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## **Pots, plants and animals: broad-spectrum subsistence strategies in the Early Neolithic of the Moroccan Rif region**

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**Abstract**

The transition from hunter-gathering to food-producing societies in the Mediterranean zone of north Africa was complex and variable, likely influenced by local ecological conditions as well as the socio-economic origins of the population. The adoption of domestic plants and animals was piecemeal, with hunting and gathering continuing as an important part of local subsistence strategies. Here, we investigate the timing and extent of the adoption of agricultural practices, namely herding and cultivation, in three diverse coastal and inland Early Neolithic sites in the Mediterranean Maghreb region, namely Ifri Oudadane, Ifri n'Etsedda and Hassi Ouenzga. Results from absorbed lipid residues extracted from 306 potsherds from these sites are correlated with information from faunal and archaeobotanical assemblages. Our findings suggest that agricultural practices, regarded as being of Neolithic origin, were never fully adopted in the Maghreb but rather that these farmer/foragers adopted a range of strategies including low-level food production (exploiting cereals and animal products, including meat and milk), gathering of wild plants and marine shellfish, and hunting both small and large sized game. These broad-spectrum farmer/foragers were clearly both flexible and resourceful and likely adapted their subsistence practices to maximise resource availability in an increasingly unpredictable environment.

## 1. Introduction

During the Holocene in the Mediterranean zone of north Africa, the transition from hunter-gathering to food-producing societies exploiting domesticates was a complex, multi-faceted process, which varied considerably through time and space. In a region marked by diverse environments and ecologies, the settlement dynamics and subsistence strategies of resident groups varied, with both local and broader regional cultural influences also playing a part in such behavioural changes.

A demographic increase in the prehistoric population of Early Holocene northeast Morocco was likely influenced by improved climatic and environmental conditions in the region e.g. a rise in water availability (Cheddadi *et al.* 2016), also evidenced in the Near East (deMenocal *et al.* 2000; Cacho *et al.* 2001; Migowski *et al.* 2006; Combourieu Nebout *et al.* 2009; Weninger *et al.* 2009; Schmiedl *et al.* 2010). This rise in temperature and water availability at the end of the Pleistocene led to the early Holocene transition from a semi-arid grasslands to open woodland with evergreen *Quercus*, seen in the Middle Atlas range (Campbell *et al.* 2017), and a modified fauna led to some adaptation of the hunter-gatherer land use strategies (Zapata *et al.* 2013; Linstädter *et al.* 2016). Human occupation at this time was characterised by Epipalaeolithic hunter-gatherer groups, well-adapted to the dry and open landscape of that earlier period, who have their roots in the Iberomaurusian of the late Upper Pleistocene (Linstädter and Kehl 2012). Although the transition from hunter-gathering to food production occurred during the so-called Epipalaeolithic to Early Neolithic stages, firstly through the adoption of technological innovations such as pottery and domesticated animals and plants by coastal populations, it is thought these initially played a marginal role in local subsistence strategies (Linstädter *et al.* 2012). Consequently, during the Neolithic period, animal herding and plant cultivation became one part of a multi-faceted subsistence strategy which also included hunting, gathering and the use of marine resources, an approach described by Smith (2001) as 'low-level food production'. In contrast, foragers occupying the hinterland of north-eastern Morocco adopted some Neolithic innovations, such as pottery, but appeared to resist the introduction of domesticates (Linstädter 2016). Ancient DNA recovered from both Epipalaeolithic and Neolithic individuals from Morocco confirms continuity in the local population during this period, with the migration of new farming people from Europe first recorded in the Late Neolithic and following periods (Fregel *et al.* 2018).

This broad-spectrum strategy may well have partly been driven by changing environmental conditions. Following the ‘dry’ 8.2 ka BP event, conditions returned to a warm and wetter period, although the boreal summer insolation (that led to the intensification of the monsoon systems) began to decline and the monsoon started to retreat southwards (Gasse 2000). Significantly, in the Western Mediterranean, in the landforms adjacent to the Alboran sea, temperatures began to decline at *c.* 7.8 ka BP, dropping  $\sim 3^{\circ}\text{C}$ , and annual and winter precipitation decreased by  $\sim 50\text{mm}$  (Dormoy *et al.* 2009). This mid Holocene trend toward cooling and drying expanded eastwards progressively and continued to 4 ka BP in the Northeast Atlantic and Mediterranean (Dormoy *et al.* 2009).

Here, we bring together, for the first time, organic residue analyses of pottery with data from faunal and archaeobotanical assemblages from three sites in the Mediterranean Maghreb, Ifri Oudadane (IOUD), Ifri n’Etsedda (INES) and Hassi Ouenzga (HAS) to provide a broad regional and chronological perspective of the transition to Neolithic practices, such as herding and cultivation, in Mediterranean northeastern Africa. All three rockshelter sites are located in very different areas of the Maghreb, namely the western littoral (IOUD, coastal site), the southern flank of the Kibdana mountains in the Lower Moulouya (INES, mountain site) and the Plain of Gerrouaou (HAS, plain site), respectively (Figures 1 and 2). This allows us to examine possible differences (and similarities) in the subsistence strategies of the groups living at these shelters.

## **2. The archaeological sites of Ifri Oudadane, Ifri n’Etsedda and Hassi Ouenzga**

### **2.1 Ifri Oudadane**

The site of Ifri Oudadane (IOUD, Figures 1 and 2a) is located on the northern coast of Morocco, west of the Melilla Peninsula and 5 km to the east of the Oued Kert River mouth and is currently 50m from the present shoreline. The site comprises a rock shelter, approximately 5m high and 15m wide, which was excavated during the years 2006, 2007, 2010 and 2011 by the joint Moroccan-German ‘Eastern Rif’ project with the participation of the Institut National des Sciences de l’Archéologie et du Patrimoine du Maroc (INSAP), the Kommission für die Archäologie Außereuropäischer Kulturen des Deutschen Archäologischen Instituts (KAAK) and the University of Cologne (Linstädter and Kehl 2012; Morales *et al.* 2013; Zapata *et al.* 2013).

Excavations revealed two metres of archaeological deposit covering a period of around 5000 years, accumulated during the first half of the Holocene, between 11.0 and 5.7 ka cal BP. This revealed two clear occupation periods showing the transition from the Epipalaeolithic to the Neolithic in the settlement, with continuity in the occupation of the cave. Evidence from the Epipalaeolithic deposits confirm the presence of hunter-gatherers who also exploited marine resources, including fishing and the consumption of mussels. The Early Neolithic (EN) was subdivided into three phases, ENA 7.6 to 7.3 cal BP, ENB 7.1 to 6.7 cal BP and ENC 6.6 to 6.4 cal BP, with ENB being the main occupation phase. In addition, the ENC phase was overlaid by the remains of a sporadic Late Neolithic (LN) occupation. The study of lithic artefacts showed no significant difference between the Epipalaeolithic and Early Neolithic period assemblages. Raw material procurement, blank production and tool kit remained unchanged, suggesting a population continuity during the transition. Interestingly, the earliest Neolithic dates from both Ifri Oudadane (6740±50 BP BETA295779, lentil) and Hassi Ouenzga (6710±50 BP KIA434, charcoal) are contemporaneous with data from Early Neolithic sites on the southern Iberian Peninsula (Linstädter and Kehl 2012; Linstädter *et al.* 2012; Morales *et al.* 2013; Morales *et al.* 2016). The Early Neolithic saw the first appearance of decorated pottery with *Cardium* impressed designs. It also marked the arrival of domesticated species such as sheep and goat, documented through bones and accumulation layers of calcite spherulites suggesting intensive penning of ovicaprids inside the shelter. Domesticated plants, including cereals (*Triticum monococcum/dicoccum*, *Triticum aestivum/durum*, *Hordeum vulgare*) and pulses (*Lens culinaris*, *Pisum sativum*, *Vicia faba*) were also present, confirmed by a large series of radiocarbon dates, in which a domesticated lentil provided the earliest date (see above) (BETA 295779; Morales *et al.* 2013; Morales *et al.* 2016). In fact, Ifri Oudadane is one of the few sites on the Mediterranean coast of Morocco where the early arrival of full Neolithic ‘traits’, including domesticated plants and animals, is documented. However, the exploitation of marine resources remained an important part of the Neolithic subsistence strategy, as did hunting and gathering (Linstädter and Wagner 2013; Morales *et al.* 2013). Certainly, the percentage of crops in the seed assemblage was less than 1% (Morales *et al.* 2016), confirming the continuing importance of wild plants in the economy of those groups.

## 2.2 Ifri n’Etsedda

The site of Ifri n’Etsedda (INES), an inland rock shelter in north-eastern Morocco, is located at the eastern fringe of the Rif range, on the southern flank of the Kbdana mountains (Figures

1 and 2c). It lies at an altitude of around 300 m a.s.l. and is embedded in a steep rock face of massive limestone (Linstädter *et al.* 2016), situated 7 km north of the lower Moulouya River (Figure 1).

Excavations between 2012 and 2014 revealed that occupation levels at the site covered around 4000 years, between 10.0 and 6.0 ka cal BP, including both Epipalaeolithic and Early Neolithic deposits. The Epipalaeolithic encompassed the period approximately 9.9 to 8.5 ka cal BP, with deposits comprising lithics and a large amount of shells, presumed to be food remains. The Early Neolithic was dated between 6288±40 (COL2377.1.1) and 5326±39 (COL2373.1.1) and, in these deposits, terrestrial snail shells remained a constant feature. Due to an interruption in settlement of more than 1000 years, the transition from the Epipalaeolithic to the Neolithic could not be directly investigated here, in contrast to the site of Ifri Oudadane. However, the study of the lithic assemblage also suggests a continuous tradition from the Epipalaeolithic into the Early Neolithic period. The main settlement phase at Ifri n'Etsedda is the ENC subphase. Pollen analyses indicate a trend towards aridization at the end of the ENC towards 6,300 cal BP. Ceramics were also present, from typical impression decorated ware at the beginning of the Neolithic to later herringbone decoration. The latter also indicates an occupation during the Late Neolithic after 6.0 cal BP. (Linstädter *et al.* 2016; Stempfle *et al.* 2018). The faunal assemblage comprised domesticated ovicaprids, from the earliest Neolithic, although these were only present at a maximum of 10% in each stratigraphic unit. The assemblage was dominated by aurochs, wild boar, wild Barbary sheep and gazelle (Linstädter *et al.* 2016).

### 2.3 Hassi Ouenzga

Hassi Ouenzga (HAS) is a small rock shelter site located in the Plain of Gerrouaou, some 40 km south of the Mediterranean coast (Figures 1 and 2b). The archaeological deposit was dated to the early 9<sup>th</sup> millennium cal BP (7930±50 BP – KIA433). From 7.5 ka cal BP (6710±50 BP – KIA434) onwards, impressed pottery appeared, showing a very particular local character (Linstädter 2003; Linstädter 2004). A few sherds decorated with *Cardium* impressions and incisions indicate relationships with the Mediterranean coast and may also suggest the early levels at HAS are contemporaneous to the Early Neolithic phase at Ifri Oudadane, when *Cardium* pottery also appeared. Domesticated fauna were present, in low abundances, but botanical remains do not show any traces of domesticated species, although the lack of remains may be a methodological problem, since systematic sampling and flotation of the sediments was not carried out here. The lithic industry also shows a clear origin in the late

Pleistocene/Early Holocene tradition. Here, a mixture of a late Epipalaeolithic lithic assemblage with Early Neolithic pottery and low abundances of domesticated species suggests this site belonged to a local hunter-gatherer community who had contacts to Neolithic groups at a time before food production had been adopted (Linstädter *et al.* 2012).

### 3. Lipid residue results

#### 3.1 Material and methods

The majority of sherds sampled from Ifri Oudadane were from the ENB phase, representing the main occupation phase between 7.1 and 6.6 ka cal BP, where pottery was characterised by single Cardium impressions. (Linstädter and Kehl 2012). Pottery from the latest phase of the Early Neolithic C (ENC) were also sampled. Pottery sampled at Ifri n'Etsedda includes the earliest Cardium-decorated pottery which first appeared in layer INES-5 (c. 7.2 ka cal BP) and also layer INES-6, dated to 6.8 – 6.5 ka cal BP. This is slightly later than the first appearance of pottery at both Ifri Oudadane and Hassi Ouenzga. Pottery from the following phase, INES-7, the main occupation phase, dated between 6.6 and 6.1 ka cal BP, contained impressions which form horizontal, vertical and oblique lines, predominantly made using combs, although when the so-called herringbone motive appears, this is formed by marine shells. Pottery sampled from layer INES-8, denoting a Late Neolithic occupation, comprised comb impressions and undecorated, polished vessels. (Stempfle *et al.* 2018). The earliest pottery sampled at Hassi Ouenzga originated from levels dated to 7.6 ka cal BP. The pottery displayed three distinct decorative motifs including, firstly, single impressions of Cardium shells, which have references to the Moroccan coast. Second were sherds decorated with cross-hatching, extremely similar to those found in the Oran area on the Algerian coast, together with a third group with decorations that only appeared at Hassi Ouenzga (Linstädter 2003; 2004).

Lipid analysis and interpretations were performed using well-established analytical procedures described in detail in the methods section (Dudd and Evershed 1998; Correa-Ascencio and Evershed 2014). All solvents used were HPLC grade (Rathburn) and the reagents were analytical grade (typically > 98% of purity). Briefly, ~2 g of potsherd were sampled and surfaces cleaned with a modelling drill to remove exogenous lipids. The cleaned sherd powder was crushed in a solvent-washed mortar and pestle and weighed into a furnace culture tube (I). An internal standard was added (20 µg *n*-tetratriacontane; Sigma Aldrich Company Ltd) together with 5 ml of H<sub>2</sub>SO<sub>4</sub>/MeOH 2 - 4% (δ<sup>13</sup>C measured) and the culture tubes were placed



on a heating block for 1 h at 70 °C, mixing every 10 min. Once cooled, the methanolic acid was transferred to test tubes and centrifuged at 2500 rpm for 10 min. The supernatant was then decanted into another furnaceed culture tube (II) and 2 mL of dichloromethane extracted double distilled water was added. In order to recover any lipids not fully solubilised by the methanol solution, 2 x 3 mL of *n*-hexane was added to the extracted potsherds contained in the original culture tubes, mixed well and transferred to culture tube II. The extraction was transferred to a clean, furnaceed 3.5 mL vial and blown down to dryness. Following this, 2 x 2 mL *n*-hexane was added directly to the H<sub>2</sub>SO<sub>4</sub>/ MeOH solution in culture tube II and whirlimixed to extract the remaining residues. This was transferred to the 3.5 mL vials and blown down under a gentle stream of nitrogen until a full vial of *n*-hexane remained. Aliquots of the extracts (containing fatty acid methyl esters, FAME's) were derivatised using *N,O*-bis(trimethylsilyl)trifluoroacetamide (BSTFA) containing 1 % *v/v* trimethylchlorosilane (TMCS; Sigma Aldrich Company Ltd.; 20 µL; 70°C, 1 h). Excess BSTFA was removed under nitrogen and the extract was dissolved in *n*-hexane for analysis by gas chromatography (GC), GC–mass spectrometry (GC–MS) and GC–combustion–isotope ratio MS (GC–C–IRMS).

All FAMEs initially underwent HTGC using a gas chromatograph (GC) fitted with a HT non-polar column (DB1-HT; 100% dimethylpolysiloxane, 15 m x 0.32 mm i.d., 0.1 µm film thickness). The carrier gas was helium and the temperature programme comprised a 50°C isothermal hold followed by an increase to 350°C at a rate of 10°C min<sup>-1</sup> followed by a 10 min isothermal hold. A procedural blank (no sample) was prepared and analysed alongside every batch of samples. Further compound identification was accomplished using GC-MS. FAMEs were then introduced by autosampler onto a GC-MS fitted with a non-polar column (100% dimethyl polysiloxane stationary phase; 60 m x 0.25 mm i.d., 0.1 µm film thickness). The instrument was a ThermoFinnigan single quadrupole TraceMS run in EI mode (electron energy 70 eV, scan time of 0.6 s). Samples were run in full scan mode (*m/z* 50–650) and the temperature programme comprised an isothermal hold at 50°C for 2 min, ramping to 300°C at 10° min<sup>-1</sup>, followed by an isothermal hold at 300°C (15 min). Data acquisition and processing were carried out using the HP Chemstation software (Rev. C.01.07 (27), Agilent Technologies) and Xcalibur software (version 3.0). Peaks were identified on the basis of their mass spectra and GC retention times, by comparison with the NIST mass spectral library (version 2.0).

Carbon isotope analyses by GC-C-IRMS were also carried out using a GC Agilent Technologies 7890A coupled to an Isoprime 100 (EI, 70eV, three Faraday cup collectors *m/z*

44, 45 and 46) via an IsoPrimeGC5 combustion interface with a CuO and silver wool reactor maintained at 850°C. Instrument accuracy was determined using an external FAME standard mixture (C<sub>11</sub>, C<sub>13</sub>, C<sub>16</sub>, C<sub>21</sub> and C<sub>23</sub>) of known isotopic composition. Samples were run in duplicate and an average taken. The  $\delta^{13}\text{C}$  values are the ratios  $^{13}\text{C}/^{12}\text{C}$  and expressed relative to the Vienna Pee Dee Belemnite, calibrated against a CO<sub>2</sub> reference gas of known isotopic composition. Instrument error was  $\pm 0.3\%$ . Data processing was carried out using Ion Vantage software (version 1.6.1.0, IsoPrime).

### 3.2 Lipid residue results

A total of 306 potsherds from Ifri Oudadane, Ifri n'Etsedda and Hassi Ouenzga were analysed using GC, GC-MS and GC-C-IRMS (Table 1). The rate of lipid recovery was 15%, 9% and 17% for Ifri Oudadane, Ifri n'Etsedda and Hassi Ouenzga, respectively (Table 1) which is lower than from ceramics recovered from Gueldaman Cave, Algeria (23%; Kherbouche *et al.* 2016; Dunne *et al.* 2017), a similar environment, and very low in comparison to ceramics analysed from the Libyan Sahara (94%; Dunne *et al.* 2012), which likely reflects the more favourable preservation conditions of arid environments. The mean lipid concentration of the sherds was 3.5, 0.3 and 0.04 mg g<sup>-1</sup> for Ifri Oudadane, Ifri n'Etsedda and Hassi Ouenzga, respectively (Table 1), with maximum lipid concentrations of 46.7, 1.3 and 0.2 mg g<sup>-1</sup> (Table 2). Overall, lipid recovery rates from all three sites were again similar to those from Gueldaman Cave, aside from two potsherds from Ifri Oudadane, OUD106 with high lipid recovery rates, at 46.7 mg g<sup>-1</sup> and OUD189 at 13.0 mg g<sup>-1</sup>.

As noted, a total of 47 lipid residues from the three sites contained sufficient concentrations (>5µg g<sup>-1</sup>) of lipids that can be reliably interpreted (Evershed 2008). These extracts comprised lipid profiles which demonstrated free fatty acids, palmitic (C<sub>16:0</sub>) and stearic (C<sub>18:0</sub>), typical of a degraded animal fat (Figure 2a, b, c), were the most abundant components (e.g. Evershed *et al.* 1997; Berstan *et al.* 2008). GC-C-IRMS analyses were carried out on the 47 FAMEs (fatty acid methyl esters, Table 2 and Figure 4) to determine the  $\delta^{13}\text{C}$  values of the major fatty acids, C<sub>16:0</sub> and C<sub>18:0</sub>, and ascertain the source of the lipids extracted. The  $\delta^{13}\text{C}$  values of the C<sub>16:0</sub> and C<sub>18:0</sub> fatty acids reflect their biosynthetic and dietary origin, allowing non-ruminant and ruminant adipose and ruminant dairy products to be distinguished (Copley *et al.* 2003; Dunne *et al.* 2012).

The  $\delta^{13}\text{C}_{16:0}$  values of the fatty acids range from -27.2 to -20.9 ‰ for Ifri Oudadane, -26.8 to -23.5 ‰ for Ifri n'Etsedda and from -27.0 to -23.7 ‰ for Hassi Ouenzga and the  $\delta^{13}\text{C}_{18:0}$  values range from -33.1 to -21.9 ‰ for Ifri Oudadane, -28.3 to -24.6 ‰ for Ifri n'Etsedda and from -29.4 to -23.4 ‰ for Hassi Ouenzga (Table 2). In comparison to  $\delta^{13}\text{C}_{16:0}$  values for modern British reference fats (Copley *et al.* 2003), the  $\delta^{13}\text{C}_{16:0}$  values suggest a variety of vegetation types ( $\text{C}_3$  and  $\text{C}_4$ ) were present in the environment. These are the two main carbon fixation pathways used by plants, with the majority of plants, including almost all woody species (trees, shrubs) of temperate and wet tropical regions and grasses which prefer wet, cool growing seasons, following the  $\text{C}_3$  pathway.  $\text{C}_4$  plants (e.g. maize, sorghum) tend to be found in sunny subtropical and tropical savannahs, generally hot environments characterised by seasonal soil water deficits (Vogts *et al.* 2012). The majority of  $\text{C}_4$  land plants belong to the grass (*Poaceae*) or sedges (*Cyperaceae*) species (Sage 1999). These data imply the animals producing these fats were subsisting on a  $\text{C}_3$  graze or browse diet but with occasional contributions from  $\text{C}_4$  plants, likely grasses, which are consumed by grazers (cattle or sheep).

Similar enrichment was observed in  $\delta^{13}\text{C}$  values of lipids extracted from Near Eastern archaeological pottery (Evershed *et al.* 2008) and in northern Greece (Whelton *et al.* 2018) where it was hypothesised to result from environmental factors such as aridity and/or the consumption of water-stressed  $\text{C}_3$  plants (Farquhar *et al.* 1989; Dunne *et al.* 2012).

### 3.3 Ifri Oudadane

The  $\Delta^{13}\text{C}$  values show that two FAMES, one from the ENB (OUD112, -3.6 ‰) and the other from the ENC-LN phase (OUD106, -6.0 ‰), can be unambiguously assigned to a ruminant dairy fat origin (11%, Table 2), plotting within the range of ruminant dairy fats determined by analysis of modern reference dairy fats from cattle and ewes raised on a strict  $\text{C}_3$  diet in Britain and  $\text{C}_3/\text{C}_4$  diets in Africa (Copley *et al.* 2003; Dunne *et al.* 2012). A further 7 residues from phase ENB (OUD098, OUD114, OUD118, OUD152, OUD184, OUD207 and OUD217 with  $\Delta^{13}\text{C}$  values of -0.9, -0.8, -2.0, -1.2, -1.0, -1.5 and -1.0 ‰, respectively), and 2 from the phase ENC/ENC-LN, OUD163 and OUD189 at -1.3 and -1.0 ‰, plot firmly within the range for ruminant adipose fat (Table 2). Four residues plot in the non-ruminant/plant range, OUD095, OUD194, OUD215 and OUD222, with  $\Delta^{13}\text{C}$  values of 1.2, 0.7, 0.6 and 0.3 ‰, respectively. These all originate from ENB levels. A further four, OUD094, OUD144, OUD203 and OUD210, plot between the ruminant/non-ruminant range with  $\Delta^{13}\text{C}$  values of -0.4, -0.5, 0.1 and -0.3 ‰, respectively, making attributions difficult and suggesting some mixing of animal

products, whether contemporaneously or during the lifetime use of the vessel. Of these, three come from phase ENB and one from ENC.

### 3.4 Ifri n'Etsedda

There is no evidence for the exploitation of dairy products at Ifri n'Etsedda and the majority of the residues plot within the ruminant adipose range, including all those from phase ENB and ENC. These comprise ETS229, ETS232, ETS252, ETS273, ETS277 AND ETS293 with  $\Delta^{13}\text{C}$  values of -1.5, -2.1, -2.3, -2.6, -2.5, -2.7 and -1.1 ‰, respectively. One Late Neolithic residue plots between the ruminant/non-ruminant range with a  $\Delta^{13}\text{C}$  value of -0.2 ‰, again suggesting some mixing of animal products in the vessel.

### 3.5 Hassi Ouenzga

The  $\Delta^{13}\text{C}$  values from Hassi Ouenzga residues show that two vessels (HAS322, -5.4 ‰ and HAS335, -3.3 ‰), from EN layer 3 (13%, Table 2), can be unambiguously assigned as ruminant dairy products, plotting within the range of ruminant dairy fats determined by analysis of modern reference dairy fats from cattle and ewes raised on a strict C<sub>3</sub> diet in Britain and C<sub>3</sub>/C<sub>4</sub> diets in Africa (Copley *et al.* 2003; Dunne *et al.* 2012). However, vessel HAS335 plots at the top of the range for dairy products, suggesting some mixing of carcass fats with dairy products. Eight lipid residues from the EN layer 3, HAS320, HAS324, HAS332, HAS336, HAS339, HAS350, HAS369 and HAS387, with  $\Delta^{13}\text{C}$  values of -1.1, -1.8, -1.2, -2.3, -1.2, -1.5, -1.2 and -2.2 ‰, and three from EN layer 4, HAS314, HAS357 and HAS394 with  $\Delta^{13}\text{C}$  values of -1.2, -0.9 and -0.9 ‰ (Table 2), plot within the ruminant adipose range. A further vessel from EN layer 3 (HAS353, -0.3 ‰) and two from EN layer 4 (HAS306 and HAS378, 0.3 and -0.3 ‰, respectively) plot on the border between the ruminant/non-ruminant range, indicative of some mixing of animal products in the vessel.

### 3.6 Aquatic and freshwater biomarkers

At Ifri Oudadane, the large amounts of fish bones and marine shells found in all archaeological layers of the site (Table 3) suggests the exploitation of marine resources remained an important part of the Neolithic subsistence strategy at this site (Linstädter and Kehl 2012; Linstädter and Wagner 2013). Consequently, FAMES from the three sites were analysed by GC-MS in SIM mode to check for the presence of aquatic (Ifri Oudadane coastal site) or freshwater (Ifri n'Etsedda and Hassi Ouenzga inland sites) biomarkers, which would denote the processing of

shellfish/crustaceans, fish and marine mammals. These comprise  $\omega$ -(*o*-alkylphenyl) alkanolic acids (APAAs) and vicinal dihydroxy acid (DHYAs) which originate from the degradation of poly- and monounsaturated fatty acids found in marine or freshwater fats and oils and are routinely used to detect marine product processing (e.g. Hansel *et al.* 2004; Craig *et al.* 2007; Hansel and Evershed 2009; Cramp *et al.* 2014; Cramp *et al.* 2015). Significantly, no aquatic or freshwater biomarkers were identified in any of the analysed potsherds.

#### 4. Faunal analysis – Comparative quantification of faunal remains versus lipid residues

##### 4.1 Data classification and aggregation

Here, we have aggregated certain phases at each site in order to maximise sample size and provide broadly comparative temporal ranges, allowing a meaningful comparison of the faunal remains with the organic residue results. The combined phases therefore comprise Hassi Ouenzga units 3-5, Ifri Oudadane ENB, Ifri Oudadane ENC-LN and Ifri n'Etsedda units 6-8. Whilst the relative proportion of all faunal remains from each of the identified stratigraphic units are shown in Table 3, only a subset of these faunal remains was used in the comparative analysis. As no aquatic biomarkers were identified in the pottery, we have excluded fish from the analysis. Equally, we have excluded all indeterminate remains, birds, amphibians and fur-bearing animals such as jackal, fox etc. This provides two datasets on the exploitation of terrestrial animal resources, one from the faunal remains and one from the lipid residues. The relative proportions of animal product categories from the pottery are calculated as a percentage of all identified fat categories per phase (Table 5 and Figure 5). Meanwhile, the relative proportion of faunal categories were calculated by grouping all identified terrestrial species into broadly comparable categories to the animal fat (product) types (Table 6 and Figure 6). These consist of three categories comprising wild ruminants, which includes Barbary sheep (*Ammotragus lervia*), Cuvier's Gazelle (*Gazella cuvieri*), Gazelle (*Gazella* sp), Aurochs (*Bos primigenius*) and Bubal Hartebeest (*Alcelaphus buselaphus*) and wild non-ruminants species, consisting of reptiles, *Sus scrofa*, *Equus* sp., Rhinocerotidae indet., Cape Hare (*Lepus capensis*), European Rabbit (*Oryctolagus cuniculus*) and Leporidae indet. The final category comprises domesticated animals that produce dairy products, e.g. sheep (*Ovis aries*), goat (*Capra hircus*) and sheep/goat category (*Ovicaprine*). Although, in reality, it is likely that a proportion of the domestic ruminant species contributed to the ruminant adipose fats in the pottery, they have necessarily been assigned only to the dairy category for the sake of this analysis for three reasons: 1) the lifetime biomass of milk yield is significantly greater than

meat and therefore the presence of dairying animals would contribute substantially more milk than meat during the lifetime of use of a single pot, 2) the predominance of wild non-ruminants in the faunal data makes the contribution of both dairy animals and ruminant species trivial in comparison to wild non-ruminants, and 3) double counting the dairy animals into both the ruminant adipose category and milk category would artificially inflate the total NISP giving false precision.

#### **4.2 Species abundance**

Wild non-ruminants are the predominant animal group at both HAS and IOUD sites (Table 3). Fish formed a substantial part of the faunal repertoire at IOUD, perhaps unsurprisingly considering the proximity of the site to the coast, comprising over 70% of all faunal remains, but do not appear at all at the inland sites INES and HAS. In contrast, at HAS, reptiles were the predominant species, comprising 85% of the total NISP. Birds, sea mammals, rabbit/hare and other small mammals such as crested porcupine also played a consistent role in the subsistence of Neolithic populations in the Rif. This broad-spectrum approach to faunal subsistence appears to continue throughout the Neolithic period, despite the introduction of domestic animals, supporting the notion that these animals only played a marginal role in local subsistence strategies (Smith 2001; Linstädter *et al.* 2012). This is in direct contrast to the early Neolithic Cardial cultures of the Iberian peninsula where, despite apparent cultural links with North Africa, domestic animals contributed a substantial portion of the subsistence economy, with frequencies of over 70% (Saña 2013). This predominance of non-domestic species, and particularly non-mammalian species, at certain sites, suggests a substantial degree of economic continuity from the Epipalaeolithic to the early Neolithic in some parts of the Rif region. Whilst small taxa predominate at HAS and IOUD, however, the faunal remains from INES are dominated by ruminant taxa. Of the identifiable Bovidae, 63% are domestic ovicaprines, suggesting they are better represented than wild ruminants, and whilst the majority of ruminant remains are indeterminate Bovidae, meaning they are indistinguishable as domestic or wild ruminants, it is likely that a substantial proportion of the indeterminate remains do, in fact, originate from domestic taxa.

#### **4.3 Comparative lipid residues and faunal analysis**

The comparison of the pottery lipid residues and faunal remains can provide greater insight into the nature of faunal exploitation in this region of Morocco although it should be noted that

both the deposition of faunal remains and the preservation of lipid residues in pottery come with a host of biases. In order to robustly compare the relative proportions of different animal products it is necessary to incorporate the uncertainty in our data. In particular, the recovery rate of lipid residues from these sites is relatively low, and therefore cannot be directly compared with the faunal categories. To accommodate these uncertainties in frequency estimates due to finite sample sizes we have performed Chi-squared tests between the faunal data and potsherd data using the raw integer counts, and graphically represent the uncertainty in the proportions on a ternary diagram, by randomly sampling from a Dirichlet distribution, using the observed counts plus 1 (a uniform prior) as shape parameters. As shown in Figures 7-10, there appears to be a systematic difference in the representation of faunal exploitation between the lipid residues and the faunal remains. The distribution associated with the faunal remains is typically smaller due to the larger sample size, and, demonstrates a predominant reliance on wild non-ruminants. Meanwhile, the lipid residues have a much broader distribution due to small sample sizes and indicate a predominance of wild ruminant or wild non-ruminant adipose processing. The small sample sizes for both the fauna and lipid residues at Ifri Oudadane ENC-LN are represented in Figure 10, where the random sample of Dirichlet deviates shows a very broad spread on all three axes.

A chi-square test was performed to test for a difference between the faunal fat proportions and potsherds fat proportions. Table 4 shows the results of these tests, confirming significant differences at all sites ( $p$ -value =  $<0.01$ ) except for the ENC-LN phase at Ifri Oudadane ( $p$ -value = 0.7) where the small sample size prohibits drawing any significant conclusions. We therefore fail to reject the null hypothesis of no difference at almost all sites. Thus, the lipid data from the potsherds do not appear to be a random subset of the archaeological dataset, providing important information about the way in which different animal products were being processed at these sites.

## 5. Plant processing

### 5.1 Plant lipid profiles and $\delta^{13}\text{C}$ values

Interestingly, a similar number of lipid profiles from each site, Ifri Oudadane ( $n=5$ , 26% of the total, OUD098, OUD114, OUD152, OUD203, OUD215, Figure 3d), Hassi Ouenzga ( $n=4$ , 25% of the total, HAS303, HAS306, HAS314, HAS369, Figure 3e) and Ifri n'Etsedda ( $n=3$ , 27% of the total, ETS229, ETS252, ETS273, Figure 3f) differ from those commonly seen in

animal product profiles. These comprise sequences of even-numbered long-chain fatty acids (LCFAs), containing C<sub>20</sub> to C<sub>30</sub> carbon atoms, generally dominated by the C<sub>20</sub> or C<sub>24</sub> (Figure 3). These LCFAs are strongly indicative either of an origin in leaf or stem epicuticular waxes (Kolattukudy *et al.* 1976; Tulloch 1976; Bianchi 1995; Kunst and Samuels 2003) or, possibly, suberin (Kolattukudy 1980; 1981; Walton 1990; Pollard *et al.* 2008), an aliphatic polyester found in all plants. Although primarily found on the surface of plant leaves, sheaths, stems and fruits, epicuticular waxes are also found associated with other plant organs, i.e. seed oils and coats, flowers, bark and husks (Bianchi 1995). Fatty acids can also be found in high abundances in some plants, for example, waxes from the leaves of the sorghum plant comprise nearly 40% very long-chain fatty acids (Bianchi, 1995), and for the sorghum grain wax, the most abundant homologues of aldehydes, alcohols and acids were C<sub>28</sub> and C<sub>30</sub> (Hwang *et al.* 2002). Long-chain fatty acids can also be found in plant oils, for example, groundnut oil comprises 4-7% of C<sub>20</sub>, C<sub>22</sub> and C<sub>24</sub> saturated and monoene acids (Gunstone 2004). However, these LCFAs are not diagnostic to families of plants and so cannot be used as anything other than a general indicator for plant processing. Where possible, the  $\delta^{13}\text{C}$  values of the long-chain fatty acids were measured to confirm the origin of the plant waxes (i.e. C<sub>3</sub> or C<sub>4</sub>, Table 7). These  $\delta^{13}\text{C}$  values fall within the known  $\delta^{13}\text{C}$  values for C<sub>3</sub> bulk plant lipids, which range from -32 to -20 ‰ for C<sub>3</sub> plants (Boutton 1991). Also present in a number of these profiles is a series of odd-over-even long-chain *n*-alkanes, ranging from C<sub>25</sub> to C<sub>33</sub>, dominated by the C<sub>29</sub> and C<sub>31</sub> *n*-alkanes. These were present in lower abundance and could not be measured isotopically. Alkanes are also common components of waxes, usually occurring in low concentrations (Koch and Ensikat 2008), although occasionally they are the dominant lipid, e.g. the leaf wax of *Cotyledon orbicularis* is almost entirely comprised of alkanes (Eglinton and Hamilton 1967). Long-chain *n*-alkane distributions occur in the range C<sub>25</sub> to C<sub>35</sub> (Chibnall *et al.* 1934), with an odd-over-even predominance (Eglinton and Hamilton 1967). The dominant chain lengths vary across plant taxonomic groups but the C<sub>27</sub>, C<sub>29</sub>, C<sub>31</sub> and C<sub>33</sub> homologues usually predominate (Diefendorf *et al.* 2011). Significantly, an analysis of leaf wax alkanes extracted from 93 species belonging to five subfamilies, *Bambusoideae*, *Pooideae*, *Arundinoideae*, *Chloridoideae* and *Panicoideae*, of the Gramineae (grass family), showed that the C<sub>29</sub> and C<sub>31</sub> *n*-alkanes dominated (Maffei 1996). This combination of LCFAs and *n*-alkanes, combined with the  $\delta^{13}\text{C}$  values of the LCFAs, in the Rif pottery profiles, strongly suggests the processing of C<sub>3</sub> plant material, likely leafy plants and/or wild grasses, within approximately one quarter of vessels from each site, possibly mixed with animal products.



## 5.2 Plant lipids and archaeobotanical data

### 5.2 Plant lipids and archaeobotanical data

How do these plant lipid profiles correlate with archaeobotanical data from the three sites? Unfortunately, flotation for plant remains was not carried out at Hassi Ouenzga and therefore identification of particular plants that may have been exploited there was not possible. However, at Ifri Oudadane, plant macro-remains were extremely well-preserved with archaeobotanical studies revealing the importance of exploiting a huge variety of both wild and domesticated plants, ranging from grasses to legumes and fruits of different tree species (Morales *et al.* 2013; Morales *et al.* 2016; Morales 2018). It is noteworthy that the Epipalaeolithic layers contained a significant number of wild plant remains (742) but the majority were found in Neolithic layers (7199) and only the Neolithic layers yielded domesticated plants. By far the most dominant species identified (5842) was seeds from the mastic tree (Figure 11; *Pistacia lentiscus*). Interestingly, ethnographic studies show that mastic fruits were used, in addition to human food, both as a source of oil for cooking and lighting in Northern Africa and Southern Spain (Rivera-Núñez and Obón-de-Castro 1991; Torres-Montes 2004) and also as a food for livestock (Zapata *et al.* 2003). Wild legumes (*Lathyrus* sp./*Vicia* sp.), large seeds with a high content of carbohydrates and proteins (Butler *et al.* 1998), were commonplace throughout the stratigraphy, suggesting they were collected for consumption. Seeds of *Vicia* sp, *Lathyrus* sp and *Lens* cf. *nigricans* have also been documented in the late Iberomaurusian levels of Taforalt (Humphrey *et al.* 2014), as well as in Holocene layers at Kaf Taht el-Ghar (Morales 2018). At Taforalt, this reliance on these wild plants, such as acorns, pine nuts and wild pulses, rich in fermentable carbohydrates, is thought to have been the cause of a disease-associated oral microbiota and early incidences of caries in hunter-gatherer populations (Humphrey *et al.* 2014). The dwarf palm (*Chamaerops humilis*) was also common throughout the sequence (Morales *et al.* 2013). This palm produces a type of date that was consumed in Northern Africa and Southern Spain, despite the astringent taste of the fruit (Rivera-Núñez and Obón-de-Castro 1991; Torres-Montes 2004). Also present, and likely eaten for food, were seeds from juniper (*Juniperus phoenicea*), wild olive (*Olea europaea*) and acorns (*Quercus* sp.). Although small (61 plant macroremains in total), the domesticated crop assemblage from Ifri Oudadane, only found in Neolithic levels, included einkorn (*Triticum monococcum*), emmer (*Triticum dicoccum*), hard wheat (*Triticum durum*), free-threshing wheat (*Triticum aestivum/durum*) and barley (*Hordeum vulgare*). Two domesticated pulses,

pea (*Pisum sativum*) and lentil (*Lens culinaris*), were also present (Morales *et al.* 2013). Radiocarbon dates on nine seeds, ranging from 6740±50 BP (Beta-295779) to 5590±40 BP (Beta-295772), confirm their presence in the Early Neolithic phase. Pollen analysis at the site also confirms the presence of grasses, likely cereal grains, in the shelter (Zapata *et al.* 2013)

Plant macroremains at Ifri n'Etsedda are still under study but pollen samples were taken from several levels, including INES4, INES6, INES9 and INES8 (Linstädter *et al.* 2016). These indicate that, during the ENB (ca. 6.8 ka cal BP, INES-6) wild olive (*Olea europaea*), carob tree (*Ceratonia siliqua*) and Atlantic terebinth (*Pistacia terebinthus*), present in the Late Epipalaeolithic (INES4), continue to be abundant, together with Phoenician juniper (*Juniperus phoenicea*), myrtle (*Myrtus communis*) and Mediterranean buckthorn (*Rhamnus alaternus*). The presence of cereal grains is also documented with percentages (≥5%) sufficient to suggest local cultures in the vicinity of the shelter (López-Sáez and López-Merino 2005). These are accompanied by anthropic indicators and the presence of ruderal taxa such as Aster, Cardueae and *Malva sylvestris* (Behre 1981; López-Sáez *et al.* 2003).

## 6. Discussion

### 6.1 Meat and milk exploitation

In isolation, lipid residue results from the three sites, Ifri Oudadane, Ifri n'Etsedda and Hassi Ouenzga, suggests the subsistence strategies of these Holocene groups, at both coastal and inland sites, were mainly focused on ruminant carcass product processing. Some hunting of wild non-ruminants featured at all sites and low levels of dairying occurred at Ifri Oudadane and Hassi Ouenzga. There is no evidence of dairying at Ifri n'Etsedda although this may simply reflect the small sample size, particularly as the faunal remains are dominated by domesticated ruminant taxa, which indicates dairying would be possible. Indeed, of the identifiable Bovidae, 63% are domestic ovicaprines.

However, examination of the faunal remains and lipid residues from pottery in tandem provides very different insights into the animal exploitation strategies and animal product processing during the Neolithic period in northeastern Morocco. Whilst the fauna reveals a notably broad-spectrum exploitation strategy, targeted particularly on relatively small non-mammalian species, such as fish and reptiles, the pots reveal a fairly specialised processing activity focused on ruminant, and to a lesser extent, non-ruminant adipose products. Even undertaking a

comparative analysis that only considers the terrestrial animal resources (Figures 7-10), it is clear the pots are being used for specific animal product types, whilst other resources are likely processed in different ways. One caveat to keep in mind, however, is that indeterminate mammals, which comprise 14% of the total NISP from all sites, were excluded from this analysis. Whilst these remains may represent a greater proportion of dairying or ruminant taxa, it seems fair to assume that the identifiable mammalian remains are a fair sample of the indeterminate remains.

This predominance of non-domestic species, and particularly non-mammalian species suggests a substantial degree of economic continuity from the Epipalaeolithic to the Early Neolithic in some parts of the Rif region. For example, the larger mammal fauna in the Iberomaurusian levels at Ifri El Baroud (Gunpowder Cave, northeast Morocco) comprises large ungulates such as wild equid species (*Equus* sp.), Cuvier's Gazelle (*Gazella cuvieri*), Bubal Hartebeest (*Alcelaphus buselaphus*), and Aurochs (*Bos primigenius*), very similar to that seen at the Rif sites discussed here (Potì *et al.* 2019). These taxa are mostly known to reside in open habitats or savannahs, which suggests that the environment in Holocene northeast Morocco was quite different from that of today (Michel *et al.* 2009).

Where dairying is present in the site lipid profiles, it is in relatively low numbers (10% at Ifri Oudadane and 13% at Hassi Ouenzga) and supported by the low frequency counts of domestic dairying animals. At Hassi Ouenzga, both dairy vessels came from layer 3 (7.1 to 5.8 cal BP), whilst at Ifri Oudadane one of the dairy vessels comes from level ENB (7.1 to 6.7 cal BP) and the other was found in Late Neolithic (ENC LN) levels. This correlates well with lipid data from the rockshelter site of Gueldaman, Algeria, where dairy products are exploited from the earliest introduction of domesticates (5%), albeit at a very low abundance (Dunne *et al.* 2017). However, this apparently systematic bias between the pots and animal bones demonstrates the need to assess dietary behaviours from multiple lines of evidence. It is likely that the majority of animals being consumed at these sites were not being processed in pots. Fish and reptiles, in particular, which contribute a large proportion of the identified taxa, may have been grilled, rather than boiled, in which case they would not be identifiable in the lipid residue profiles. This is confirmed by the complete absence of lipid biomarkers denoting aquatic or freshwater product processing at the three sites.

Ifri Oudadane is one of the few sites on the Mediterranean coast of Morocco where the early arrival of Neolithic practices of herding and cultivation, utilising domesticated plants and

animals, is documented. For example, as noted, the shelter was clearly used to pen flocks, evidenced by the presence of both coprolites and calcite spherulites in the Early Neolithic deposits (Linstädter and Kehl 2012). However, the exploitation of marine resources remained an important part of the group's subsistence strategy, as did hunting and gathering (Linstädter and Wagner 2013; Morales *et al.* 2013). Similarly, at Ifri n'Etsedda, although arriving slightly later, food production was marked by the appearance of ovicaprids and the site served as a shelter connected to herding activities, although hunting of medium to large game continued. This may be connected to local topography and/or to selective exploitation that maximised specialised hunting strategies (Potì *et al.* 2019). The groups exploiting the rockshelter at Hassi Ouenzga began to exploit domesticates contemporaneously with those at Ifri Oudadane. The low abundance of lipids recovered from these vessels, in comparison to both Ifri Oudadane and Ifri n'Etsedda, might suggest the shelter was used more sporadically. Data from all three sites suggest that the full adoption of practices regarded as 'Neolithic', such as the herding of animals and the cultivation of plants did not take place and the groups continued to rely on a broad-spectrum subsistence strategy. This was likely because of changing environmental conditions, as evidenced by the aridification that occurs during the first half of the 6<sup>th</sup> millennium BP indicated by the record at both Ifri Oudadane and Ifri n'Etsedda (Linstädter and Kehl 2012; Linstädter *et al.* 2016). Decreases of the western Mediterranean Sea surface temperature (Cacho *et al.* 2001) and the end of initial soil formations in the alluvial deposits of the lower Moulaya valley attest to these environmental shifts (Linstädter and Kehl 2012). These dry conditions would have likely led to increased grazing pressure for stock animals. Furthermore, the decrease in anthropic herbs at both sites suggests a reduction in the cultivation of domesticated plants.

## 6.2 Plant processing

The role of wild plants in the subsistence of Late Pleistocene and Early to Mid-Holocene groups in Mediterranean north Africa is still not fully understood, largely due to poor preservation of putative botanical remains and, until recently, the lack of systematic sampling of archaeobotanical remains at sites. However, it seems likely that plant food had a more important role in the diet of Palaeolithic hunter-gatherers than was previously supposed (Humphrey *et al.* 2014; Morales 2018, Barton *et al.* 2018). For example, at Huah Fteah cave in northeast Libya, usewear and residue analysis of a seed grinding stone, from occupation layers dating to *c.* 31 ka, revealed the processing of *Aegilops* sp. (goat grass). Furthermore, a similar

assemblage of charred seed remains, including Aleppo pine (*Pinus halepensis*), juniper (*Juniperus phoenicea*), wild legumes (*Lathyrus/ Vicia* sp.), mastic tree (*Pistacia lentiscus*) and acorns (*Quercus coccifera*), among other taxa, was recorded in Late Pleistocene and Early Holocene layers. All produce edible fruits and/or seeds (Barker *et al.* 2010). A recent synthesis of plant macroremains from three sites in Morocco, Grotte des Pigeons at Taforalt, Ifri Oudadane (included here) and Kaf That el-Ghar and El Mekta in Tunisia, also suggests the systematic gathering and thermal processing of a number of the same wild plants. Rich in carbohydrates, lipids and proteins, these include acorns, pine nuts, juniper berries, wild pistachio nutlets, wild legumes and grasses. These were recorded at all four sites, spanning the period from the Iberomaurusian through to the Neolithic (Morales 2018).

The adoption of new (thermally resistant) ceramic technology by Neolithic hunter-gather groups in the Rif region opens the possibility of novel processing techniques, such as prolonged boiling, which would have rendered new plantstuffs comestible, unlocking their nutritional potential and extending their storage life (Arnold 1985). For example, the increase in the number of remains of the seeds of the mastic tree (*Pistacia lentiscus*) and other wild taxa found in Neolithic layers at Ifri Oudadane could be linked to the introduction of pottery, which could have been used to boil the fruits and release their oils with less effort than with other methods, such as grinding. Furthermore, the cooking of these high energy-yielding plant foods, such as wild legumes, acorns and nuts (rich in carbohydrates), common at Rif sites, would also have led to an increased speed of digestibility and consequent energy gain (Carmody and Wrangham 2009).

The combination of LCFAs and *n*-alkanes, and associated  $\delta^{13}\text{C}$  values for the LCFAs, in c. 25% of the pottery lipid profiles at each site strongly suggests the processing of  $\text{C}_3$  plant material, likely leafy plants and/or wild grasses, within these vessels, possibly mixed with animal products. The recovery of archaeobotanical remains of leafy plants is rare at archaeological sites due to their fragility, but ethnographic studies in traditional hunter-gatherer and farming groups indicate that fresh leaves are a common food plant (Cotton and Wilkie 1996; Weisskopf 2018). As for wild grasses, seeds of wild oat (*Avena* sp.) and other large seeded grasses such as *Phalaris* have been recorded in Epipalaeolithic and Neolithic sites of the region (Morales 2018), suggesting the likely processing of wild grasses within the Maghreb vessels. Thus, despite the somewhat 'invisible' nature of plant processing (Hillman *et al.* 1989a; 1989b), these data confirm the importance of wild plants in the subsistence strategies

of these groups. It should be noted that animal products and plants are often not consumed alone but rather prepared in different combinations of processed ingredients, which together constitute a meal or dish (Hastorf 2016). The availability of a new technology, pottery vessels for cooking, in particular, boiling, may have expanded the number of plants consumed and the combination of ingredients available to make a meal. Thus, the introduction of ceramics for food processing in the Neolithic probably increased the important role that wild plants played in the subsistence strategies of these groups.

## 7.0 Conclusion

Here, we investigate the timing and extent of the adoption of agricultural practices, namely herding and cultivation, in three diverse coastal and inland Early Neolithic sites in the Mediterranean Maghreb region, Ifri Oudadane, Ifri n'Etsedda and Hassi Ouenzga. Organic residue analyses of pottery combined with data from faunal and archaeobotanical assemblages from these three sites has provided a broad regional and chronological perspective of the transition to so-called Neolithic practices of herding and cultivation in this area. Our findings confirm that these practices were never fully adopted in the Maghreb but rather that these farmer/foragers adopted a range of strategies including low-level food production (exploiting cereals and animal products including meat and milk), gathering wild plants and marine shellfish and hunting both small and large sized game. Significantly, our examination of the faunal remains and lipid residues from pottery in tandem provides a new insight into both the animal exploitation strategies and animal product processing in pottery vessels during the Neolithic in northeastern Morocco. These data suggest that, in the Rif region, pots played a particular role for these groups and were used specifically to process the products of medium-large ungulates, whether wild or domesticated. The fauna most commonly exploited, reptiles and small mammalian taxa, were likely not processed in pots, but cooked by other methods. The presence of lipids denoting plant processing in vessels, in around one quarter of vessels at all sites, also confirms the importance of plants in the diet of these forager/farmers. The combination of specific animal products and plants within the vessels suggests these groups appeared to utilise the new technology of pottery in a very diverse manner. Furthermore, both the plant and animal lipids provided important information on the environment present in the region during the Early Neolithic, with  $\delta^{13}\text{C}$  values showing a predominance of  $\text{C}_3$  (temperate) plants.

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**Author contributions**

The project was designed by KM, JD, JL, ND, AM and RPE, and the paper was written by JD, KM, JL and JM. JD and TG performed analytical work and data analysis. RH and SL analysed the faunal assemblages and PB helped with sampling and provided figures for the paper.

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## Figures and tables

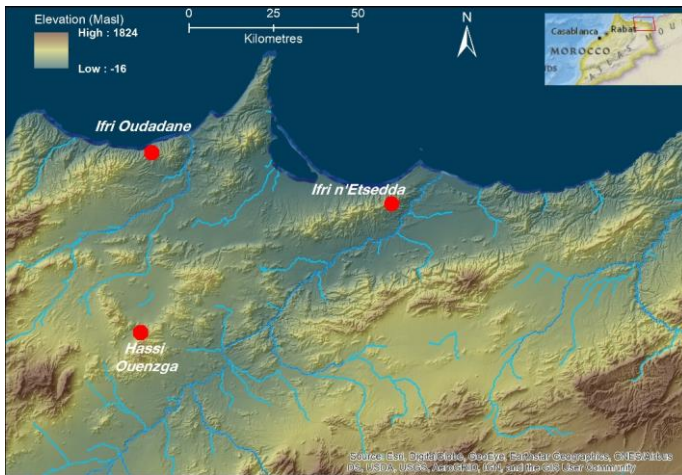


Figure 1. Map of three sites analysed here, Ifri Oudadane, Hassi Ouenzga and Ifri n'Etsedda, in the Moroccan Rif area

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3 Figure 2. Images of three sites sampled a. Ifri Oudadane, b. Hassi Ouenzga and c. Ifri n'Etsedda

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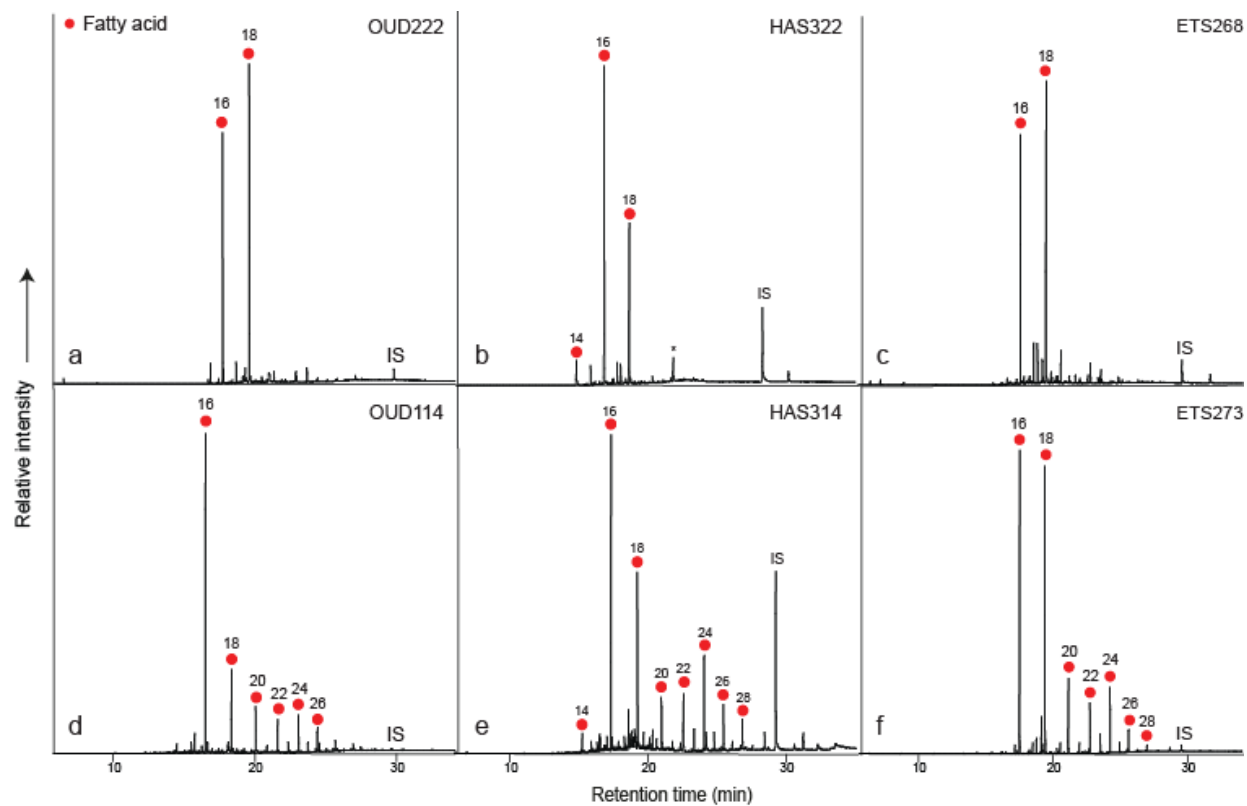


Figure 3. Partial gas chromatograms of trimethylsilylated FAMES showing typical degraded animal fat and plant lipid profiles from a. and d. Ifri Oudadane, b. and e. Hassi Ouenzga and c. and f. Ifri n'Etsedda, respectively. Red circles, *n*-alkanoic acids (fatty acids, FA); IS, internal standard,  $C_{34}$  *n*-tetratriacontane.

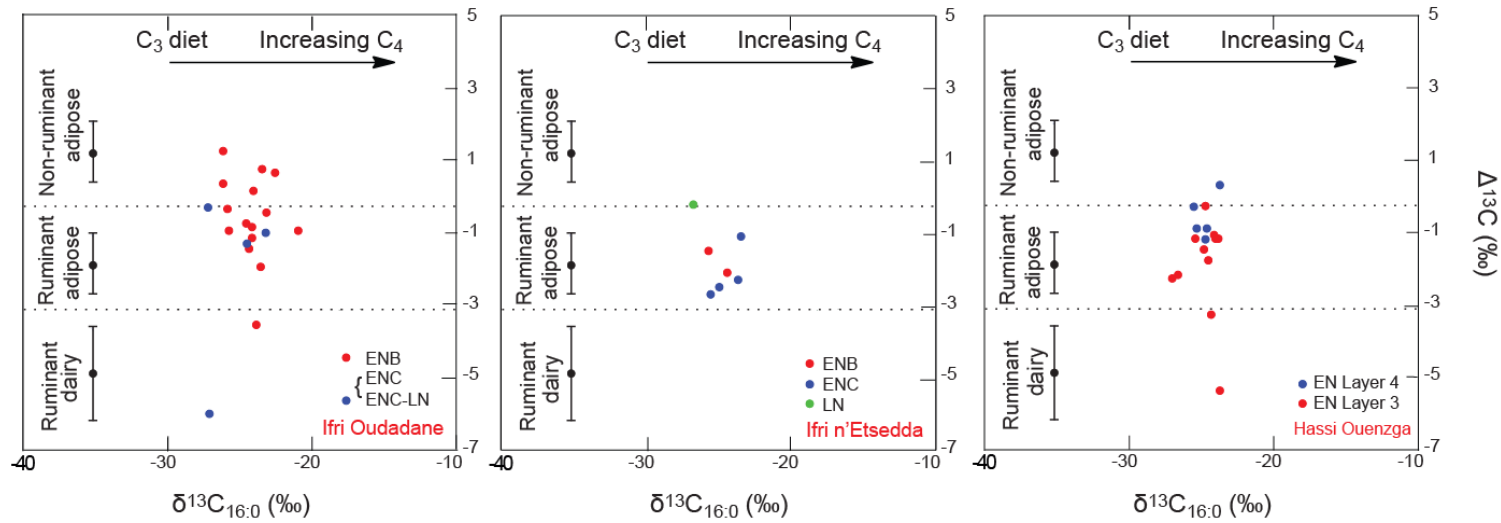


Figure 43. Graphs showing:  $\Delta^{13}\text{C}$  ( $\delta^{13}\text{C}_{18:0} - \delta^{13}\text{C}_{16:0}$ ) values from the sites of Ifri Oudadane, Ifri n'Etседда and Hassi Ouenzga. The ranges shown here represent the mean  $\pm$  1 s.d. of the  $\Delta^{13}\text{C}$  values for a global database comprising modern reference animal fats from Africa (Dunne et al., 2012), UK (animals raised on a pure  $\text{C}_3$  diet) (Dudd and Evershed, 1998), Kazakhstan (Outram et al., 2009), Switzerland (Spangenberg et al., 2006) and the Near East (Gregg et al., 2009), published elsewhere.

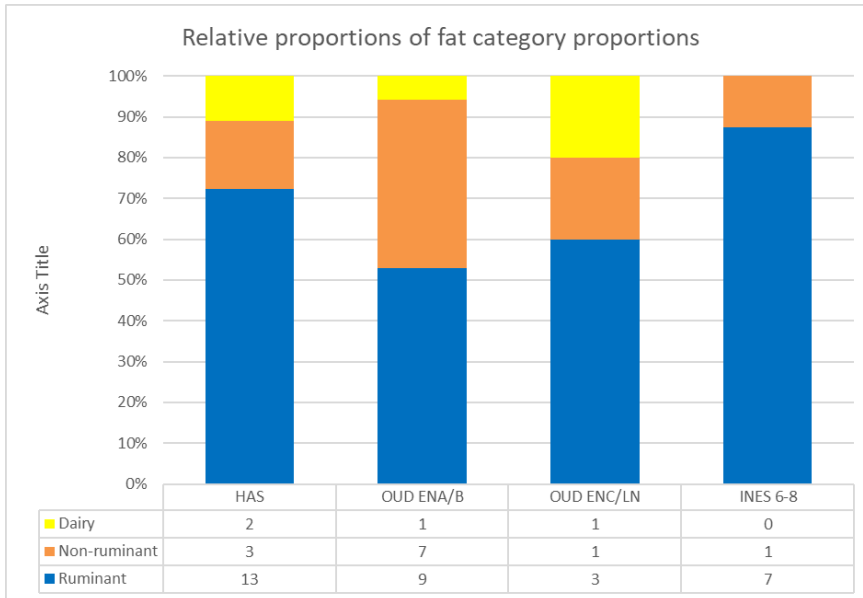


Figure 5. Relative proportions of fat categories as a percentage of all fat classifications per phase (note: plant categories are not included)

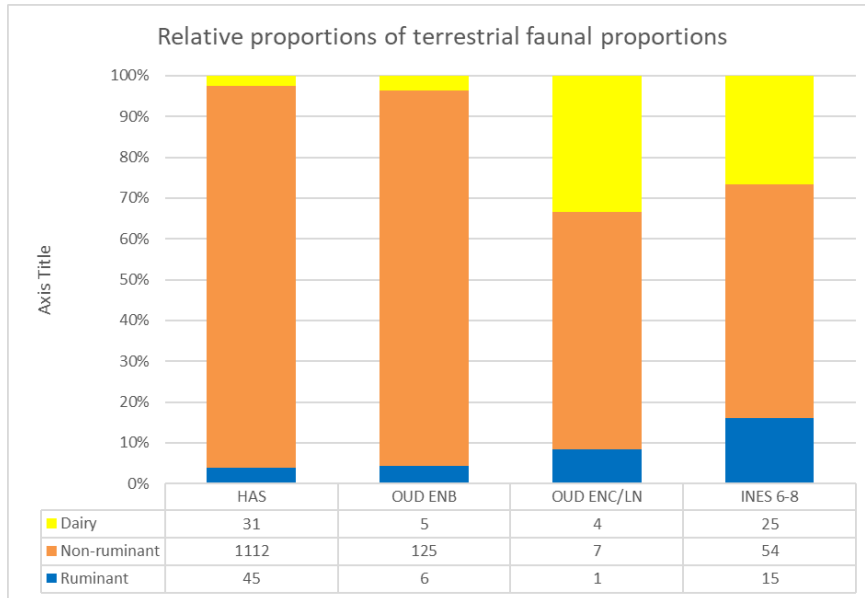


Figure 6. Relative proportions of faunal categories, based on the NISP shown in table 1 (note: all fish, birds and indeterminate mammals are not included)

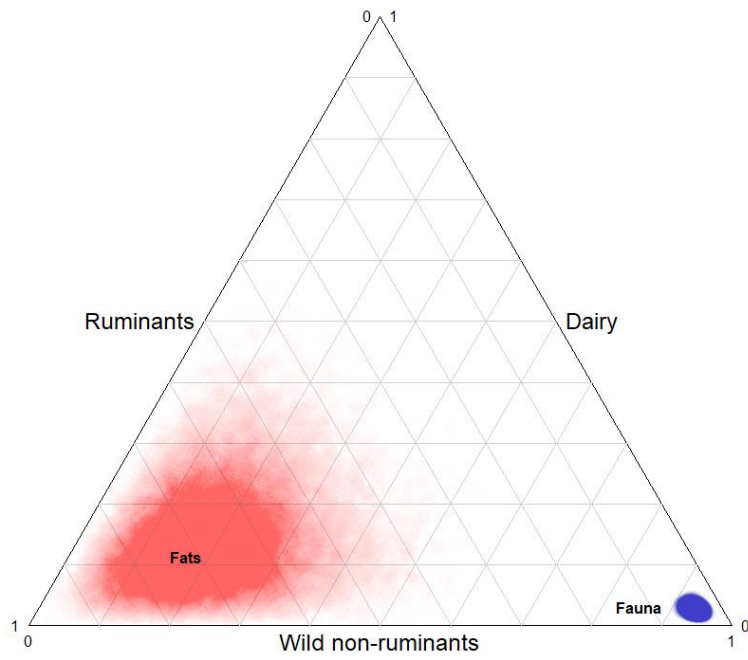


Figure 7. Triangle plot of faunal remain proportion and lipid residue proportions (confidence range represented using random Dirichlet samples of the integer counts in tables 5 and 6 plus a uniform prior) for the faunal remains and lipid residues from HAS



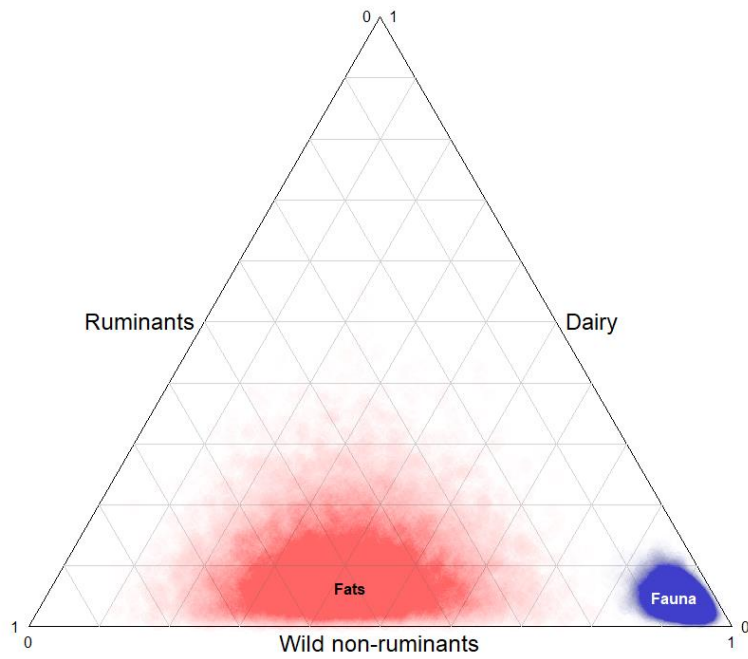


Figure 8. Triangle plot of faunal remain proportion and lipid residue proportions (confidence range represented using random Dirichlet samples of the integer counts in tables 5 and 6 plus a uniform prior) for the faunal remains and lipid residues from IOUD ENB

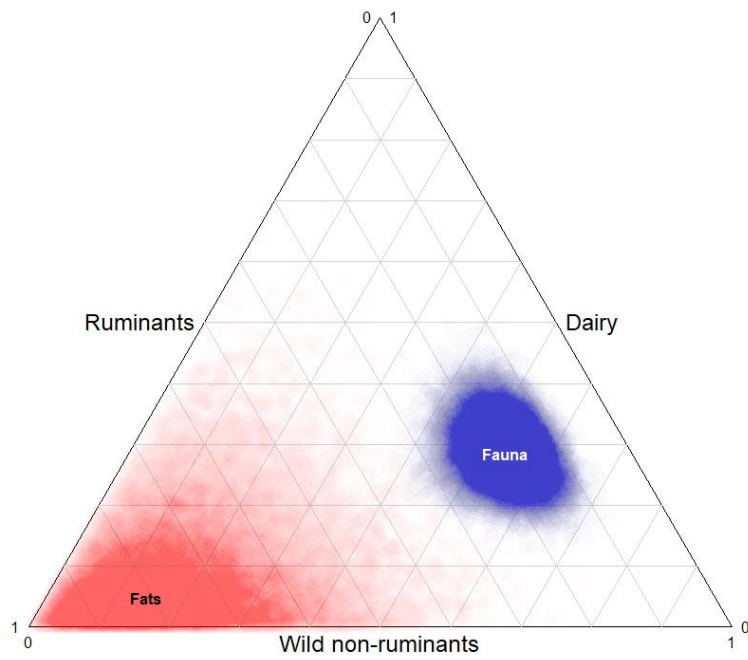


Figure 9. Triangle plot of faunal remain proportion and lipid residue proportions (confidence range represented using random Dirichlet samples of the integer counts in tables 5 and 6 plus a uniform prior) for the faunal remains and lipid residues from INES 6-8

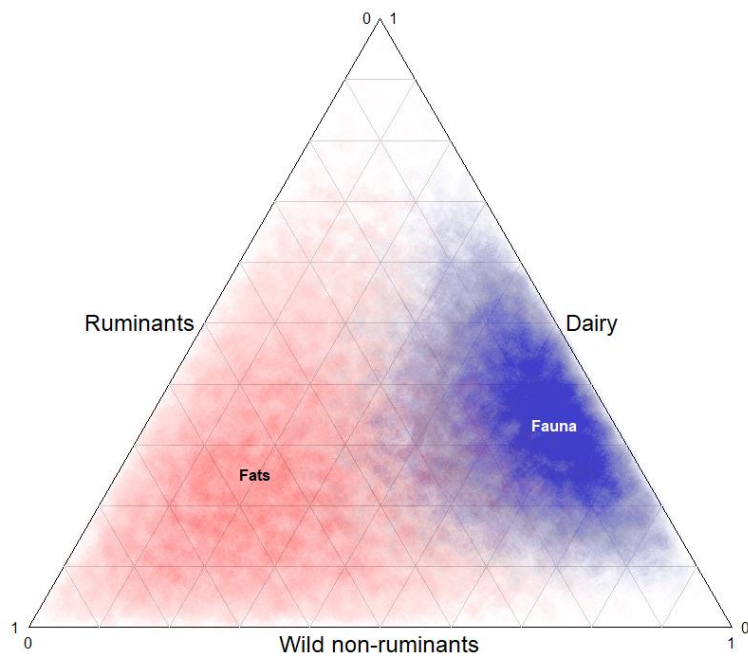


Figure 10. Triangle plot of faunal remain proportion and lipid residue proportions (confidence range represented using random Dirichlet samples of the integer counts in tables 5 and 6 plus a uniform prior) for the faunal remains and lipid residues from IOUD ENC/LN

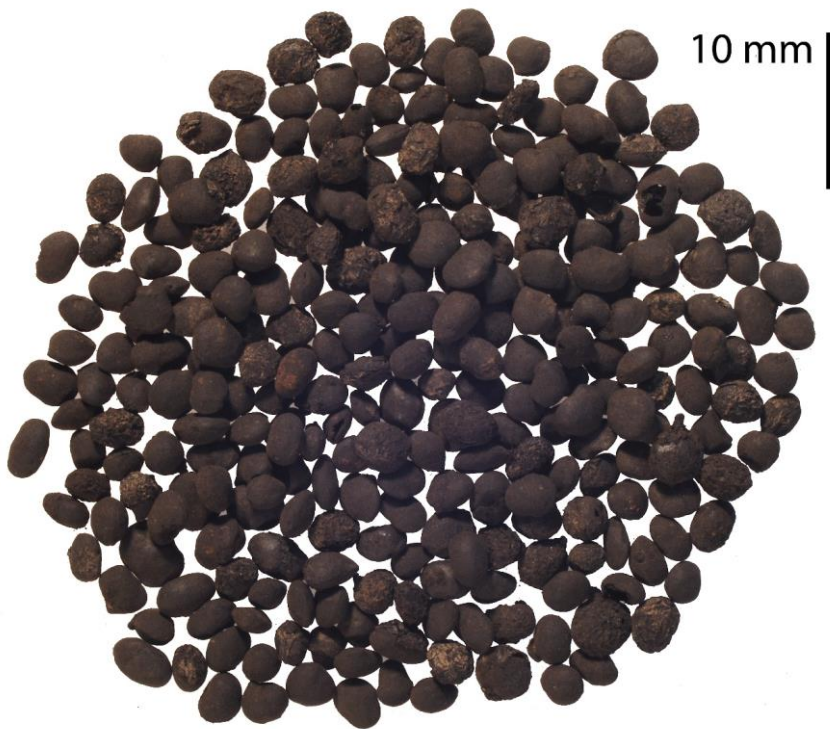


Figure 11. Concentration of *Pistacia lentiscus* seeds from the Neolithic layers of Ifri Oudadane (Photo: J. Morales)

Table 1. Summary of potsherds analysed, organic residues recovered, % lipid recovery, mean lipid concentrations and maximum lipid concentrations

| Site           | Number of potsherds analysed | Organic residues recovered | % Lipid recovery | Mean lipid concentrations mg g <sup>-1</sup> | Maximum lipid concentrations mg g <sup>-1</sup> |
|----------------|------------------------------|----------------------------|------------------|--|---|
| Ifri Oudadane  | 130                          | 19                         | 15               | 3.5  | 46.7  |
| Ifri n'Etsedda | 81                           | 7                          | 9                | 0.3  | 1.3   |
| Hassi Ouenzga  | 95                           | 16                         | 17               | 0.04   | 0.2   |

Table 2. Laboratory number, stratigraphic level, period, lipid concentrations (µg g<sup>-1</sup>), total lipid concentration in extract (µg), δ<sup>13</sup>C and Δ<sup>13</sup>C values and attributions of Ifri n'Etsedda, Hassi Ouenzga and Ifri Oudadane potsherds.

| Sherd number | Stratigraphic level | Period | Lipid                  |                             |                                   | Δ <sup>13</sup> C | Classification |                                   |
|--------------|---------------------|--------|------------------------|-----------------------------|-----------------------------------|-------------------|----------------|-----------------------------------|
|              |                     |        | concentration (ug g-1) | Total lipid in extract (ug) | δ <sup>13</sup> C <sub>16,0</sub> |                   |                | δ <sup>13</sup> C <sub>18,0</sub> |
| ETS229       | INES-6              | ENB    | 108.9                  | 211.2                       | -25.7                             | -27.1             | -1.5           | Ruminant adipose                  |
| ETS232       | INES-6              | ENB    | 63.9                   | 88.8                        | -24.4                             | -26.5             | -2.1           | Ruminant adipose                  |
| ETS252       | INES-7              | ENC    | 282.7                  | 684.2                       | -23.7                             | -25.9             | -2.3           | Ruminant adipose                  |
| ETS268       | INES-8              | LN     | 96.7                   | 304.7                       | -26.8                             | -27.0             | -0.2           | Ruminant/non-ruminant adipose     |
| ETS273       | INES-7              | ENC    | 1317.5                 | 2305.6                      | -25.0                             | -27.5             | -2.5           | Ruminant adipose                  |
| ETS277       | INES-7              | ENC    | 151.3                  | 311.7                       | -25.6                             | -28.3             | -2.7           | Ruminant adipose                  |
| ETS293       | INES-7              | ENC    | 256.5                  | 330.9                       | -23.5                             | -24.6             | -1.1           | Ruminant adipose                  |
| HAS306       | Layer 4             | EN     | 50.0                   | 136.2                       | -23.7                             | -23.4             | 0.3            | Plant/non-ruminant adipose        |
| HAS314       | Layer 4             | EN     | 19.0                   | 86.6                        | -24.7                             | -25.9             | -1.2           | Ruminant adipose                  |
| HAS320       | Layer 3             | EN     | 13.3                   | 33.9                        | -24.1                             | -25.2             | -1.1           | Ruminant adipose                  |
| HAS322       | Layer 3             | EN     | 54.9                   | 76.0                        | -23.7                             | -29.1             | -5.4           | Dairy Fat                         |
| HAS324       | Layer 3             | EN     | 12.6                   | 26.8                        | -24.5                             | -26.3             | -1.8           | Ruminant adipose                  |
| HAS332       | Layer 3             | EN     | 51.8                   | 87.3                        | -23.8                             | -25.0             | -1.2           | Ruminant adipose                  |
| HAS335       | Layer 3             | EN     | 208.5                  | 891.6                       | -24.3                             | -27.6             | -3.3           | Dairy Fat                         |
| HAS336       | Layer 3             | EN     | 26.7                   | 68.3                        | -27.0                             | -29.4             | -2.3           | Ruminant adipose                  |
| HAS339       | Layer 3             | EN     | 74.9                   | 197.2                       | -25.4                             | -26.6             | -1.2           | Ruminant adipose                  |
| HAS350       | Layer 3             | EN     | 9.2                    | 23.9                        | -24.8                             | -26.3             | -1.5           | Ruminant adipose                  |
| HAS353       | Layer 3             | EN     | 55.1                   | 121.2                       | -24.7                             | -25.0             | -0.3           | Ruminant/non-ruminant adipose     |
| HAS357       | Layer 4             | EN     | 22.0                   | 71.8                        | -25.3                             | -26.2             | -0.9           | Ruminant adipose                  |
| HAS369       | Layer 3             | EN     | 31.2                   | 84.2                        | -24.0                             | -25.2             | -1.2           | Ruminant adipose                  |
| HAS378       | Layer 4             | EN     | 33.2                   | 60.4                        | -25.5                             | -25.8             | -0.3           | Ruminant/non-ruminant adipose     |
| HAS387       | Layer 3             | EN     | 8.8                    | 16.7                        | -26.6                             | -28.7             | -2.2           | Ruminant adipose                  |
| HAS394       | Layer 4             | EN     | 32.6                   | 94.9                        | -24.6                             | -25.5             | -0.9           | Ruminant adipose                  |
| OU094        | -                   | ENB    | 116.3                  | 212.8                       | -25.8                             | -26.2             | -0.4           | Ruminant/non-ruminant adipose     |
| OU095        | -                   | ENB    | 141.2                  | 231.6                       | -26.1                             | -24.9             | 1.2            | Non-ruminant adipose              |
| OU098        | -                   | ENB    | 924.2                  | 1866.8                      | -24.1                             | -25.0             | -0.9           | Ruminant adipose                  |
| OU106        | -                   | ENC-LN | 46719.6                | 99980.0                     | -27.1                             | -33.1             | -6.0           | Dairy Fat                         |
| OU112        | -                   | ENB    | 210.5                  | 463.1                       | -23.8                             | -27.5             | -3.6           | Dairy Fat                         |
| OU114        | -                   | ENB    | 956.5                  | 2085.3                      | -24.5                             | -25.3             | -0.8           | Ruminant adipose                  |
| OU118        | -                   | ENB    | 69.6                   | 182.2                       | -23.5                             | -25.5             | -2.0           | Ruminant adipose                  |
| OU144        | -                   | ENB    | 651.0                  | 1061.1                      | -23.1                             | -23.6             | -0.5           | Ruminant/non-ruminant adipose     |
| OU152        | -                   | ENB    | 270.0                  | 704.6                       | -24.1                             | -25.3             | -1.2           | Ruminant adipose                  |
| OU163        | -                   | ENC    | 437.8                  | 1129.4                      | -24.5                             | -25.8             | -1.3           | Ruminant adipose                  |
| OU184        | -                   | ENB    | 267.0                  | 453.9                       | -20.9                             | -21.9             | -1.0           | Ruminant adipose                  |
| OU189        | -                   | ENC-LN | 13064.1                | 33313.3                     | -23.2                             | -24.1             | -1.0           | Ruminant adipose                  |
| OU194        | -                   | ENB    | 450.9                  | 1091.1                      | -23.4                             | -22.7             | 0.7            | Non-ruminant adipose              |
| OU203        | -                   | ENB    | 452.6                  | 923.4                       | -24.0                             | -24.0             | 0.1            | Non-ruminant adipose              |
| OU207        | -                   | ENB    | 105.9                  | 196.0                       | -24.3                             | -25.7             | -1.5           | Ruminant adipose                  |
| OU210        | -                   | ENC    | 135.0                  | 282.1                       | -27.2                             | -27.5             | -0.3           | Ruminant/non-ruminant adipose     |
| OU215        | -                   | ENB    | 272.4                  | 444.0                       | -22.5                             | -21.9             | 0.6            | Non-ruminant adipose              |
| OU217        | -                   | ENB    | 247.4                  | 329.0                       | -25.7                             | -26.7             | -1.0           | Ruminant adipose                  |
| OU222        | -                   | ENB    | 254.8                  | 496.8                       | -26.1                             | -25.8             | 0.3            | Non-ruminant adipose              |

Table 3. Total NISP from all stratigraphic units. The # refers to the faunal categories represented in Figure 6. A=Wild ruminants, B=Wild non-ruminants, C=Dairy animals, D=Other

|  | # | HAS         | OD-<br>ENA | OD -<br>ENB | OD-<br>ENC-LN | INES<br>-5 | INES<br>-6  | INES<br>-7  | INES<br>-8 | INES-<br>9  | INES-<br>10 |
|--|---|-------------|------------|-------------|---------------|------------|-------------|-------------|------------|-------------|-------------|
|  |   | 7.6-<br>6.9 | 7.6-7.3    | 7.1-6.7     | 6.6-5.7       | 7.2        | 6.8-<br>6.5 | 6.6-<br>6.1 | LN         | NEO-<br>DIS | SUB         |
| Pisces indet.  | B | 0           | 579        | 2948        | 248           | 0          | 0           | 0           | 0          | 0           | 0           |
| Amphibia indet.                                      | B | 0           | 0          | 0           | 0             | 3          | 0           | 3           | 0          | 0           | 0           |
| Cheloniidae  | B | 0           | 0          | 7           | 0             | 0          | 0           | 0           | 0          | 0           | 0           |
| <i>Monachus monachus</i>                             | B | 0           | 1          | 1           | 0             | 0          | 0           | 0           | 0          | 0           | 0           |
| <i>Mauremys leprosa</i>                              | B | 0           | 0          | 1           | 0             | 10         | 1           | 18          | 0          | 0           | 1           |
| Reptile indet.                                       | B | 0           | 0          | 0           | 0             | 10         | 10          | 23          | 3          | 4           | 14          |
| <i>Testudo indet.</i>                                | B | 1084        | 24         | 78          | 2             | 14         | 0           | 7           | 1          | 272         | 128         |
| <i>Testudo greaca</i>                                | B | 0           | 0          | 0           | 0             | 2          | 0           | 9           | 0          | 0           | 3           |
| <i>Chameleo sp.</i>                                  | B | 0           | 0          | 0           | 0             | 0          | 0           | 3           | 0          | 0           | 0           |
| Aves indet.  | D | 13          | 18         | 94          | 7             | 3          | 3           | 34          | 1          | 62          | 176         |
| <i>Struthio camelus</i>                              | D | 0           | 0          | 0           | 5             | 2          | 2           | 5           | 1          | 68          | 73          |
| <i>Ammotragus lervia</i>                             | A | 15          | 1          | 2           | 0             | 1          | 0           | 2           | 0          | 10          | 4           |
| <i>Gazella cuvieri</i>                               | A | 0           | 0          | 0           | 0             | 3          | 1           | 8           | 0          | 12          | 22          |
| <i>Gazella sp.</i>                                   | A | 19          | 0          | 4           | 1             | 0          | 0           | 0           | 0          | 0           | 0           |
| <i>Bos primigenius</i>                               | A | 7           | 0          | 0           | 0             | 0          | 0           | 4           | 0          | 1           | 2           |
| <i>Alcelaphus buselaphus</i>                         | A | 4           | 0          | 0           | 0             | 0          | 0           | 0           | 0          | 0           | 2           |
| <i>Capra hircus</i>                                  | C | 4           | 0          | 0           | 0             | 0          | 0           | 0           | 0          | 0           | 0           |
| <i>Ovis aries</i>                                    | C | 14          | 0          | 0           | 0             | 0          | 0           | 0           | 0          | 0           | 0           |
| Ovicaprines  | C | 13          | 1          | 5           | 4             | 6          | 9           | 16          | 0          | 0           | 4           |
| Bovidae indet.                                       |   | 41          | 1          | 19          | 1             | 147        | 112         | 332         | 45         | 732         | 223         |
| <i>Mammalia indet.</i>                               |   | 0           | 165        | 419         | 124           | 15         | 52          | 105         | 4          | 78          | 71          |
| <i>Sus scrofa</i>                                    | B | 2           | 4          | 22          | 5             | 0          | 3           | 0           | 0          | 2           | 4           |
| <i>Equus sp.</i>                                     | B | 4           | 0          | 1           | 0             | 1          | 2           | 1           | 1          | 0           | 0           |
| Rhinocerotidae indet.                                | B | 2           | 0          | 0           | 0             | 0          | 0           | 0           | 0          | 0           | 0           |
| <i>Lepus capensis</i>                                | B | 7           | 0          | 0           | 0             | 0          | 0           | 0           | 0          | 0           | 0           |
| <i>Oryctolagus cuniculus</i>                         | B | 1           | 0          | 0           | 0             | 0          | 0           | 0           | 0          | 0           | 0           |
| Leporidae indet.                                     | B | 6           | 1          | 17          | 0             | 0          | 0           | 1           | 0          | 0           | 2           |
| carnivora indet.                                     | D | 3           | 0          | 0           | 0             | 0          | 0           | 0           | 0          | 0           | 0           |
| <i>Canis lupus familiaris</i>                        | D | 21          | 0          | 0           | 0             | 0          | 0           | 0           | 0          | 0           | 0           |
| <i>Vulpes vulpes</i>                                 | D | 7           | 0          | 0           | 0             | 0          | 1           | 0           | 0          | 0           | 0           |
| <i>Felis silvestris</i>                              | D | 2           | 0          | 0           | 0             | 0          | 0           | 0           | 0          | 0           | 0           |
| <i>Caracal caracal</i>                               | D | 0           | 1          | 0           | 0             | 0          | 0           | 0           | 0          | 0           | 0           |
| <i>Canis aureus</i>                                  | D | 0           | 0          | 1           | 0             | 0          | 0           | 0           | 0          | 0           | 0           |
| <i>Leptailurus serval</i><br><i>/Caracal caracal</i> | D | 1           | 0          | 0           | 0             | 0          | 0           | 0           | 0          | 0           | 0           |
| <i>Panthera leo</i>                                  | D | 0           | 0          | 1           | 0             | 0          | 0           | 0           | 0          | 0           | 0           |
| <i>Atelerix algirus</i>                              | D | 2           | 0          | 2           | 0             | 0          | 0           | 0           | 0          | 0           | 0           |
| <i>Hystrix cristata</i>                              | D | 4           | 1          | 4           | 0             | 0          | 0           | 4           | 3          | 3           | 2           |

Table 4. Chi-square test results

|                      | X-squared | Degrees freedom | P-value   |
|----------------------|-----------|-----------------|-----------|
| Hassi Ouenzga        | 188.7     | 2               | <2.2e-16  |
| Ifri Oudadane ENB    | 40.996    | 2               | 1.253e-09 |
| Ifri Oudadane ENC-LN | 5.3196    | 2               | 0.6996    |
| Ifri N'Etsedda 6-8   | 20.415    | 2               | 3.688e-05 |

Table 5. Fat category proportions from the lipid residues per phase

|                      | Ruminant |      | Non-ruminant |      | Dairy |      |
|----------------------|----------|------|--------------|------|-------|------|
|                      | N        | %    | N            | %    | N     | %    |
| Hassi Ouenzga        | 13       | 72.2 | 3            | 16.7 | 2     | 11.1 |
| Ifri Oudadane ENB    | 9        | 52.9 | 7            | 41.2 | 1     | 5.9  |
| Ifri Oudadane ENC-LN | 3        | 60   | 1            | 20   | 1     | 20   |
| Ifri N'Etsedda 6-8   | 7        | 87.5 | 1            | 12.5 | 0     | 0    |

Table 6. Fat category proportions from the faunal remains per phase

|                      | Ruminant |      | Non-ruminant |      | Dairy |      |
|----------------------|----------|------|--------------|------|-------|------|
|                      | N        | %    | N            | %    | N     | %    |
| Hassi Ouenzga        | 45       | 3.8  | 1112         | 93.6 | 31    | 2.6  |
| Ifri Oudadane ENB    | 6        | 4.4  | 125          | 91.9 | 5     | 3.7  |
| Ifri Oudadane ENC-LN | 1        | 8.3  | 7            | 58.3 | 4     | 33.3 |
| Ifri N'Etsedda 6-8   | 15       | 17.2 | 47           | 54   | 25    | 28.7 |

Table 7.  $\delta^{13}\text{C}$  values of measurable LCFAs, indicative of plant processing, extracted from potsherds from all three sites sampled.

| Sample number | C <sub>20</sub> FA | C <sub>22</sub> FA | C <sub>24</sub> FA | C <sub>26</sub> FA | C <sub>28</sub> FA | C <sub>30</sub> FA |
|---------------|--------------------|--------------------|--------------------|--------------------|--------------------|--------------------|
| HAS303        | -27.4              | -                  | -28.2              | -                  | -                  | -                  |
| HAS306        | -26.8              | -27.8              | -30.7              | -                  | -                  | -                  |
| HAS314        | -26.7              | -27.9              | -28.5              | -29.9              | -28.7              | -28.3              |
| HAS369        | -27.9              | -                  | -                  | -                  | -                  | -                  |
| ETS229        | -29.4              | -29.5              | -30.7              | -30.2              | -30.7              | -31.1              |
| ETS252        | -26.9              | -28.2              | -28.8              | -29.5              | -                  | -                  |
| ETS273        | -27.2              | -28.5              | -                  | -                  | -                  | -                  |
| OU114         | -27.0              | -28.9              | -29.8              | -31.6              | -                  | -                  |
| OU152         | -27.0              | -28.2              | -                  | -                  | -                  | -                  |
| OU215         | -22.1              | -                  | -23.3              | -                  | -                  | -                  |

