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# Relationships between lipid profiles and use of ethnographic pottery: an exploratory study

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## Abstract

Investigating the organic content of archaeological pottery has largely focused on identifying food commodities, but their use and mode of processing still need to be thoroughly investigated. The present study aims to explore the diversity of organic residue absorption patterns, over a wider range of functions than previously studied by experimentation, by analysing ceramics still in use today. A field survey in Bedik Country, Senegal, where the use of pottery is still alive, was conducted to document the uses of ceramics and to interview potters and users of the vessels. As a preliminary study, nine ceramics whose use was recorded were investigated through 59 samples for their absorbed molecular profiles, lipid concentrations, and the preservation of triglycerides and C<sub>18</sub> unsaturated fatty acids. The interpretations were first carried out as a blind test and then compared with the actual use. Lipid concentrations and molecular profiles indicated a diversity of contents, and the comparison of samples taken along the vertical transects of the vessels resulted in pottery function hypotheses that were broadly aligned with the actual uses. Cooking pots for fat-rich products were successfully identified, but the various documented patterns showed that lipid accumulation in ceramics is more complex than expected. Although caution is required to adopt this approach for archaeological pots, the vessel for fermenting plant products has been identified. Last, this work pointed out that ceramics can be used for a wider range of purposes than those usually considered for archaeological pottery, such as steaming or cooking non-food products.

## Keywords

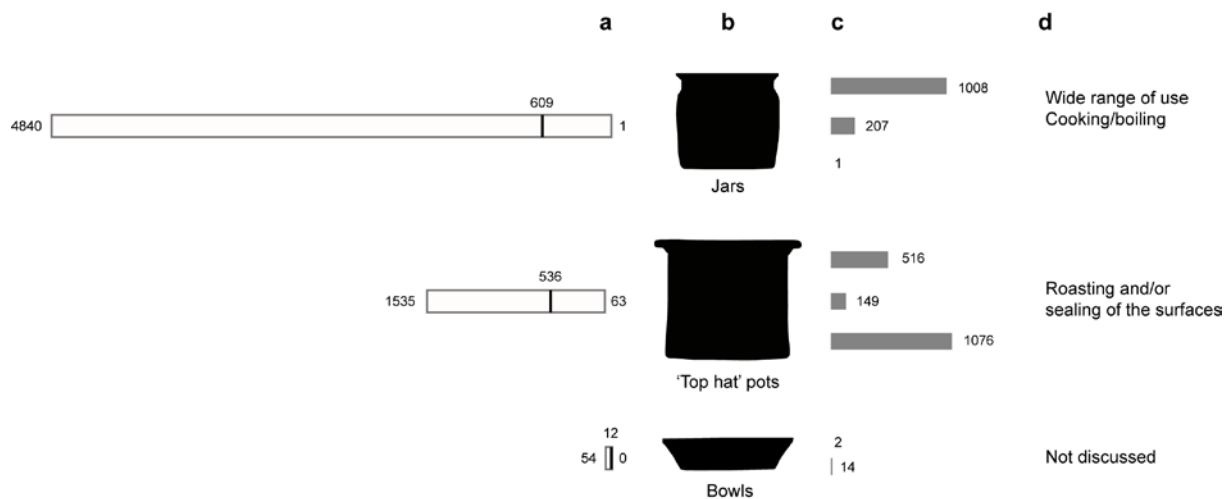
Pottery function, Ethnoarchaeology, Organic residues analysis, Senegal, Africa

## Introduction

Pottery is one of the most common artefacts found at Holocene archaeological sites, and shapes and decorations are frequently used to identify and define ancient cultures. However, ceramics are, above all, containers used to fulfil various functions related to food (storage, transport, preparation, including cooking and non-thermal transformation, service, consumption), hygiene, body care and medicine (e.g. heating water for body wash, diffusing odoriferous substances, storage of unguents) and craft (metal working, pigments and dyes manufacturing, hide, tanning, adhesive making, etc.) within the societies that made and used them (e.g. Charters et al., 1993; Drieu et al., 2020a; Lucquin et al., 2007; Maniatis et al., 2001; Regert et al., 2003; Rice, 1987; Rovira and Ambert, 2002; Salque et al., 2013; Šoberl et al., 2014). For a given category of activity such as cooking, various processes may be implemented: boiling or simmering, roasting or braising, steaming, frying or grilling (Bats, 1988; Charters et al., 1993; Corbeau and Poulain, 2002; Rice 1987).

Studying the contents of a vessel provides valuable information regarding its use. Micro- and macroscopical remains with characteristic morphology are sometimes identified inside the pottery or in the residues adhering to the walls: pollen grains, fragments of plants, fish scales, etc. (e.g. Duplaix-Rata, 1995; Kubiak-Martens et al., 2015; Raemaekers et al., 2013). The analysis of liquid or semi-liquid products absorbed into the walls during use by analytical chemistry methods also provides significant information by identifying a large range of commodities: animal carcass fats, dairy products, beeswax, resins, etc. (e.g. Craig et al., 2013; Cubas et al., 2020; Evershed et al., 2008b; Roffet-Salque et al., 2015). However, it remains difficult to evaluate how these contents were used or processed in archaeological pottery, except when the vessels are of a very specific shape (e.g. Heron et al., 2013; Salque et al., 2013), when use-wear (abrasion, soot deposits or charred residues) are visible on the walls (e.g. Fanti et al., 2018; Forte et al., 2018; Vieugué et al., 2016), or when molecular transformation markers are detected (Evershed et al., 2008a; Raven et al., 1997). Unfortunately, in many cases, this information has been lost (high degree of pottery fragmentation preventing shape reconstitution, poor preservation of pottery surfaces preventing use-wear study, absence of transformation markers, etc.).

At an early stage in the implementation of organic residue analysis, differences in lipid accumulation (i) between pottery types and (ii) between base, body and rim of a single vessel were detected; these patterns were suggested to be caused by the modalities of use of the vessel (Charters et al., 1993). In particular, on an assemblage of 62 archaeological reconstructed vessels from late Saxon and medieval West Cotton settlements (Northamptonshire, UK), Stephanie Charters and colleagues compared (i) the mean lipid concentration in the different groups of ceramic vessels, (ii) the range of lipid concentration inside these groups, and (iii) the lipid distribution between base, body and rim in each vessel. This analysis which has since been revisited and completed (Dunne et al., 2020a), led the authors to propose for the first time interesting relationships between shape and function (Fig. 1). Jars with a very variable amount of lipid from one vessel to another, but large mean amounts, and a specific accumulation of lipids at the rim, were considered as cooking pots. The moderate to high lipid concentration at the base of the 'Top hat' vessels has been interpreted as the results of waterproofing with a fatty product or use for roasting. On the contrary, jugs and bowls presenting low amount of lipids (except for the few spouted bowls) were difficult to interpret.



**Fig. 1** Different lipid absorption patterns in jars, 'top hat' pots and bowls from the West Cotton site (after Charters et al., 1993). a) Maximum, minimum (rectangle) and average (black bar) extraction yields on each type of vessel; b) vessel shapes; c) example of lipid distribution along the vertical profile; d) proposed interpretations in terms of use

Many parameters related to the use of pottery may affect the amounts of lipids absorbed and their preferential absorption in certain areas of the vessel: (i) the characteristics of the product contained in the pot, including its chemical composition (and properties), its solid or liquid state and the presence of water, (ii) how the vessel is used, including its exposure to fire, its filling level, its frequency of use, its lifespan, etc., and (iii) the characteristics of the clay material, including porosity. Experimental analyses were carried out to explore these mechanisms and to interpret lipid ranges and profiles in terms of modalities of use (Charters et al., 1997; Evershed, 2008a). Investigations with replica vessels allowed estimation of the 'reasonable value for the lipid capacity of a potsherd' (c. 10 mg g<sup>-1</sup>) and highlighted the variations in lipid concentration in potsherds depending on the nature of the commodities prepared in the vessels: boiling lamb meat provided a concentration 150 times higher than boiling *Brassica* leaves (Evershed, 2008a, p. 30). Boiling of products rich in fats or waxes proved to produce a decreasing profile, from the highly concentrated rim of the vessel to the lipid-depleted base (Charters et al., 1997). This specific distribution, similar to a pattern sometimes identified in archaeological vessels (Charters et al., 1993, 1995; Fanti et al., 2018; Šoberl et al., 2014), is explained by the accumulation of lipids (hydrophobic and less dense than water) at the filling limit and their significant degradation at the base of the vessel, which is in direct contact with the heat source. Roasting experiments have produced a very similar profile, although the samples from the body and the base of the pot appeared more concentrated than for boiling (Evershed, 2008a). In this case, the accumulation of lipids at the rim of the pot was supposed to originate from the combined effects of capillary phenomena and splashing of heated fats (Evershed, 2008a).

These results from the 1990's / 2000's have then been little exploited for the interpretation of archaeological assemblages, despite their significant informative potential in terms of modality of use. Focusing on the analysis of potsherds, far more common in archaeology than full vessel profiles, lipid analysis has mainly focused on the identification of the commodities contained in the pots (e.g. Copley et al., 2005a; Craig et al., 2013; Cubas et al., 2020; Dudd & Evershed, 1998; Dunne et al., 2016; Evershed et al., 2008b; Mukherjee et al., 2007; Roffet-Salque et al., 2015). It is only in recent years that work has focused on understanding pottery function by combining extraction yields, structural molecular information, isotopic data and/ or morphological criteria (e.g. Brychova et al., 2021; Drieu et al., 2020a, 2021; Dunne et al., 2020a; Fanti et al., 2018; Heron et al., 2015; Matlova et al., 2017; Šoberl et al., 2014; Stojanovski et al., 2020). Various absorption patterns along the vertical profile have been identified in some of these studies (e.g. Šoberl et al., 2014), showing that the experimental models

available (boiling, roasting) do not constitute a comprehensive reference framework for interpreting the use of ancient pottery, which may have been used for a wider range of functions (steaming, simmering, toasting, fermenting, storing, burning, tanning, making soap, washing, etc.). In addition, other parameters than lipid concentration are likely to vary between the base, body and rim of a vessel, but have never been studied: triglyceride hydrolysis and fatty acid oxidation, for example, are known to occur during the use of pottery vessels (e.g. Copley et al., 2005b; Evershed, 2008a). Tracking where these mechanisms were most likely to occur within the vessel could provide valuable information on how the vessels were used, in terms of exposure to heat, water, oxygen or micro-organisms. However, in the absence of a robust framework, it was not reasonable to carry out multiple destructive sampling of the rare full vessel profiles preserved in archaeological context. As a result, while organic residue analyses have provided valuable information regarding the exploitation of natural resources in the past, the way these products were processed and consumed still needs complementary investigations.

Ethnoarchaeological approaches have demonstrated a strong potential to look into the relationships between pottery shape and use (e.g. Burry, 2003; De Ceuninck, 1994; Gallay, 2012; Grillo, 2014; Henrickson and McDonald, 1983; Mayor, 1994; Smith, 1985) or pottery use-wear and function (e.g. Arthur, 2002, 2003; Reid and Young, 2000; Skibo, 1992, 2013). However, few ethnographic pots have been subjected to chemical analysis, while this approach would be particularly well suited for creating models describing how different uses impact the chemical signature that is absorbed into the walls. This is probably due to the multiple difficulties of accessing ceramics still in use today. Indeed, few populations still make and use ceramic vessels in a traditional way (Huysecom et al. 2016). In addition, various taboos sometimes prevent the documentation and collection of vessels in use (Dunne et al. 2019).

In the early 1990s, James Skibo (1992; 2013) carried out the chemical analysis of organic residues from 11 ethnographic ceramic vessels and 10 archaeological potsherds related to Kalinga people (Philippines, Asia) in order to assess the informational potential and understand the degradation mechanisms of food crusts. Of the 11 ethnographic pots selected for analysis, 6 were used for boiling rice and 5 were used for boiling meat/vegetables on a daily basis. All of the 10 selected archaeological potsherds were probably part of cooking pots whose contents remained unknown. The samples, taken primarily from the base of pottery, were analysed using GC-MS. This ethno-archaeological study, which was pioneering in the field of pottery use, highlighted the presence of fatty acids in all the ethnographic pots and archaeological sherds analysed, showing that the lipids survived to anthropic (repeated exposure to fire) and natural (long-term burial) degradation processes. It also showed that the ratio of the three best-preserved fatty acids (palmitic  $C_{16:0}$ ; stearic  $C_{18:0}$  and oleic  $C_{18:1}$ ) allowed the distinction between rice and meat/vegetable cooking pots, even though this distinction raised some issues on the archaeological remains due to the differential alteration of the fatty acids.

During the 2000-2010s, only few chemical analyses of organic residues were carried out on ethnographic pottery of which the function was known. In order to evaluate the impact of long-term use of ceramic vessels on lipid degradation, Richard Evershed (2008a) analysed one unglazed Tsoukali vessel from Metataxades (Greece, Europe) which was used for cooking pork stew once a year for forty years (Evershed, 2008a). Several samples were taken alongside the profile of the pot and conventionally analysed by GC-MS. This study clearly showed that the repeated use of a ceramic vessel over fire had an impact on the lipid composition because the triacylglycerols had been degraded to di-, monoacylglycerols and free acids (Evershed, 2008a). Following this, Sharon Fraser et al. (2012) analysed by GC-MS and GC-C-IRMS one ethnographic pottery vessel and four archaeological potsherds related to the Talensi group (Northern Ghana, Africa). The authors inferred that both the archaeological vessels and the ethnographic pottery contained plant products, an hypothesis that has to be

considered cautiously as not all molecular peaks were examined and interpreted (in particular the large amounts of C<sub>16:1</sub>).

Very recently, Julie Dunne et al. (2019) have surface collected and analysed by GC-MS and GC-C-IRMS a series of 63 potsherds discarded on the landscape in Kenya, Africa. The samples are related to Samburu pastoralist communities, whose subsistence system is ethnographically well known. The study compares the products identified in the 48 % of samples that provided significant lipid yield and the data from ethnographic research on food traditions. By showing that the interpretations resulting from the chemical analysis of the lipids absorbed in the potsherds are not always in line with the food practices of the populations considered, as other vessels and tools than pottery may have been used for the preparation and consumption of food, this paper emphasises the value of developing approaches on modern samples related to ethnographic data.

Among the few lipid studies of modern pottery related to ethnographic information reported in the literature (Dudd, 1999; Dunne et al., 2019; Evershed, 2008a; Fraser et al., 2012; Skibo, 1992, 2013), one has investigated the variability of lipid concentration between the base, body and rim of the pots (Evershed, 2008a). This study, based on the analysis of one ceramic vessel showed a decreasing profile from top to bottom (Evershed, 2008a), similar to the boiling pattern mentioned above.

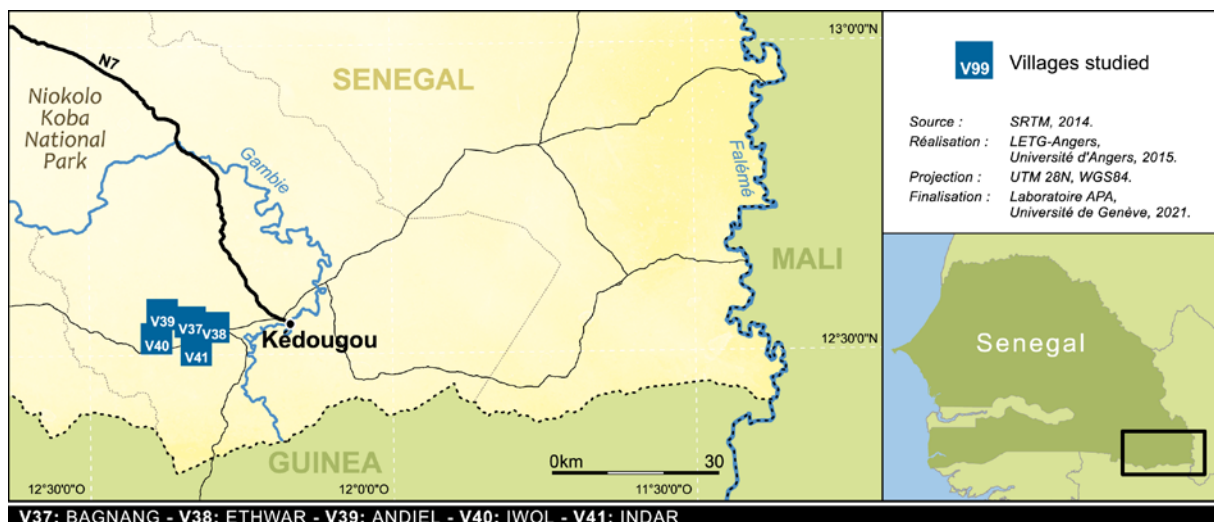
In order to explore further the relationships between lipid accumulation within the different parts of pottery walls and function of the vessels, we needed to analyse the organic content of modern pots for which the commodities processed and the modalities of use were known. One of the challenges, at a time when ceramic traditions are being abandoned (e.g. in our field work of the Falémé Valley; Huysecom et al., 2016), was to find a context where these traditions are still alive. A previous work of prospection led us to focus our first investigations on Bedik villages in the South-East of Senegal where potters are still active and pottery largely used on a daily basis for a wide range of purposes with various modes of use (Mayor and Vieugué in Huysecom et al., 2017, pp. 179–190). Field work allowed the documentation of 87 pots with various functions. As an exploratory study, 9 pots of different functions were chosen among them for blind test chemical analysis.

We present here the sampling methodology and the results of structural and quantitative chemical analysis on these ceramic vessels. Although this study focuses mainly on how the pots were used and not on the identification of their contents, the results will first briefly describe the extraction yields and molecular profiles, which may provide some information on the modalities of use (total concentration, anthropogenic transformation markers, etc.). The modalities of use will be further investigated based on lipid yield and vertical profiles of lipid concentration. The degradation of triacylglycerols and C<sub>18</sub> unsaturated fatty acids, that might indicate where hydrolysis and oxidation are favoured, will be tested as new proxies to study the use of the pots. Chemical interpretations will then be compared with the recorded actual uses of these vessels to assess the variety of profiles resulting from the different vessel functions and to provide insight into the mechanisms that led to the observed patterns.

## Materials and methods

### Study area and ceramic assemblage

The Bedik Country, located in eastern Senegal west of Kédougou, between the Guinean border and the Niokolo-Koba National Park (Fig. 2), belongs to the area called "cultural landscapes of the Bassari Country", classified as a UNESCO World Heritage Site since 2012 (UNESCO, 2012). This region brings together several distinct cultural groups, all anchored around mountainous massifs belonging to the first foothills of the Fouta Djallon, including the Bedik, settled on the dolerite hills and in the plains around Bandafassi, the Bassari, grouped around Salémata, and the Fulani, around Dindéfello. The Bedik people occupy a circular area of about 40 km in diameter, but their territory was once much larger (Ferry, 1967, 1985). They have their own language (*menik*) and are mainly catholic, while Islam is practiced in the lowland villages, but they have kept numerous aspects of their ancestral culture and religion, among them beer making.

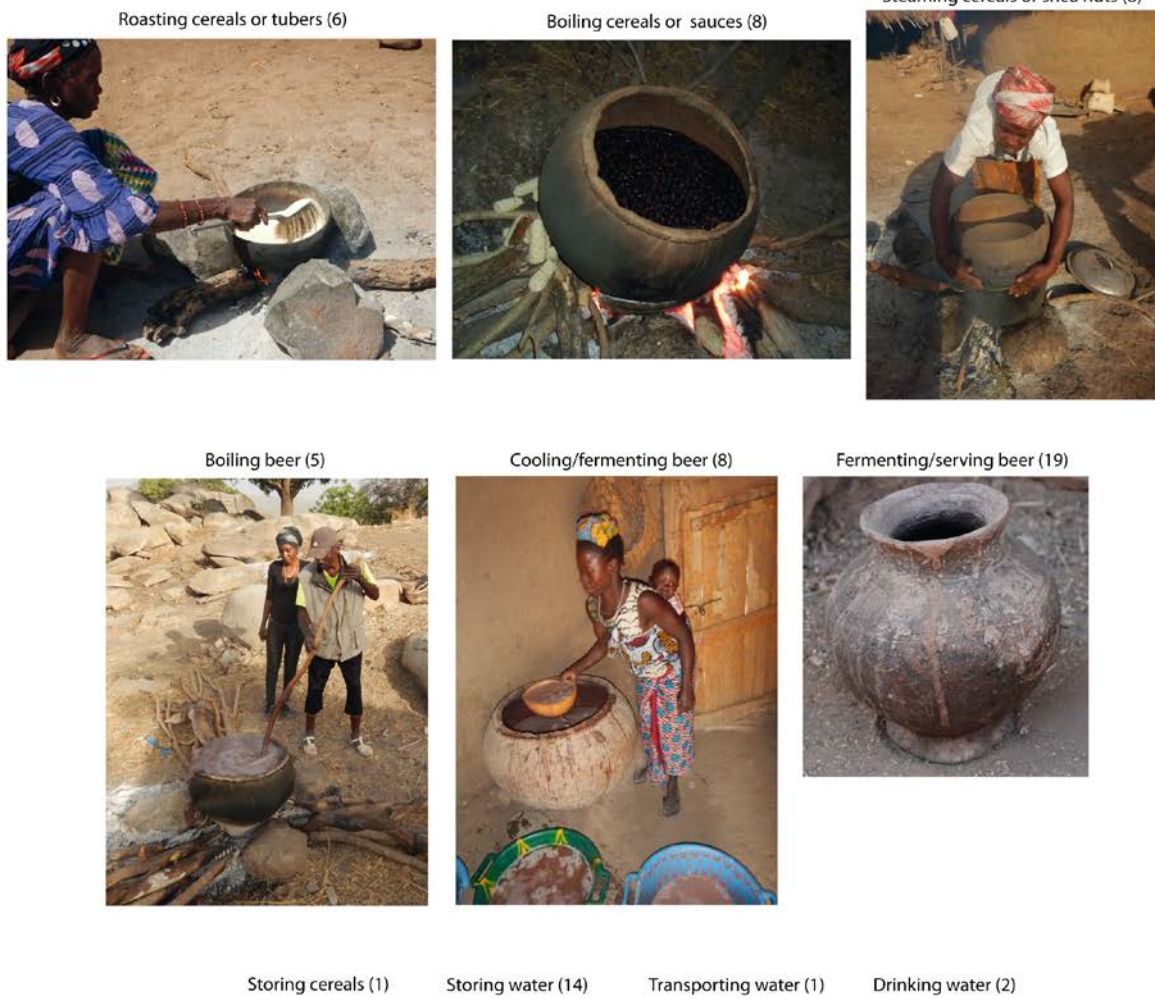


**Fig. 2** Location of Bedik Country and the villages where the studied vessels have been collected (Map Anne Mayor and David Glauser)

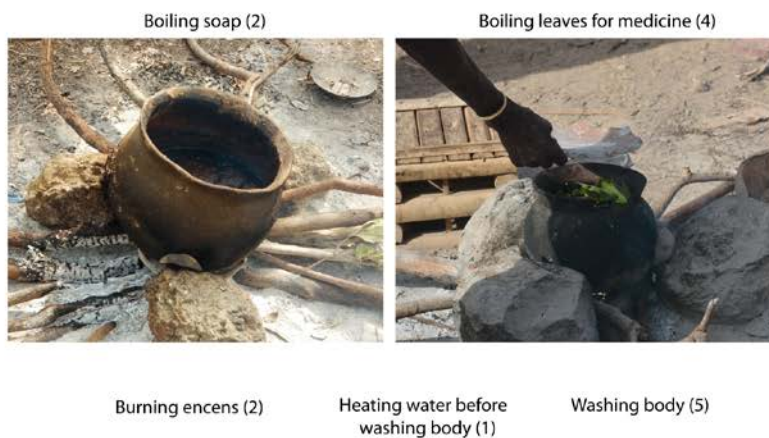
Following documentation of the manufacturing processes of pottery (from fashioning to firing) and sampling of the raw materials involved in the paste preparation and surface treatment, ethnographic surveys focused on pottery use were conducted in seven Bedik villages: Ethwar, Bagnang, Indar, Andiel, Iwol, Ethiès-haut and Ethiès-bas (Mayor and Vieugué in Huysecom et al., 2017, pp. 179-190; Fig. 2). Because of the hundreds of used pottery present in these villages, it was impossible to study all of them. A selection of ethnographic pots to be documented had to be made. In order to have a representative sample of the functional range of pottery which are still in use among the Bedik communities, we decided to analyse a significant set of ceramic vessels involved in the various food, medicinal and handicraft activities. A total of 87 pottery vessels of 16 different uses were thus documented (Fig. 3). The number of pots studied per function varies depending on their frequency in the Bedik villages.



## FOOD ACTIVITIES (72 pots)



## HYGIENE/HEALTH/WELL-BEING (14)



## HANDICRAFT (1)

Boiling dye (1)

*Fig. 3 The different functions of pottery identified among the Bedik communities. The number of ceramic vessels documented per function is indicated in brackets*



In order to document accurately the function of Bedik pottery, we set up a comprehensive study method which takes into account both typometry and use-wear of ceramic vessels. After recording, drawing and taking photographs of the 87 selected Bedik vessels, interviews were conducted with the users (Online Resource 1). The questions mainly focused on:

(1) *The functioning of the pots.* What is your vessel used for? Is it used for storing, transporting, boiling, frying, roasting, steaming, serving, consuming or for another purpose? Did its use change over time?

(2) *The content of the ceramic vessels.* What substances do you usually put inside your pot? In what form(s)? What was the last content? What were past contents if different?

(3) *The lifespan of the pots.* How old is your pot? Do you still use it? If that is not the case, when did you abandon it?

(4) *The use frequency of the ceramic vessels.* How often do you use this pot on average? Is it every day, once a week, once a month or occasionally /seasonally?

This thorough fieldwork made it possible to gather accurate information regarding the function of the 87 Bedik pottery selected for study.

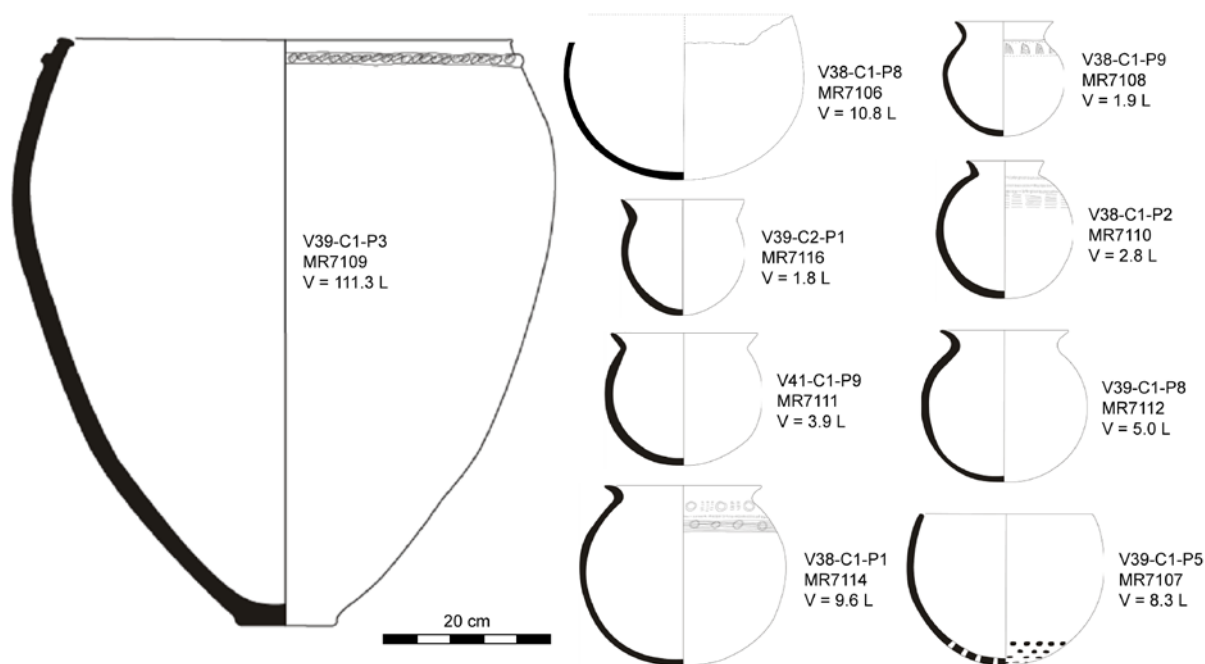
### Sampling of the pots

Because most of the 87 documented pots were still in use in the Bedik families, it was not possible to purchase all these pots for performing residue analyses in laboratory, as it would have prevented them to achieve their usual domestic tasks. A selection had therefore to be made. In order to ensure that the Bedik people did not lack the vessels likely to be collected, we focused on the best represented functional classes of ceramic vessels and selected an emblematic pottery for each of them. Nevertheless, ceramic vessels that only contained water (such as pottery used for storing water or washing) were not sampled, as they would obviously not yield any lipids. Of the 87 Bedik ceramic vessels initially documented, 9 were purchased and brought back to Europe for residue analysis (i.e. 10% of the corpus; Table 1 and Fig. 4).

These pots differ mainly in their volume (from 1.8 to 111.3 L) and the size of their orifice. In addition, while most vessels have a round, solid bottom, two (MR7107 and MR7109) differ by their multi-perforated and pointed bottoms respectively. All of them have everted rims, except two (MR7109 and MR7106) showing straight rims (Fig. 4).

Fieldwork number	Chemistry lab number	Village
VC38-C1-P8	MR7106	Ethwar
VC39-C1-P5	MR7107	Andiel
VC38-C1-P9	MR7108	Ethwar
VC39-C1-P3	MR7109	Andiel
VC38-C1-P22	MR7110	Ethwar
V41-C1-P9	MR7111	Indar
VC39-C1-P8	MR7112	Andiel
VC38-C1-P1	MR7114	Ethwar
VC39-C2-P1	MR7116	Andiel

*Table 1 Vessels considered in this study.*



**Fig 4** Pottery vessels analysed in this study. The scale is the same for all ceramics and their volume is indicated in the figure.  
© Drawings Anne Mayor and Julien Vieugué

In the laboratory, the entire vessels were cut in four parts with a grinder, a quarter being dedicated to chemical analysis (Fig. 5). Potsherds (c. 2 cm x 2 cm) were sampled along the vertical profile of all the vessels. For some pots, horizontal transects were similarly sampled (Fig. 5). When the whole vessel was not available (vessels broken during transport – MR7111 and MR7114), the sampling was based on the evaluation of the position of the fragments along the profile, in order to document the entire height. A total of 59 samples were analysed.



**Fig 5** Sampling strategy on the whole vessels. Example of pot MR7107. © Photos Arnaud Mazuy, CNRS

### Sample extraction and analysis

In order not to bias the analysis protocols, the study of organic residues trapped into the porous wall of the Bedik pottery was conducted as a blind test. The surface of each potsherd was scraped with a clean scalpel to remove any potential contamination from handling. The samples (approx. 2 g) were then crushed with mortar and pestle. A volume of 20  $\mu\text{L}$  of internal standard (*n*-C<sub>34</sub>, 1 g L<sup>-1</sup>) was added to the powder, which was then extracted in a mixture of dichloromethane/methanol (2:1, v/v) in an ultrasonic bath (2 x 15 min). The samples were centrifuged and the supernatant was evaporated under a gentle stream of nitrogen. The extracts were derivatised with BSTFA (*N,O*-bis(trimethylsilyl)trifluoroacetamide with 1% trimethylchlorosilane) for 1 h at 70°C, then dissolved in

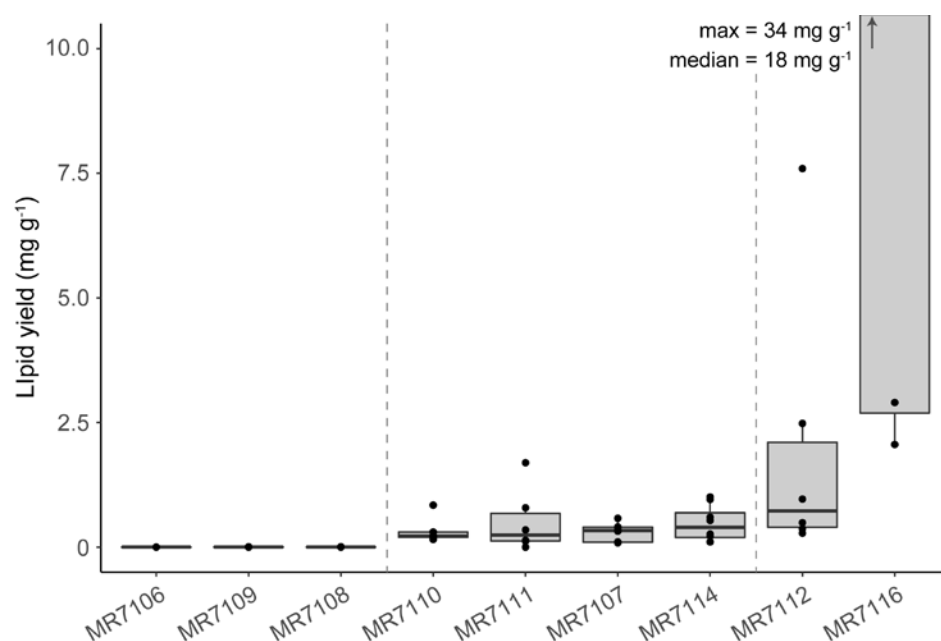
cyclohexane for injection in Gas Chromatography (GC) and Gas Chromatography-Mass Spectrometry (GC-MS).

The chromatograph was an Agilent Technologies 7890A unit, equipped with a fused capillary non-polar column (DB5-HT, 15 m x 0.32 mm x 0.1  $\mu\text{m}$  film thickness, Agilent J&W), and 1  $\mu\text{L}$  of sample was injected via an on-column injector. The carrier gas was helium and the temperature was programmed as follows: a first ramp from 50°C to 100°C at 15°C  $\text{min}^{-1}$ , followed by a second ramp to 375°C at 10°C  $\text{min}^{-1}$  and 10 minutes of isothermal hold. For GC-MS analysis, samples were injected into a Shimadzu GC 2010 PLUS chromatograph coupled with a Shimadzu QP 2010 ULTRA mass spectrometer. The injection of 1  $\mu\text{L}$  of extract was done in a DB5-HT column (15 m x 0.32 mm i.d., 0.1  $\mu\text{m}$  film thickness, Agilent J&W), via a splitless injector. The temperature program was as follow: hold at 50°C for 1 min, increase to 150°C at 20°C  $\text{min}^{-1}$ , increase to 320°C at 10°C  $\text{min}^{-1}$ , and increase to 380°C at 15°C  $\text{min}^{-1}$ , and a final isothermal hold for 10 min. The temperature of the interface was maintained at 340°C. The source operated by electron ionization (EI mode, 70 eV), and the mass spectra were acquired between 50 and 950  $m/z$ .

## Results

### 1. Lipid concentrations

The amounts of lipids are very variable between vessels (0 to 33.6  $\text{mg g}^{-1}$ ; Fig. 6). This suggests either that they contained products with a variable proportion of lipids, that they had various functions, which differently favoured the absorption of lipids or they had different use frequencies.



**Fig 6** Box plot of the lipid concentrations in each vessel analysed. The median and the 1st and 3rd quartile are represented by the grey rectangles. The dots show all measured values. For clarity of the figure, the y-axis scale excludes two dots of MR7116. Dotted lines indicate the three groups of vessels, based on extraction yields, which are discussed in the text

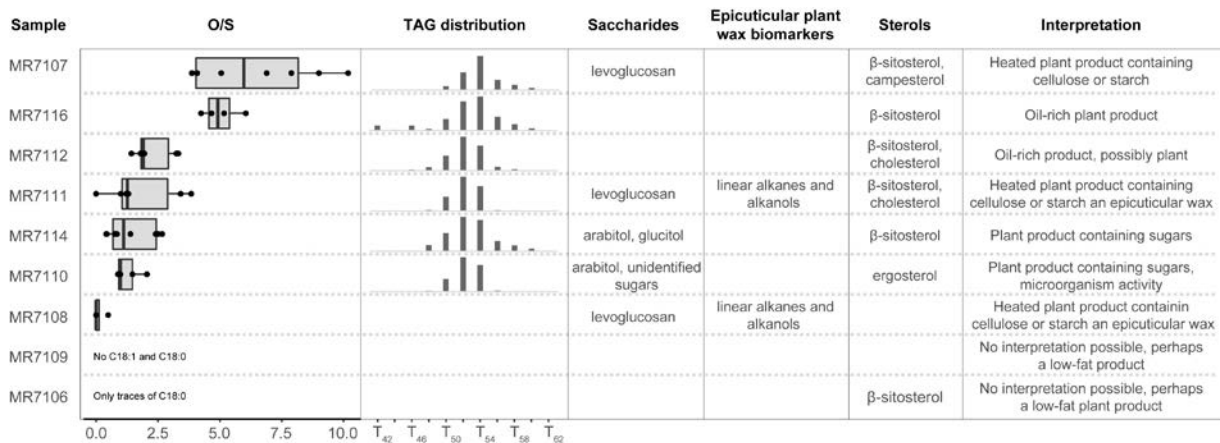
Three vessels yielded very low amounts of lipids ( $< 9 \mu\text{g g}^{-1}$ ; MR7106, MR7108 and MR7109; Fig. 6). The almost absence of lipids makes it difficult to interpret the function of these vessels based on chemical data, but it can be proposed that they were used for low-fat products. An alternative hypothesis would be that their mode of use did not favour the absorption of lipids (e.g. storage of dry/solid contents), or resulted in a significant degradation of lipids (e.g. very intense heating).

MR7107, MR7110, MR7111 and MR7114 yielded medium ranges of lipids (between 50 and 1700  $\mu\text{g g}^{-1}$ ), while the lipid yield was globally much higher in MR7112 and MR7116 (between 200 and 33573  $\mu\text{g g}^{-1}$ ; Fig. 6). Lipid-rich commodities, such as animal products or oleaginous plants are probably responsible for the high lipid yields in the latter two samples. The maximum amount of 33.6  $\text{mg g}^{-1}$  detected in the upper part of MR7116 is higher than the maximum values obtained in experiments of lamb cooking or olive oil soaking (21.8 and 13.5  $\text{mg g}^{-1}$  respectively; Evershed, 2008a), by conventional solvent extraction. It is half the maximum concentration obtained in potsherds from surface collection in Kenya (64.9  $\mu\text{g g}^{-1}$ ; Dunne et al., 2019) after acidified methanol extraction, a treatment known to be significantly more efficient than the conventional one (Correa-Ascencio and Evershed, 2014). On the contrary, low-fat plants, such as leafy vegetables or cereals may account for the low amounts absorbed in MR7107, MR7110, MR7111 and MR7114, of the same order of magnitude (median between 224-399  $\mu\text{g g}^{-1}$ ; Fig. 6) as those obtained by a few cabbage, nettle or einkorn cooking episodes (respectively 262, 239 and 388  $\mu\text{g g}^{-1}$ ; Charters et al., 1997; Debono Spiteri, 2012; Evershed, 2008a). However, it cannot be ruled out that uses reducing the absorption of fats (e.g. low liquid content) or leading to their degradation (e.g. intense heating) may be responsible for the lower extraction yields.

## 2. Initial molecular evidence of the content

Many of the foodstuffs used in Bedik pottery and recorded during fieldwork, in particular plant products, are not generally investigated in archaeological ceramics using organic residue analysis, and the chemical composition of some of them is not even documented in the chemistry or biochemistry literature. In order to be able to accurately identify the commodities in the vessels with chemical methods, it will be necessary to study the molecular composition of a wide range of authentic modern products from West Africa, which will be the purpose of future research. However, preliminary and general interpretations can already be drawn about the natural origin of the contents (Fig. 7, Online Resource 2). Triacylglycerols (TAGs) were detected in all six vessels with more than 9  $\mu\text{g g}^{-1}$  of lipids and in small amounts in one sample from MR7109. All samples yielded longer TAGs ( $\text{T}_{56}\text{-T}_{62}$ ; Fig. 7, Online Resource 2) than the animal fats presented in the literature in organic residue analysis. This large number of carbon atoms indicates that TAGs are partly composed of long-chain fatty acids ( $> \text{C}_{20:0}$ ), which are characteristic of plant products (Dunne et al., 2016). The ratio of unsaturated fatty acids with 18 carbon atoms to their saturated homologue (O/S) points to plant oil in MR7107 and MR7116 (O/S  $> 3.8$ ; Fig. 7, Online Resource 2). The detection of phytosterols ( $\beta$ -sitosterol and campesterol) in six vessels supports the plant hypothesis (Fig. 7, Online Resource 2). Cholesterol could indicate the presence of animal fats, but it also exists in some plants (Christie, 1989, p.12). Ergosterol in MR7110 (Online Resource 2 and S3) indicates fungal activity, linked to a specific use (Isaksson et al., 2010) or the start of natural degradation of the contents (Dudd et al., 1998; Whelton et al., 2021), for example during transportation of the vessels to the analytical laboratory. In general, it should be noted that caution is required in the interpretation of sterols as they may derive from contaminations (Whelton et al., 2021). Linear alkanes ( $\text{C}_{27}\text{-C}_{33}$ ) and alkanols ( $\text{C}_{26}\text{OH-C}_{34}\text{OH}$ ) in MR7108 and MR7111 (Fig. 7, Online Resource 2) suggest the presence of epicuticular plant waxes (Dunne et al., 2016). MR7107, MR7108, and MR7111 probably contained cellulose or starch as levoglucosan, resulting from the thermal treatment of these compounds (Heron et al., 2016; Shoda et al., 2018; Styring et al., 2013), was detected (Online Resource 2 and S3). The presence of sugars, characterised by the  $m/z$  217 ion (arabitol, glucitol, and unidentified saccharide compounds; Online Resource 2 and S3) in MR7110 and MR7114 also suggests the presence of a plant product. Finally, methyl esters of fatty acids ( $\text{C}_{16:0}$ ,  $\text{C}_{18:1}$  and  $\text{C}_{18:0}$ ), in addition to the trimethylsilyl derivatives, were detected in two samples of MR7116. The formation of these compounds, already observed in archaeological pots (Drieu et al., 2018), remains difficult to explain as it requires exposure to very high temperatures (800°C; Raven et al., 1997). Sets

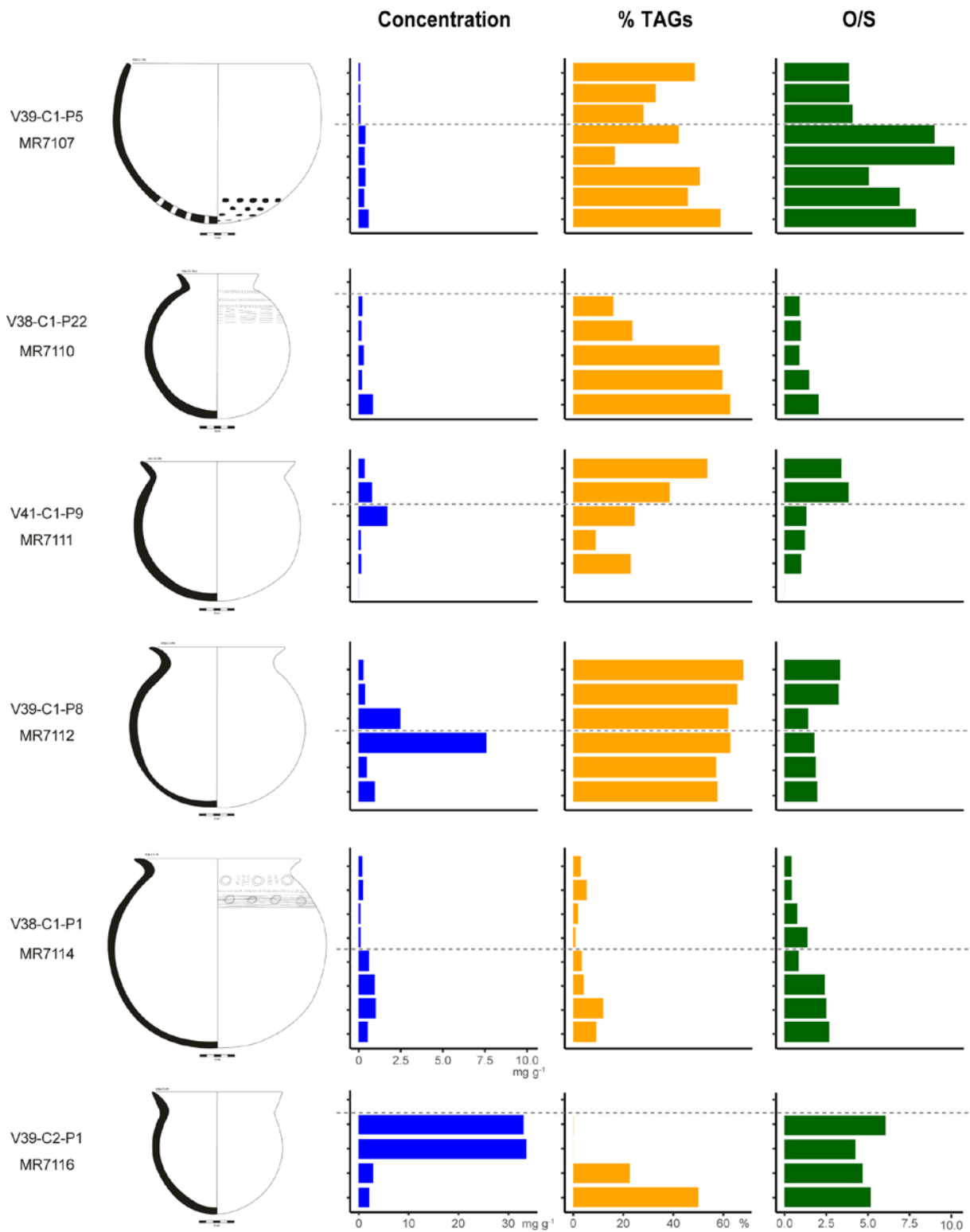
of biomarkers are never exactly the same from one vessel to another, suggesting that all their contents are different.



**Fig. 7** Information obtained from molecular profiles and providing insight into the natural origin of the products contained in the vessels

### 3. Vertical profiles of lipid concentration

The vertical concentration profiles of the vessels that have yielded enough lipids are very variable (Fig. 8), suggesting a diversity of function, despite the general similarity of most vessel shapes. MR7116 has a concentration profile similar to the experimental boiling pattern published in the literature (Charters et al., 1993, 1997; Evershed, 2008a). The high lipid concentration at the top of the vessel, with a very sharp decreasing profile towards its base, suggests that this vessel was used for heating fatty substances in an aqueous medium. For MR7111 and MR7112, similar use with a filling level at mid-body, could explain the larger amount of lipids detected there, compared to the neck and base. Lipids absorbed above the filling level could be due to spattering or capillary effects (Evershed, 2008a). The relative homogeneity of the lipid distribution along the walls of MR7107 and MR7110, on the contrary, suggests a content without phase distinction (i.e. a thick or doughy content), and which has been little or no heating, or a sealing of the surface with a fatty product (Charters et al., 1993). A more complex profile, but overall increasing from top to bottom, is identified in MR7114, suggesting again a thick or doughy content and no or only moderate heating, with mid-body filling. In MR7107 and MR7114, the clear shift in lipid concentration at mid-body suggests that this is the most usual filling limit. Again, the detection of lipids above this limit could be due to capillary action. On the contrary, no clear breaks appear in the concentration of MR7110, suggesting that it was completely filled in.

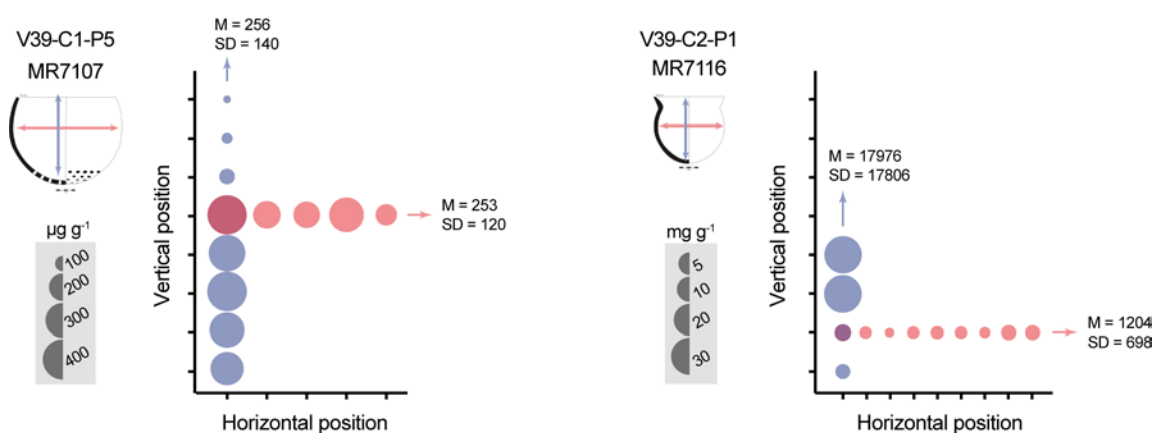


**Fig 8** Lipid distributions identified along the vertical profile of vessels that yielded more than  $10 \mu\text{g g}^{-1}$  of lipids. The grey line indicates the proposed main filling limit for each vessel. TAG: triacylglycerols. O/S: ratio between unsaturated acids with 18 carbon atoms and stearic acid



#### 4. Horizontal variability of lipid concentration

The study of lipid distribution along a horizontal transect in MR7107 and MR7116 vessels also shows variations in extraction yields (Fig. 9). Such horizontal variability had already been observed on experimental pots (mean =  $262 \mu\text{g g}^{-1}$ , SD =  $128 \mu\text{g g}^{-1}$ ; Charters et al., 1997), in a range similar to that of MR7107 (mean =  $253 \mu\text{g g}^{-1}$ , SD =  $120 \mu\text{g g}^{-1}$ ). Unlike vertical variability, horizontal variability appears to be random rather than following an absorption pattern (Fig. 9). It is probably due to local differences in parameters such as porosity, mineralogy, surface temperature, etc., but not to how the vessel was used. This horizontal variability is likely to have no effect on the vertical patterns where the concentration shows sharp increases or decreases. As an example, for MR7116, the maximum recorded horizontally ( $2901 \mu\text{g g}^{-1}$ ) is lower than the value obtained in the upper band ( $33573 \mu\text{g g}^{-1}$ ). However, it may have an impact where the variations along the vertical transect are small. Sampling over the entire surface would be necessary to verify, with Charters et al. (1997), that the vertical profiles remain globally the same regardless of the location of the sampling.



**Fig 9** Comparison of the latitudinal and longitudinal distribution of lipids in MR7107 and MR7116 vessels. The size of the bubbles is proportional to the extraction yield. M stand for the mean values ( $\mu\text{g g}^{-1}$ ) and SD for the standard deviations ( $\mu\text{g g}^{-1}$ ) along the vertical and horizontal axis

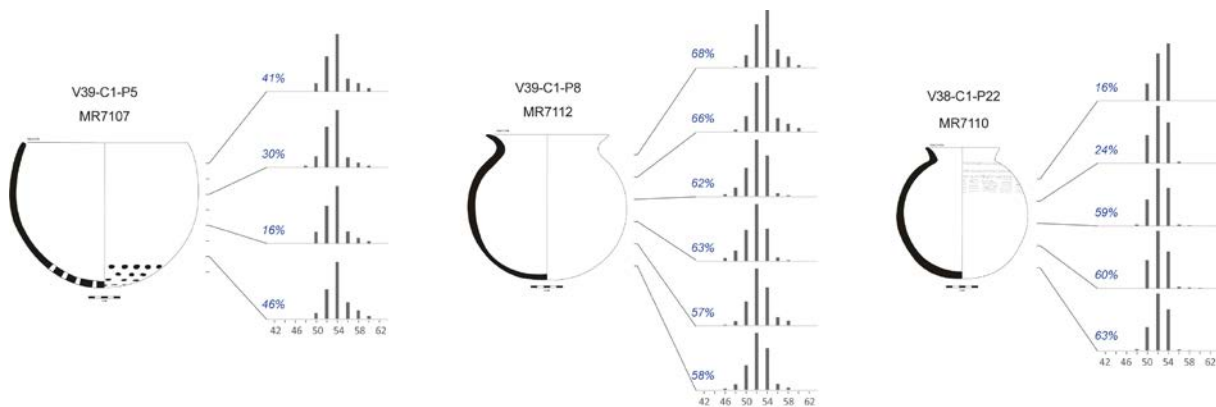
#### 5. Vertical profiles of triacylglycerol preservation and composition

TAGs, the main components of fats and oils, are hydrolysed into their by-products (di- and monoacylglycerols, fatty acids and glycerol) by various mechanisms (Dudd et al., 1998; Evershed et al., 1995, 1991). The presence of water, high or low pH, heating and microorganisms are key factors in this processes. In order to explore preferential hydrolysis patterning, the percentage of TAGs (%TAGs) was compared along the vertical transects of the vessels.

MR7110 and MR7116 display increasing profiles of %TAGs from the top to the base of the vessel, suggesting that TAGs are preferentially hydrolysed at the surface of the contents. For MR7116, since boiling has been proposed as a mode of use, it can be assumed that the interface between water and lipids created by bubbling is a preferred medium for the hydrolysis of TAGs. In the case of MR7110, the lipid concentration profile suggested little or no heating. Since a difference in pH across the contents of the vessel is also unlikely, it can be assumed that the preferential hydrolysis of TAGs at the top of the pot is due to microorganism activity, which requires a supply of oxygen. The accumulation of microorganisms at the surface is, for example, evident in the growth of a yeast-“veil” during the development of certain wines (Cordero-Bueso et al., 2018). The detection of ergosterol in this vessel could strengthen this interpretation by indicating yeast or fungi activity (Dudd et al., 1998; Isaksson et al., 2010), but it is impossible to determine whether this microbial activity is anthropogenic or the

result of natural degradation (e.g. occurring during transport to Europe). In MR7111 and MR7112, it can be assumed that heating (suggested from the lipid concentration profiles) is responsible for the preferential degradation of TAGs at the base and the decreasing %TAGs from top to bottom. The high %TAGs in MR7112 could be explained by gentle heating, while the sharper decrease of %TAGs in MR7111 could be related to more intense fire exposure (higher heat or more frequent use). The better preservation of TAGs above what we assume to be the filling limit in these vessels could be attributed to the relative absence of water, compared to the parts of the walls in direct contact with the content. The highly variable profile of %TAGs in MR7107 suggests that the contents were exposed very heterogeneously to heat or the action of microorganisms. In the case of a heated system, the heat source would not be located at the base, but around mid-body. This suggests that the characteristics of the pot (perforated at the base) are not related to a roasting function. In MR7114, the %TAGs, like the lipid concentration, does not follow a very clear profile. These variabilities remain difficult to interpret but may suggest changes in filling levels or modes of exposure to heat. It should be noted that the %TAGs are very low in this pot (max. 12%) compared to the others (up to 68%), suggesting a particular mode of use (e.g. repeated heating, intense exposure to water), a significant delay between the last use and the analysis, or a product containing more free fatty acids than TAGs.

TAG distributions are generally stable along the vertical transects of the vessels, with the exception of MR7110 and MR7112 (Fig. 10). In MR7112, the distribution is centred on  $T_{52}$  in the lower two-thirds of the pot, and on  $T_{54}$  in the upper third. This could be explained by a more pronounced degradation of TAGs in the upper part of the ceramic, due to mechanisms that preferentially degrade the shortest TAGs, as already observed in the literature (Dudd and Evershed, 1998). However, the %TAGs are slightly higher in the upper part of the vessel. An alternative hypothesis could be successive uses of two different products with differing filling levels. In MR7110, the TAG distribution of the rim sample (max  $T_{54}$ ) is different from the other distributions in the vessel, all centred on  $T_{52}$ . This pattern together with the low %TAGs at the rim (16%), highlight a degradation mechanism that preferentially hydrolysed the shortest TAGs at the top of the vessel.



**Fig 10** Examples of TAG profiles along the vertical wall. The bar charts show the relative concentration of each TAG, individualised according to its carbon atom number (horizontal axis). The %TAGs are shown in blue italics

## 6. Vertical profiles of C<sub>18</sub>-fatty acid ratio

Oxidation patterns, driven by biological ( $\beta$ -oxidation) or chemical mechanisms during use, were also examined along the vertical transects. By favouring the degradation of unsaturated fatty acids compared to their saturated homologues, they lead to a decrease in the O/S ratio (Den Dooren De Jong et al., 1961; Evershed et al., 1991). Such mechanisms are favoured by exposure to heat, light and the presence of certain metal ions (Aillaud, 2001, p. 145; Dudd, 1999, p. 214).



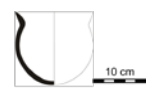
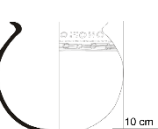
Three types of O/S profiles are identified in the studied vessels. In MR7111 and MR7112, the global decrease in O/S from top to bottom suggests that heating the base of the vessel, as proposed based on the concentration profile, also catalysed oxidation reactions. In MR7110, on the other hand, oxidation took place mainly at the top of the vessel. This observation is in agreement with the hypothesis of use for fermentation: both the activity of microorganisms at the surface and the prolonged contact of the surface lipids with oxygen may explain this profile. The O/S profile in MR7114 also suggests preferential oxidation towards the top of the vessel, but this observation does not allow us to clarify the hypotheses concerning the functioning of this pot. The complex O/S profile in MR7107 is in agreement with the hypothesis of a heterogeneity of the degradation spots by showing uneven contact with oxygen or a heat source. The O/S profile does not show any particular pattern of MR7116 and remains difficult to interpret.

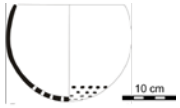

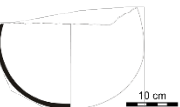
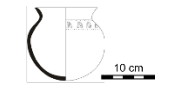
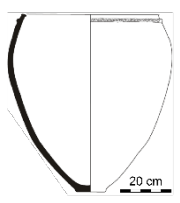
## Discussion: comparison of chemical data to actual pottery use

### 1. Actual uses observed on the field

Of the nine ethnographic pottery vessels analysed (Table 2), six were used for food activities. Three of them were used to process cereals (rice, maize, millet, fonio, sorghum) by boiling (MR7114), steaming (MR7107) and dry roasting (MR7106). Two were used for processing peanuts by roasting (MR7106) or boiling with meat and various vegetables (MR7116). Two were used for beer making, respectively for boiling and fermenting (MR7109 and MR7110). The three other vessels were used for the hygiene and medicinal purposes. One was employed for boiling oily seeds to make soap (MR7112) and the two others for boiling medicinal leaves (MR7108 and MR7111). The frequency of use varies from daily, weekly, monthly to occasionally (less than once a month) and use-life varies between one year and around 40 years, but the ceramics chosen are assumed to have been all used in a same way with the same commodities over time.

This blind test study has shown that even in the context of ethnographic pottery with a good preservation of chemical signals, it is not so easy to find their actual use and content. We categorised the vessels into three based on patterning in the molecular analyses. The interpretations we have drawn up on the basis of the chemical data are generally not in contradiction with the actual use of pottery (Table 2 and Online Resource 4).

Category	Drawing	Vessel number	Reference for chemical analysis	Lipid concentration	Assumed use based on molecular analysis	Actual use	Sphere of activity	Use frequency	Age
Ceramic vessels with medium to high fat content used for boiling		V41-C1-P9	MR7111	0-1942 $\mu\text{g g}^{-1}$	Plant products containing starch or cellulose, epicuticular waxes and medium fat content. Heating with water. Mid-body filling.	Boiling fillitoto ( <i>Piliostigma reticalum</i> ) or acodjoe leaves for medicinal purposes	Hygiene and medicinal	occasionally	3 years
		V39-C1-P8	MR7112	280-7594 $\mu\text{g g}^{-1}$	Plant products with high fat content. Heating with water. Mid-body filling.	Making soap by boiling seeds of Senegal mahogany ( <i>Khaya senegalensis</i> ). Cooking over a low heat for long periods of time (hours or even days).	Hygiene and medicinal	monthly	1 year
		V39-C2-P1	MR7116	800-33573 $\mu\text{g g}^{-1}$	Very high fat content product, possibly plant oil. Heating with water. Filling to the rim.	Boiling sauces made of peanut paste, gombo ( <i>Abelmoschus esculentus</i> ), baobab leaves, néré ( <i>Parkia biglobosa</i> ) and/or meat, and bouillon cube. Direct cooking on high heat, relatively short (30-60 minutes).	Food	daily	1 year
		V38-C1-P1	MR7114	103-1008 $\mu\text{g g}^{-1}$	Thick or doughy plant products with sugars and medium fat content. Use difficult to determine. Mid-body filling ?	Boiling rice, maize flour or pearl millet in water. Abandoned for 6 months due to cracks.	Food	weekly	3 years

Ceramic vessels with medium fat content not used for boiling		V39-C1-P5	MR7107	85-578 $\mu\text{g g}^{-1}$	Thick or doughy plant products with medium fat content and cellulose or starch. Heterogeneous exposition to heat or microorganisms. Mid-body filling.	Steaming fonio or pearl millet or maize flour over another vessel.	Food	daily	2 years
		V38-C1-P22	MR7110	155-843 $\mu\text{g g}^{-1}$	Thick or doughy product with medium fat content. Little or no heating, microorganisms activity (natural degradation or fermentation). Filling to the rim.	Fermenting and serving cereal beer.	Food	occasionally	7 years
Ceramic vessels with little or no lipids (undetermined use)		V38-C1-P8	MR7106	< 9 $\mu\text{g g}^{-1}$	Almost absence of lipids leads to three hypotheses: 1 – use of low-fat products 2 – mode of use does not favour the absorption of lipids 3 – significant degradation of lipids	Dry roasting of peanuts or fonio ( <i>Digitaria exilis</i> ).	Food	weekly	2 years
		V38-C1-P9	MR7108	< 9 $\mu\text{g g}^{-1}$	Plant products containing starch or cellulose, epicuticular waxes and very low fat content. Use difficult to determine.	Boiling leaves, including kinkiliba ( <i>Combretum micranthum</i> ), bamboo, lemon and mango leaves for medicinal purposes.	Hygiene and medicinal	daily	4 years
		V39-C1-P3	MR7109	< 9 $\mu\text{g g}^{-1}$	Almost absence of lipids leads to three hypotheses: 1 – use of low-fat products 2 – mode of use does not favour the absorption of lipids 3 – significant degradation of lipids	Boiling pearl millet / sorghum beer at high heat for several hours (used for feasts 5 or 6 times a year during 30 years), then storage of clay (10 years).	Food	occasionally	c. 40 years

**Table 2** Comparison between assumed uses based on molecular analysis and actual uses observed in the field. A more complete table with details of the structural and quantitative analysis of the containers is available in Supplementary Information (Online Resource 4)

## 2. Ceramic vessels with medium to high fat content interpreted as boiling pots

The characterisation of MR7111, MR7112 and MR7116 as 'cooking pots' meets the known use of these vessels (Table 2). Interestingly, each of these vessels, although all used for boiling, displays different vertical patterns. Further investigation is needed to explain the discrepancy in profiles between MR7111 and MR7112 on the one hand and MR7116 on the other, which may be due to differences in cooking time or temperature.

Levoglucosan and epicuticular wax biomarkers were detected in MR7111, a pot used to boil leaves. The amount of extracted lipids (up to  $1694 \mu\text{g g}^{-1}$ ) is surprisingly high for a vessel used to boil leaves, compared to what is described in experiments in the literature: a few boiling of cabbage and nettle led to extraction yields of 262 and  $239 \mu\text{g g}^{-1}$ , respectively (Charters et al., 1997; Debono Spiteri, 2012; Evershed, 2008a). To our knowledge, there is no study of the lipid composition of *filitoro* (*Piliostigma reticalum*) or *acodjoe* leaves, which should be carried out to understand these extraction yields. It should be noted that inconsistencies between the information recorded in the field and the chemical data could sometimes be related to the fact that interviewed Bedik users may have omitted to provide certain details regarding the use of their ceramic vessels. As an example, the addition of a small amount of oil or fat to a recipe may be considered anecdotal by the users but would contribute significantly to the chemical signal extracted from the ceramics.

The seeds of Senegal mahogany (*Khaya senegalensis*), processed in MR7112, are very rich in lipids (Eromosele et al., 1998). Comparison of the fatty acids concentration in the seeds shows a O/S = 3 (Okieimen and Eromosele, 1999), close to the values measured at the top of MR7112 (3.24 and 3.32). This result confirms that the oxidation mechanisms took place preferentially at the bottom of this vessel where O/S < 2. The use of this vessel to make soap, i.e. to deliberately hydrolyse an oil, could account for the difference in TAG profile along the vertical transect. The distribution identified in the body and base of the vessel probably corresponds to a TAG profile of Senegal mahogany seeds degraded by chemical hydrolysis mechanisms, generated by their heating in the presence of water and ash. The TAGs profile observed in the two samples closest to the rim could correspond to an unaltered profile of Senegal mahogany oil, due, for example, to capillarity phenomena before the beginning of the hydrolysis process. Analyses of Senegal mahogany seed TAGs would be necessary to ascertain this hypothesis. If the assumption were to be validated, it is observed that chemical hydrolysis mechanisms preferentially degrade the heavier TAGs, in contrast to enzymatic hydrolysis mechanisms, which are the most commonly considered in organic residue analysis in archaeological pottery (Dudd and Evershed, 1998).

The vessel that absorbed the most lipids (MR7116) was used for high-fat products, including peanuts and meat. It is usually assumed that the animal fat signal hides that of plants, as it is generally more concentrated in lipids (Evershed, 2008a). Here, the molecular extracts of MR7116 suggested a plant rather than an animal product (long chain TAGs and O/S > 3.8), which suggests that it was the peanuts paste, rich in fat, that masked the meat signal. This could be explained by the rare cooking of meat, compared to peanut paste. The common use of bouillon cubes, baobab leaves, *nééré* (*Parkia biglobosa*), and in particular gombo (*Abelmoschus esculentus*), rich in C<sub>18:1</sub> (Jarret et al., 2011), in this vessel may also have influenced the fatty acid ratios. The study of the lipid profile shows a distribution similar to that produced experimentally by the boiling of fatty ingredients (Charters et al., 1997; Evershed, 2008a).

Acid extractions should be carried out to confirm the absence of  $\omega$ -(*o*-alkylphenyl)alkanoic acids (APAAs), but interestingly no markers of thermal transformation of fats (mid-chain ketones and APAAs; Bondetti et al., 2021; Hansel et al., 2004; Raven et al., 1997) were detected in the studied samples.



Since some of the products processed in the pots are very rich in lipids, including unsaturated ones (e.g. oily seeds of Senegal mahogany in MR7112, and peanuts and meat in MR7116), it is surprising to note the absence of these markers. This may be due either to the absence of minerals catalysing the formation of such compounds in the clay, or to the fact that cooking episodes were not long enough or not at the right temperature to produce them. As we observe that thermal transformation markers are rarely a reliable indicator of cooking pots, we suggest that lipid concentration and TAG preservation along vertical profiles could be used as additional cooking proxies.

### 3. Ceramic vessels with medium fat content interpreted as non-cooking pots

The function of MR7110 is indeed associated with the fermentation of low-fat products (Table 2). Although the natural origin of fermented products (e.g. grapes, cereals, milk) can be identified in archaeological pottery, the fermentation process itself remains difficult to detect because the molecules it produces are the same as those generated by natural degradation (Drieu et al., 2020b; Steele, 2013; Whelton et al., 2021). The preferential degradation of lipids (both enzymatic hydrolysis of TAGs and oxidation of unsaturated fatty acids) at the top of the vessel could be used to support the hypothesis of fermentation in archaeological pots. A natural degradation after discarding the vessel would, on the contrary, be likely to occur similarly at all levels of the pot, and therefore not show a specific profile. This hypothesis will have to be verified by studying a larger number of ethnographic vessels used for fermentation.

The discussion about the use of MR7107 from the chemical data is not inaccurate, as the contents are effectively non-liquid and steaming through the perforations is likely to cause local heterogeneities in temperature and humidity (Table 2). However, the chemical analysis could not account for the exact function of the vessel. It is interesting to note that steam can serve as a medium for the absorption of lipids into ceramic walls, when only liquids were generally considered to do so (e.g. Correa-Ascencio and Evershed, 2014; Evershed, 2008b).

Moderate amounts of lipids in MR7114 (between 200 and 1000  $\mu\text{g g}^{-1}$ ) and the presence of sugars are well correlated with a cereal-based content. The very low %TAGs in this vessel is difficult to interpret but could be due to its abandonment for 6 months between the last use and the sampling. We failed to identify the boiling function of this vessel based on the vertical patterns, which proved to be an interpretative challenge (Table 2).

For these three vessels, on the basis of the vertical lipid concentration profile, we had proposed a solid or pasty content, assuming that a liquid content associating lipids and water would lead to a two-phase system and an accumulation of lipids in the top of the vessel. In two of these cases (MR7110 and MR7114), the content was actually liquid, based on water and cereals. This result demonstrates that lipid concentration profiles can only provide accurate information on the mode of use, in particular boiling, when the processed products are rich in fat.

#### 4. Ceramic vessels with little lipids whose use could not be interpreted

The solid and dry nature of peanuts and fonio and the high temperatures used for roasting in MR7106 probably account for the low extraction yields (Table 2).

We missed the boiling function of MR7108 and MR7109 because of their too low-fat content. The very small amounts of lipids extracted from MR7109 can be linked to multiple factors. Beer is a low-fat product, and is therefore likely to leave only small organic traces in the pottery walls. However, the MR7110 container, also used to contain beer, yielded much more lipids. This result could, however, be explained by the only occasional use of MR7109. Moreover, beer was cooked in this container on a high and long heat, which may have favoured the degradation of the weak organic signal. In addition, the abandonment of this initial use in the last 10 years before the collection and analysis of the vessel may account for further degradation of the organic matter.

The boiling of leaves explains both the low lipid yield and the presence of epicuticular wax (alkanes and alkanols) and levoglucosan (heated starch or cellulose) in MR7108. It is interesting to note, however, that the amounts of wax extracted (between 0 and 8  $\mu\text{g g}^{-1}$ ) are much lower than those obtained after the experimental boiling of cabbage leaves (up to 400  $\mu\text{g g}^{-1}$  at the rim; Charters et al., 1997). Since this amount of lipid was reached after only 10 cooking experiments of 20 minutes, the poor extraction yield in MR7108 is probably due to the small amount of wax on the surface of the leaves. Depending on the type of plants boiled in archaeological pots, it is therefore very likely that no molecular signal is still perceptible after degradation. This result explains why the detection of a plant epicuticular wax signal remains rare in archaeological contexts and suggests that when present (e.g. Dunne et al., 2016, 2020b; Grillo et al., 2020) it is likely to originate from particularly wax-rich plants and not from any type of leaf. Further work will now have to be undertaken on the leaves exploited by the Bedik people in order to gain new reference data and to try to find some molecular diagnostic criteria to identify them. In addition, the search for polar molecules, such as fruit acids, with appropriate extraction methods (e.g. Garnier and Valamoti, 2016), should be implemented to determine whether it is possible to improve interpretations in terms of content (molecular profile) and function (absorption profiles along the vertical transect).

## Conclusion

The study of the distribution of lipids along the vertical profiles of nine ethnographic vessels from the Bedik country in Senegal shows that lipids are likely to be absorbed into the walls of the vessels, even when they are not used for cooking/boiling. The distribution of lipids along the vertical profiles does contain information about their use, confirming what had been suggested experimentally but these new results demonstrate that investigating this proxy is much more complex than expected. In addition, we have shown that additional distribution criteria could be used to study the function of the vessels. The %TAGs profile indicates the favoured hydrolysis positions, related to microbial activity, temperature or enhanced contact with water. The O/S fatty acid ratio profile provides information on the preferred location of oxidation processes, catalysed by heat, prolonged contact with air and/or microbial activity. In combination with the lipid concentration profiles, these data may improve the interpretation of the modalities of use. Horizontal variability of lipid accumulation appears to be random and not informative.

From the comparison between the molecular signal and the actual use of the vessels, we propose preliminary interpretative patterns. In addition to the decrease of lipid concentration from the top to the bottom of the vessel and the preferential hydrolysis of the TAGs at the top of the vessel related to

the boiling of fatty products, our analyses reveal other boiling patterns, probably related to various filling levels. %TAGs and O/S profiles increasing from the top to the bottom of the container seem to be associated with fermentation. From the %TAGs and O/S profiles, it is also possible to work on the function of perforated vessels. Heterogeneous profiles appear to characterise steaming, while a filtration function, frequently referred to in the literature (Salque et al., 2013; Regert et al., 2001), is expected to produce an accumulation of lipid at the bottom of the vessel, without preferential location for hydrolysis and oxidation. These hypotheses will now have to be tested by studying a larger number of ethnographic vessels used for boiling, steaming and fermenting.

Such results, even if they were obtained on a restricted number of vessels, confirm that quantitative lipid analysis is a valuable tool to investigate pottery content and use. This study also shows the limitations of organic residue analysis. The use of MR7112 to make soap and MR7108 and MR7111 to boil medicinal leaves shows that caution is required for interpreting cooking pots, as they are not consistently used for food preparation. Ethno-archaeological data will be therefore of great importance to build a comprehensive interpretative reference framework. Molecular analysis failed to identify cereal boiling in MR7114. It can be suggested that due to their low lipid concentration, cereals do not produce such sharp and clear absorption profiles as fat-rich foods.

The observed variability also calls for caution when comparing concentrations between pots, especially when sampling is not systematically done at the same level on the vessel. Rims are often preferred for lipid extraction (Charters et al., 1993, 1997; Evershed, 2008a; <https://historicensland.org.uk/images-books/publications/organic-residue-analysis-and-archaeology/>) but we show here that lipids are not always concentrated at the rim of the pots since their accumulation is strongly related to the way of using the vessels. Although these first results need to be confirmed with the study of a larger number of vessels, we suggest that mid-body, where lipids were consistently detected, may also be an interesting alternative position for sampling, as already practiced in case of a large variety of pottery shapes (Drieu et al., 2020a). Furthermore, the variability of the TAG profiles along the vertical transect, due to the non-homogeneous degradation of the TAGs in the vessel according to its use, calls for caution in the identification of commodities based on the TAG profile. Similarly, our data show that the relative fatty acid composition varies along the vertical transect depending on the way the vessel is used. As already discussed (Whelton et al., 2021), fatty acid ratios cannot be used to formally identify a commodity, even when the sampling is carried out in the upper part of a boiling/simmering vessel, as has been suggested (Kooiman et al., 2021). The example of the MR7114 vessel is particularly telling as oxidation in this cereal boiling vessel has preferentially altered the O/S ratio in the upper part of the vessel.

The inability to identify the use and contents of three vessels through organic residue analysis also supports previous findings from ethnoarchaeological studies, which have shown that chemical analyses only shed light on part of the dietary practices, and may even result in a biased understanding (Dunne et al., 2019). Furthermore, it should be noted that these interpretative hypotheses could be drawn on ethnographic samples, i.e. on molecular extracts that have probably undergone little or no natural degradation after disposal. In some samples (MR7106, MR7108, MR7109, some potsherds of MR7107, MR7111 and MR7114), the survival of such low lipid signal in an archaeological context would be unlikely.

These results will be strengthened during the Swiss National Science Foundation (SNF) interdisciplinary programme SINERGIA “Foodways in West Africa – an integrated approach on pots, animals and plants”. In this project, we will explore these avenues by expanding the corpus considered and multiplying the contexts of study. Indeed, several questions still remain to be addressed: can the lipid accumulation profiles obtained be generalised to all vessels with similar function? Is the response of the pottery even

more diverse than what was shown with this first restricted sampling? What are the molecular and isotopic characteristics of the commodities processed in the vessels? Particularly, for plant products, is it possible to distinguish the various leaves and seeds used? Can triacylglycerols, when preserved, be used to discriminate between different plant products?

The acquisition of new data among various communities of Senegal with different food systems will be useful to address these issues, as well as the analysis of modern reference products (leaves, seeds, etc.). Research will also be developed to combine the results obtained through molecular analysis with other proxies including phytoliths and starch but also use-wear and petrographic approach (Cantin and Mayor, 2018) and data from archaeology, history and social anthropology.

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