a quadrupole analyser and a 70 eV Electron Ionization (EI) source. The temperature of the source was set at 200°C and that of the interface at 280°C. The mass spectrometer operated in full scan mode with a mass scan of *m/z* 50 to 950.

Extraction with acidified methanol

This extraction method and the instruments used to study the extracts are detailed in Drieu *et al.* (2021).

Dataset

DCM/MeOH extraction data for pottery and contextual sediment samples are available in annex 1 and 2 respectively. Dataset for extractions after acid methylation are available in Drieu *et al.*, 2021.

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