OPEN ACCESS

University of BRISTOL

Peer reviewed version

Link to published version (if available):
10.1016/j.quaint.2016.01.005

Link to publication record in Explore Bristol Research

PDF-document

University of Bristol - Explore Bristol Research

General rights

"Middle Holocene hunting and herding at Gueldaman Cave, Algeria: an integrated study of the vertebrate fauna and pottery lipid residues"

Kherbouche, F[1]., Dunne, J[2]., Merzoug, S[1]., Hachi, S[1]. and Evershed, R.P[2].

1. Centre National de Recherches Préhistoriques, Anthropologiqes et Historiques, 3, rue Franklin Roosevelt, 16000 Alger

2. Organic Geochemistry Unit, School of Chemistry, University of Bristol, Cantock's Close, Bristol BS8 1TS, UK

Corresponding author: Dr Julie Dunne, Organic Geochemistry Unit, School of Chemistry, University of Bristol, Cantock's Close, Bristol BS8 1TS, UK, julie.dunne@bristol.ac.uk

Abstract:

Pathways to food production in Holocene north Africa are complex and varied and, for the human groups living there, are likely heavily influenced by varying factors such as local ecosystems and available resources. Molecular and isotopic analysis of absorbed food residues from 140 pottery vessels from Neolithic Gueldaman Cave site confirms that the exploitation of domesticated animals (sheep and goat), for their carcass fats, and their secondary products, e.g. dairy, began in Mediterranean north Africa in the 5th millennium BC. Findings from organic residue analyses are confirmed by the slaughter profiles from the faunal assemblage which suggest a mixed meat/milk economy.

Keywords: Neolithic, Gueldaman, Algeria, Organic residues, Dairying, Carcass fats

1. Introduction

It has long been known that pathways to food production in Africa are complex and varied. In Holocene north Africa, the adoption of domesticates and the existence of pastoralism became an established and widespread way of life long before the domestication of plants, which occurred much later (c. 4000 BP; Gifford-Gonzalez, 2005; Garcea, 2006). Here, it seems likely that the development of subsistence strategies would have been heavily shaped by the unstable, often marginal environments that north African hunter-gatherers lived in. Then, predictable access to resources would have been their major concern, rather than the intensification of yield more applicable to early farmers in the Levant (Marshall and

1

Hildebrand, 2002). The 'patchy spread of food production' in Africa is known, where, in contrast to European prehistory, African hunter-gatherers and food producers (pastoralists, agriculturists) continued to co-exist (Marshall and Hildebrand 2002). It is likely that spatial variation in climatic and environmental conditions, together with availability of food resources, dictated whether managing livestock or hunting, or combinations thereof, took place.

This is demonstrated by the three distinct regions in Holocene north Africa, each of which follows separate pastoral trajectories: 1) Mediterranean north Africa including the Maghreb, 2) The Nile Valley and the adjacent dry hinterlands, and 3) Saharan Africa from west of the Nile to West Africa (Gifford-Gonzalez, 2005). In Saharan Africa, pastoralism spread unevenly from the eastern Sahara to the Acacus (Libya) and Tibesti (Chad) massifs between *c*. 7000 to 5000 cal BP. Early Holocene patterns of plant use persisted and Saharan pastoralists still hunted and fished (Smith, 1980; Gautier, 1987; Marshall and Hildebrand, 2002; Lucarini 2014). In contrast to drying Saharan environments, the Sudanese Nile offered more reliable, fertile resources. Pastoralists utilised large semi-permanent camps such as at Esh Shaheinab and Geili, and domesticates, mainly cattle, were dominant at sites such as Kadero *c*. 5000 to 4000 BP, where wild plants were also intensively exploited (Gautier, 1984; Caneva, 1988; Krzyzaniak, 1991; Haaland, 1992, 1995; Marshall and Hildebrand, 2002).

Significantly, the prehistory of Mediterranean north Africa follows a different trajectory. In recent years several research projects investigating the 'Neolithisation' of this part of north Africa have added significantly to our understanding of the region. However, our knowledge of the cultural processes, and its spatiotemporal extent, leading to the adoption of Neolithic innovations such as the exploitation of domesticates, is still fragmentary. Both the geographic extent and chronology of the Neolithic period in Mediterranean north Africa are not fully understood, primarily due to a sparse and fragmentary archaeological record, with many areas of the region being unexplored (Linstädter, 2008; Lubell *et al.*, 2009; Lucarini 2013).

In the eastern Maghreb, the Capsian period, denoted by broad-spectrum hunting and gathering strategies, ends late, at around 7000 cal BP. The Capsian culture is followed by the Neolithic of Capsian tradition (Néolithique de Tradition Capsienne) although the extent to which Neolithic economic practices (such as herding) are adopted by the later Capsian groups remains unresolved (Roubet, 2001; Rahmani, 2004; Linstädter, 2008). Located in eastern

Algeria and southern Tunisia, in the regions of Constantine, Gafsa and Tebessa, our understanding of the diet and subsistence practices of these groups is limited to the site of Grotte Capéletti in the Aurès Mountains, Algeria. Here, at *c.* 6800 cal BP, the people practiced transhumance as part of a pastoral lifestyle, exploiting domesticated cattle, sheep and goats (Roubet, 2001; Roubet, 2003; Lubell *et al.*, 2009).

The analysis of organic residues absorbed within the fabric of ceramic vessels, using molecular and isotopic techniques, has been shown to be a powerful tool both in the investigation of past diet and subsistence practices and in the reconstruction of animal management practices (e.g. Copley et al., 2003; Craig et al., 2005; Evershed et al., 2008; Outram et al., 2009; Dunne et al., 2012). Organic residue analysis has allowed the identification of terrestrial animal fats as proxies for carcass processing and secondary product exploitation, aquatic products, plant oils and waxes denoting vegetable and plant oil consumption and beeswax, resins, tars and bitumen used in a wide range of technological and cultural activities (e.g. Heron *et al.*, 1994; Dudd and Evershed, 1998; Stern *et al.*, 2003; Hansel *et al.*, 2004; Stern *et al.*, 2008; Cramp *et al.*, 2011; Salque *et al.*, 2013).

Although domesticated animals have been identified in the Mediterranean African Neolithic the inception of dairying practices and the spatiotemporal extent of their exploitation for dairy products is not known. The use of secondary products e.g. milk, blood, wool and traction, which can be obtained from domestic animals through their lifespan, marks an important step in the history of domestication (Sherratt, 1981; 1983) and is of considerable interest in reconstructing past diets as there are major economic and nutritional gains from using these animals for their milk and other products (e.g. Holmes, 1970). Significantly, as well as providing an important source of calories, milk and milk products provide a dependable and renewable source of foodstuff – they are 'lifetime products'.

As discussed, the three distinct pastoral trajectories in Neolithic north Africa are known, despite this, the timing and extent of the inception of dairying practices in north Africa and the development of 'secondary products' economies are still poorly understood in comparison to what we now know of the first appearance of milking in the Near East (Evershed *et al.*, 2008b). Significantly, the exploitation of domesticates for their carcass and dairy products at Takarkori rockshelter, in Saharan Africa, *c.* 7000 cal BP (5th millennium BC), was identified for the first time based on the $\delta^{13}C$ and $\Delta^{13}C$ values of preserved fatty

acids from pottery residues (Dunne et al., 2012; 2013). These findings demonstrated an extensive processing of dairy products in pottery vessels in the Libyan Sahara during the Middle Pastoral period (*ca.* 7200-5800 BP, 5200-3800 BC), suggesting a full pastoral economy as the cattle were intensively exploited for their secondary products. Of note are the range of different forages the animals subsisted on, either composed completely of C_3 plants, varying combinations of C_3 and C_4 plants to a diet comprising wholly C_4 plants, suggested that Saharan pastoralists were practising differing pastoral modes of subsistence during this period (Dunne et al., 2012a; 2013). This insight into the palaeoecology of the region can help us understand the relationships between people and their environment, particularly, as in this instance, over a period of significant climate and environmental change.

2. Gueldaman Cave

The rich and diverse archaeological record uncovered during recent excavations at Gueldaman Cave, Algeria demonstrates its potential as a key site for understanding the north west African Neolithisation process (Kherbouche *et al.*, 2014). This has provided a unique opportunity for a programme of organic residue analysis to apply molecular and isotopic techniques to the analysis of absorbed food residues extracted from Neolithic ceramic vessels from the site. This will help elucidate diet and subsistence practices in the Neolithic period of Mediterranean north Africa, provide insight into the possible inception of dairying practices and contribute to broader understandings of the differing pathways to food production in the region.

2.1 Gueldaman Cave - geographical, geological and historical settings.

Adrar Gueldaman, from the Berber for mountain (Adrar) and devoted to the god of waters (Gueldaman) is an eastern Mediterranean ridge situated in the western Tellian Babors in the Tell Atlas mountains, Algeria. The site of Gueldaman (GLD1) is located on this ridge (Fig. 1A), at 507 m altitude, in a large karst network together with five other caves, two of which, GLD2 and GLD3, also contain deposits of prehistoric origin (Kherbouche *et al.*, 2014).
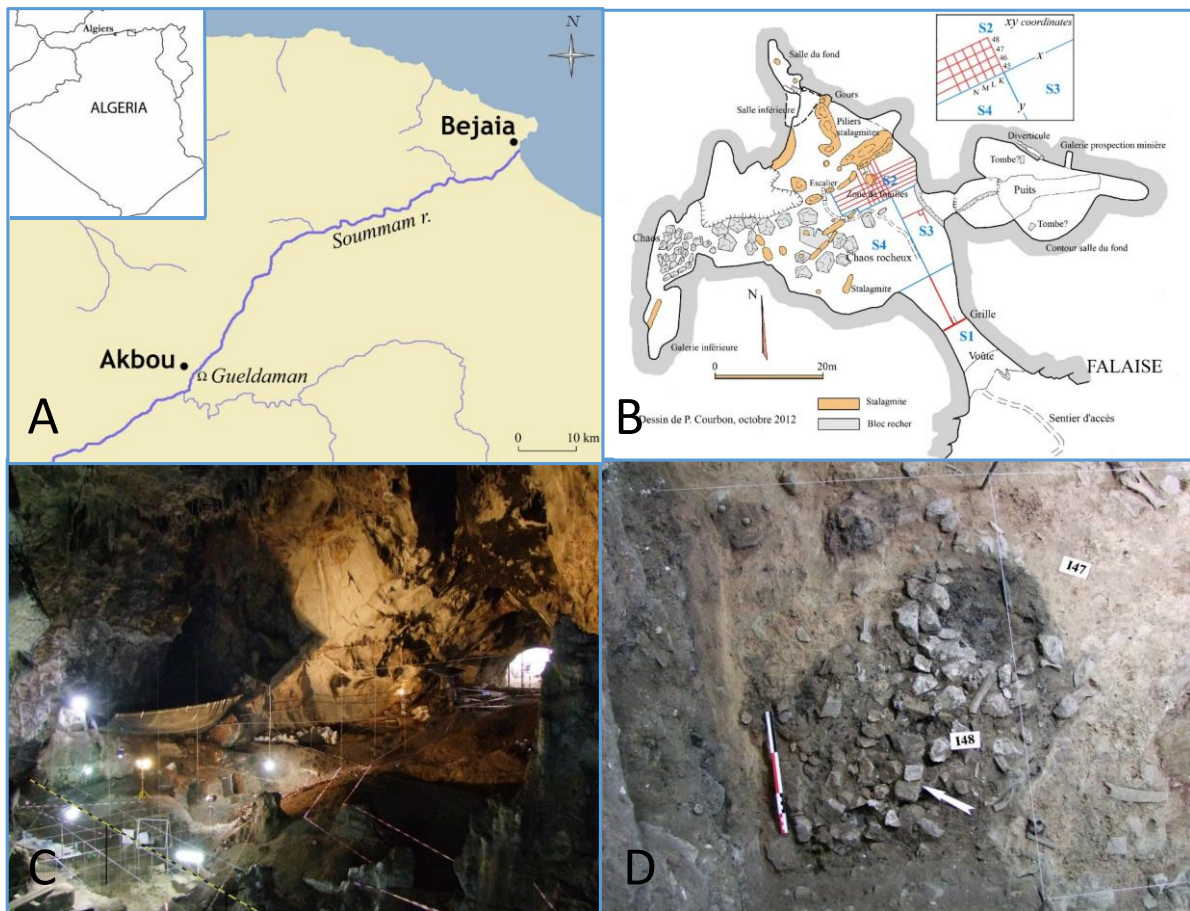
Figure 1. A. Map showing the location of Gueldaman site, Algeria; B. Map of GLD1 cave showing the limits of the 4 sectors (S1-S4) and the XY coordinate system; C. Excavated S2 and S3 areas, a view from the back of the cave (S2) towards the entrance (S1); D. Hearth structure found in layer UA1 (6835 cal BP).

GLD1 was first excavated by de Beaumais and Royer (1926) and mounds of spoil from these excavations remain in situ, mostly alongside their trenches within the cave (Kherbouche *et al.*, 2014). Although not chrono-stratigraphically detailed, de Beaumais and Royer interpreted the site as Early Neolithic through finds of polished bone tools and axes, human and faunal remains, potsherds and some lithics (flint and quartzite). Four seasons of excavations, from 2010 to 2013, together with a full examination of the 1926 spoil heaps, have now been undertaken by the Centre National de Recherches Préhistoriques, Anthropologiques et Historiques (CNRPAH), Algeria.

The GLD1 cave comprises a 6 m semi-circular opening, facing southeast, inside, a 10 m high and 6 m wide dome shaped corridor leads to the main space ("Grande Salle") which is about 80 m long (Fig. 1B). In 1926, de Beaumais and Royer described the maximum depth of

deposit as 5 m; however, excavations in 2010 revealed greater depth with the bedrock still not reached. New excavation focused first on two small areas in sectors S2 and S3 (Fig 1. B and C) but the potential surface area for investigation covers 1000 m², consequently, exploration of a larger horizontal area began in 2013 (Kherbouche *et al.*, 2014).

Large amounts of charcoal were found in the stratigraphical sections of both S2 and S3 areas, particularly in archaeological layers, UA2, UA3 and UA4, suggesting significant anthropogenic activity in the cave (note: correlation between layers and archaeological units is shown in Table 1). The first, well-dated, North African hearth structure of Neolithic date was discovered in S2, at the top of layer UA1, the oldest level at 6835 cal BP (Fig. 1B). It has an almost circular shape surrounding a high and horizontal concentration of calibrated stones on a charcoal bed, possibly suggestive of a type of cooking grill. Several bones were found both inside and outside the structure (Fig. 1D).

Several wood charcoals of 0.5 to 1.5cm length from sections S2 and S3 were radiocarbon dated by the LSCE laboratory in Gif-sur-Yvette, France; dates are shown in Table 1. The chronostratigraphy of the first 12 layers in S2 is known with quite good precision, but it is important to note that layer 13 (UA1) is only dated at its top (at 1.6m) in S3 and not at its bottom which is located around 2.1 m. As a consequence, it is possible that the vestiges found in this layer are older than 6835 cal BP. New radiocarbon dates are underway to ascertain the age of the oldest levels.

Table 1. Radiocarbon dates of wood charcoals for Sector 2 of Gueldaman cave GLD 1.

Sample reference no.	Sector	Square	Depth (cm)	Layer	Archaeological unit	^{14}C BP	Median ^{14}C Cal BP	2σ	1σ
SacA39410		N48	84	7	UA4	4020±30	4494	4420-4569	4411-4785
SacA39411		N48	86	7	UA4	3975±30	4415	4305-4526	4290-4569
SacA39409	S2	M48	91	7	UA4	3945±30	4403	4290-4516	4244-4522
SacA36982		N47	108	9	UA3	4355±30	4918	5032-4851	4960-4863
SacA23883		L48	124	11	UA2	5250±35	6003	5924-6178	5935-6171
SacA23884		L48	132	11	UA2	5260±30	6025	5933-6178	5942-6173
SacA36981		M47	147	12	UA2	6120±35	7002	7157-6907	7154-6937
SacA29727		H34	32	11	UA2	5210±30	5961	5909-6166	5926-5989
SacA29730	S3	F37	90	11	UA2	5280±30	6073	5944-6181	5991-6176
SacA29728		H34	160	13	UA1	5995±40	6835	6739-6941	6759-6890

The archaeological assemblage from these excavations comprised a variety of artefacts, including human (a fragment of a human mandible and two isolated teeth) and faunal

remains, lithic and bone tools, grinding equipment with limonite pigment, ochre, ceramics and ornaments (Kherbouche *et al.*, 2014).

3. The faunal assemblage

The zooarchaeological analyses of the GLD1 faunal assemblage (Table 2) included the invertebrate and vertebrate remains from both the 2011-2012 excavations from Sectors S2 and S3 and the 1920's excavations (found in the spoil heap 2010-2012). This comprised macromammal remains (NISP=2035) from levels dated to the Neolithic (UA1 to UA4). Full details of the zooarchaeological analyses of the GLD1 faunal assemblage can be found in Merzoug et al., this volume.

The faunal assemblage was well-preserved and all skeletal parts were represented. Sheep (*Ovis aries*) and goats (*Capra hircus*), are identified as domestic species according to morphological and metric criteria proposed by Boessneck *et al.* (1964); Payne (1985); Prummel and Frisch (1986); Zeder and Pilaar (2010) and Zeder and Lapham (2010). The distinction between aurochs and domestic cattle is based on metrical criteria in the absence of specific anatomical elements (such as horns or skull bones) from the Bovini remains (e.g. Brugal, 1984-1985; Hadjouis, 1985; Peters, 1986).

 Sheep and goat identified at GLD1 from the earliest level 13 (UA1), dated between 7002 and 6835 cal BP, dominate the assemblage (58.5% of NISP). Both *Bovini* (Aurochs/cattle, 19% of NISP) and *suids* (Boar/pig, 16% of NISP) are also well represented throughout the stratigraphic sequence: wild specimens in early units and domestic specimens in late units for *Bovini*, while suids seems to be wild rather than domestic. The presence of wild species (gazelle, aoudad, hartebeest), throughout the stratigraphic sequence, suggests that hunting was likely still practiced to some extent. Carnivores are extremely rare (0.6% of NISP), represented only by canid species, jackal and red fox. Porcupine and lagomorphs (hare and rabbit) represent 3% of the NISP.

Table 2. Composition of the macromammal assemblage from the 2011-2012 excavation (GLD 1 - Sectors S2 and S3): NISP= Number of Identified Specimens per taxon.

Archaeological levels		UA1		UA2		UA3		UA4			
Taxon		NISP	%	NISP	%	NISP	%	NISP	%	Total	%NISP
Bos primigenius/Bos taurus	Aurochs/cattle	42	12.84	180	27.65	126	30.88	42	6.47	390	19.16
Ovis aries/Capra hircus	Sheep/Goat	205	62.69	291	44.70	170	41.67	524	80.74	1190	58.48
Sus scrofa/sus domesticus	Boar/pig	64	19.57	128	19.66	90	22.06	40	6.16	322	15.82
Alcelaphus buselaphus	Hartebeest	2	0.61	2	0.31	3	0.74	0	0.00	7	0.34
Gazella sp.	Gazelle	1	0.31	8	1.23	5	1.23	5	0.77	19	0.93
Ammotragus lervia	Barbary Sheep	3	0.92	8	1.23	3	0.74	22	3.39	36	1.77
Canis aureus	Golden jackal	1	0.31	5	0.77	1	0.25	1	0.15	8	0.39
Vulpes vulpes	Red fox	2	0.61	1	0.15	1	0.25	0	0.00	4	0.20
Hystrix cristata	Porcupine	0	0.00	0	0.00	3	0.74	0	0.00	3	0.15
Lepus capensis/Oryctolagus cuniculus	Hare/rabbit	7	2.14	28	4.30	6	1.47	15	2.31	56	2.75
NISP		327		651		408		649		2035	100

The age at death of the animals was recorded through the stages of eruption, replacement and wear of teeth on mandibles (Payne, 1973; 1987; Helmer and Vigne, 2004). The age at which domestic animals are slaughtered reflects the relative value placed on the different products that can be extracted from the animal (meat, milk, wool). For example, in studying modern sheep herds in Turkey, Payne (1973) established that meat production required massive slaughtering of young and sub-adult males between 6-18 months, whilst specialised milk production was characterised by the killing of around 50% of very young lambs (before the age of two months). However, it should be noted that the study of Neolithic culling profiles demonstrated that most are of mixed origin, e.g. meat and milk (Helmer, 1992; Helmer and Vigne, 2004). Consequently, the age profile of a faunal assemblage from an archaeological site provides information on herding strategies and animal husbandry practice and can be a powerful tool in detecting milking strategies in archaeological populations.
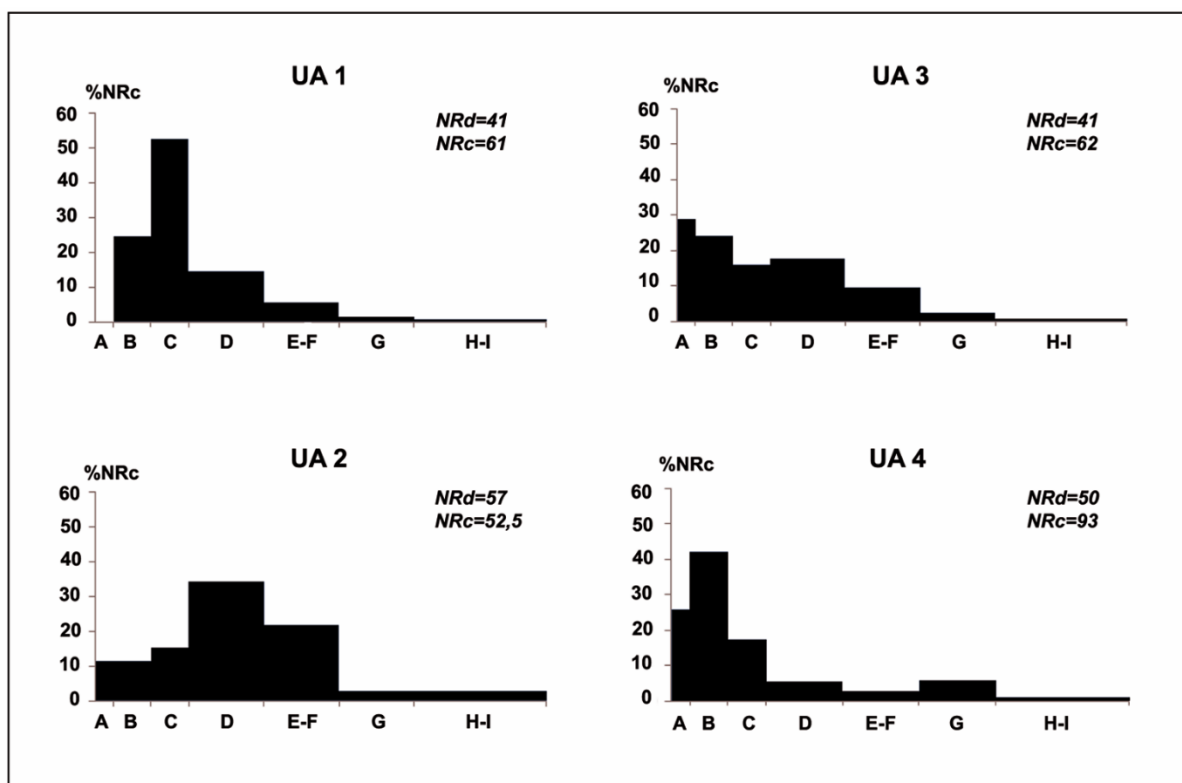
Figure 2. Age mortality profiles of sheep/goat from Neolithic-dated units UA1 to UA4 (NRd = Number of dental remains; NRc=Number of corrected dental remains, A=0-2 months, B=2-6 months, C=6-12 months, D=1-2 years, E-F=2-4 years, G=4-6 years, H-I= > 6 years).

The age mortality profiles from archaeological units UA1 to UA4 are shown in Figure 2. The profile in UA1 (the earliest levels at 7002-6835 cal BP) demonstrates a large slaughtering peak of the age class C (6-12 months), suggesting tender meat production. This type of age mortality profile is similar to profiles from French Neolithic sites such as La Balance (Helmer and Vigne, 2004). In contrast, the age mortality profiles from archaeological units UA2 to UA4 (6300-4403 cal BP) reflect a mixed production of meat and secondary products characterised by the culling of more than 50% of very young lambs (class A) and a massive slaughtering of age classes B to D for tender meat. The age peak showing high incidences of slaughter of sheep/goat at 4-6 years is also suggestive of the mixed milk-meat model, characterised by the slaughter of old milk-producing females (Vigne and Helmer, 2007). The age mortality profile from UA2 to UA4 corresponds with the management practices of sheep and goat herds from Neolithic sites in southern France and Italy and final PPNB sites in the Levant which show both mixed (meat-milk) production and an increase in fleece use between the early and late Neolithic (Vigne and Helmer, 1999; Helmer et al., 2007).

9

In summary, the faunal assemblage suggests a mixed economy dominated by the husbandry of sheep and goats, with some hunting taking place. The age mortality profile from the earliest level (UA1) suggest that the earliest domesticates were mainly exploited for their meat; with milk production increasingly becoming an important component of their subsistence practices in levels UA2 to UA4, demonstrated by the high abundances of Class A (0-2 months) animals killed. The cull of high numbers of Classes B-D animals that provide tender meat suggests a mixed meat/milk economy. This was likely supplemented by the snaring and collecting of a variety of small animals and, presumably, gathering of plants, such as wild grains, fruits, and nuts. Analysis of charcoal, fruits and seeds from the site are underway and will be published separately.

4. The ceramic assemblage

A total of 1050 potsherds were recovered from the 2010-2012 excavations. These were all handmade using the coiling technique with the addition of mineral and organic components as tempering agents. Examples of all morphological parts of vessels were present; conical and hemispheric bottoms (Fig. 3), handle elements of different types, curved rims and body sherds. The majority derive from what appear to be cooking vessels of 20-30 cm rim diameter (Kherbouche *et al.*, 2014). A significant number of vessels (68%) are decorated, with impression or incision techniques, including comb impression, half-circle notches, horizontal rows of dots and incised crossed lines, although only on the upper parts of pots and in the rim zones.
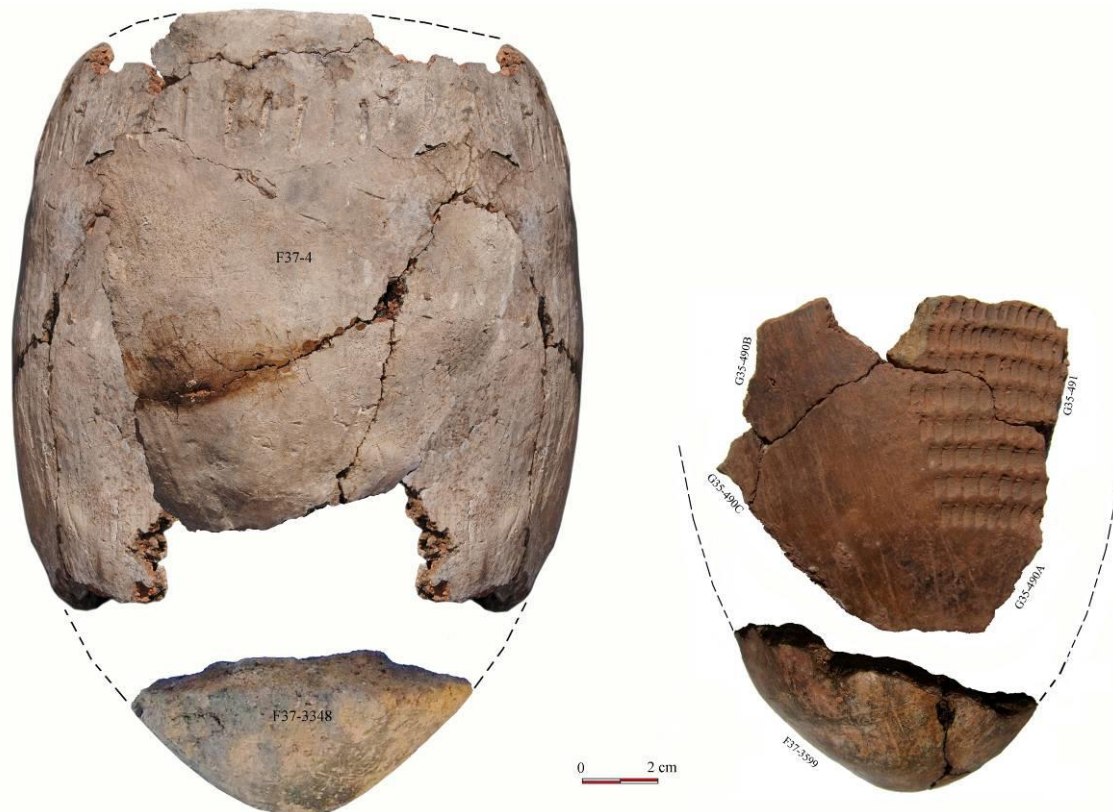
Figure 3. Different types of partly reassembled pots from levels UA1 (right) and UA2 (left)

Most of the assemblage originates from Neolithic occupation levels covering the 6th to the 7th millennia BP (fifth millennium BC; see Figure 4 for vertical distribution of potsherds). Kherbouche *et al.* (2014) suggest that the strong relationship between charcoal and ceramic concentrations (mainly located in Section 2, between levels UA1 to UA5; Fig. 4) should be interpreted as evidence for cooking activity.

4.1 Organic residue analysis - results

A total of 140 potsherds, from 2012 (*n*=62) and 2013 (*n*=78) excavations, were investigated according to well-established analytical procedures described in the 'Material and Methods' section (e.g. Dudd *et al.*, 1999; Copley *et al.*, 2003b; Evershed *et al.*, 2008b). The potsherds were recovered from S2 (layers UA2 and UA3) and S3 (layer UA1). The spatial projection of the S2 sample positions in vertical plan XZ is shown in Figure 4.
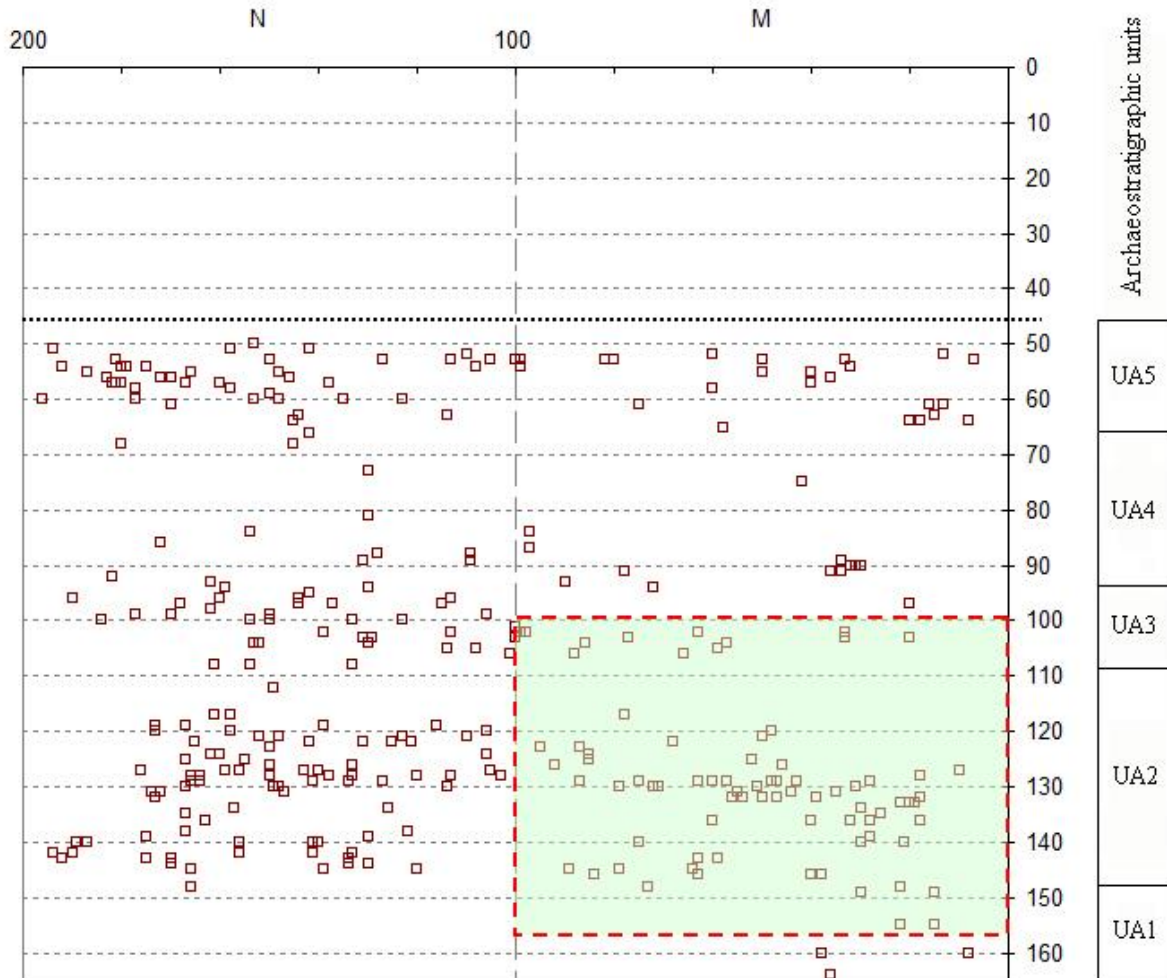
Figure 4. Vertical distribution of potsherds found in Sector 2- Zone MN47/48. The points are a geometric projection in the transverse section 47/48. The potsherds selected for residue analysis are shown inside the rectangle with dotted borders. Archaeostratigraphic units are shown alongside.

Total lipid extracts (TLEs) were recovered from *c*. 20% of the potsherds from the 2012 excavations and 26% from the 2013 assemblage. The rate of lipid recovery is low in comparison to ceramics analysed from the Libyan Sahara (94%; Dunne *et al.*, 2012a), which likely reflects the more favourable preservation conditions of arid environments. The mean lipid concentration of the sherds was 139.1 µg g^{-1}, with a maximum lipid concentration of 617.6 µg g^{-1} (Table 3).

To date, analysis of the TLE's from the Gueldaman cave with sufficient concentrations of lipid (*n*=30) demonstrates that the free fatty acids, palmitic ($C_{16:0}$) and stearic ($C_{18:0}$) are the most abundant components. Odd numbered fatty acids ($C_{15:0}$ and $C_{17:0}$) are also present, possibly indicative of a bacterial origin, resulting from microbial activity in the rumen,

characteristic of a ruminant fats origin, although no branched chain fatty acids were present. TAGs and their degradation products (DAGs and MAGs) were observed in 50% of the residues ($n=15$), within the range C_{48} to C_{54}, usually dominated by C_{52} (Figs. 5 and 6). Lower molecular weight TAGs (C_{42} to C_{46}) which characterise dairy fats were absent (Dudd and Evershed, 1998).
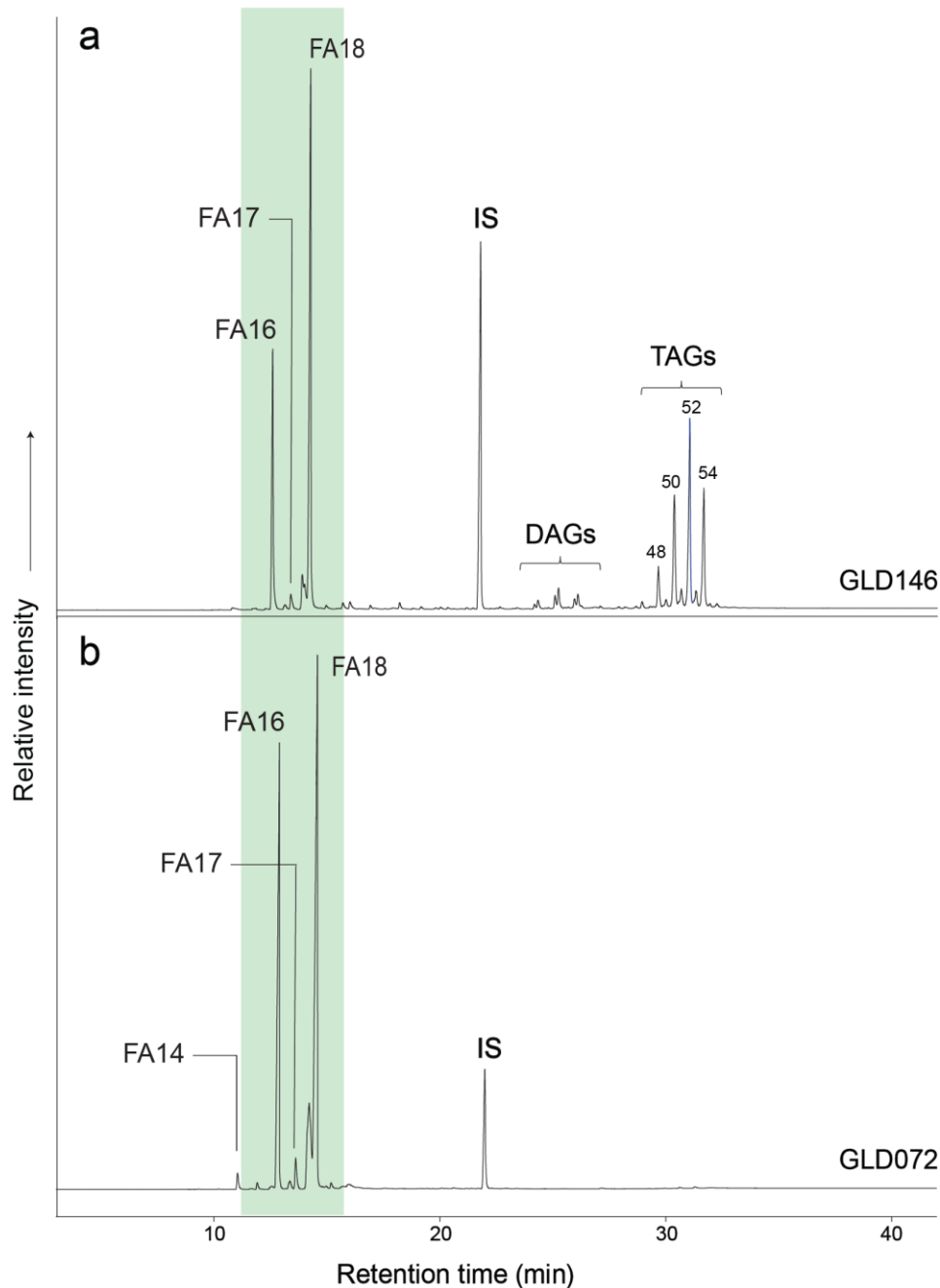

Figure. 5. Partial gas chromatograms of trimethylsilylated TLEs from Gueldaman, Algeria, a) well-preserved animal fat (GLD146) showing TAGs and DAGs, and b) degraded animal fat

(GLD072). FAx are fatty acids where x is the carbon chain length; DAG, diacylglycerols; TAG, triacylglycerols; IS, internal standard, *n*-tetratriacontane (*n*-C34).
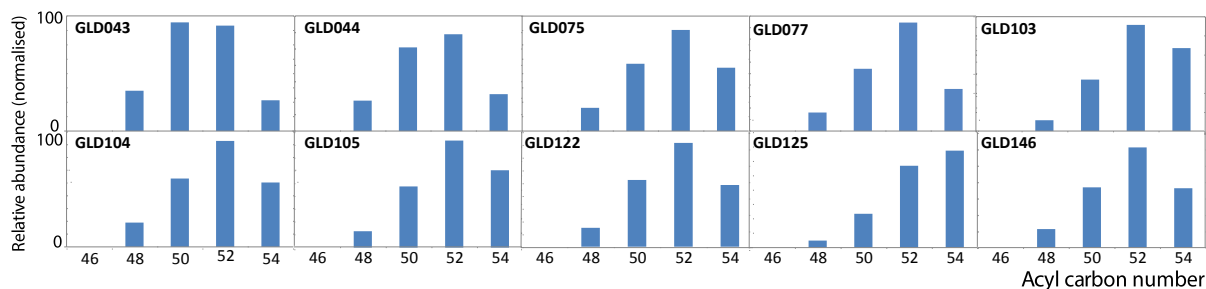

Figure. 6. Histogram showing TAG distributions present in the TLEs extracted from Gueldaman potsherds. Acyl carbon number distributions range from C48 to C54, usually maximising at C52. The histograms are normalised to the most abundant homologue.

In one vessel (GLD058), odd carbon number ketones were present ($C_{31:0}$, $C_{33:0}$ and $C_{35:0}$) in extremely high abundances, in fact, total lipid concentration in the extract totalled more than 2 mg g^{-1}, the highest concentration in the entire assemblage. Experimental work has shown these ketones, present in a monomodal distribution, originate from the pyrolysis of acyl lipids via ketonic decarboxylation reactions, which occur in unglazed ceramic vessels during cooking when the temperature exceeds 300° C. These ketones are thought to accumulate gradually with repeated use (Evershed *et al.*, 1995; Raven *et al.*, 1997), suggesting that vessel GLD058 was regularly used to cook foods at high temperatures.

GC-C-IRMS analyses were carried out on 28 TLEs to determine the $\delta^{13}C$ values of the major fatty acids, $C_{16:0}$ and $C_{18:0}$, and ascertain the source of the animal fats extracted from GLD residues through the use of the $\Delta^{13}C$ proxy (Fig. 7 and Table 3). The $\delta^{13}C_{16:0}$ values of the fatty acids range from -28.3 to -22.4 ‰ and the $\delta^{13}C_{18:0}$ values range from -33.2 to -25.5 ‰. In comparison to $\delta^{13}C_{16:0}$ values for modern British and African reference fats (Copley *et al.*, 2003b; Dunne *et al.*, 2012a), the $\delta^{13}C_{16:0}$ values exhibit differences of -0.1 to -5.9 ‰ (African reference fats) and -2.6 and -0.3 to -8.5 ‰ and -6.2 ‰, respectively (British reference fats). These values suggest that the animals producing these fats were subsisting on a diet comprising mainly C_3 plants but with some C_4 input. As the mean $\delta^{13}C_{16:0}$ and $\delta^{13}C_{18:0}$ values are more enriched by *c*. 3 to 4 ‰, respectively, in comparison to modern British reference fats, this confirms the addition of some C_4 plants. Similar enrichment was observed in $\delta^{13}C$ values of lipids extracted from Near Eastern archaeological pottery (Evershed *et al.*, 2008b) where it was hypothesised to result from the animals producing the fats consuming a

14

proportion of C_4 plants in their diets and/or water-stressed C_3 plants (Farquhar *et al.*, 1989; Dunne *et al.*, 2012a).

Table 3. Lipid concentrations (µg g^{-1}), total lipid concentrations in extracts (µg), $\delta^{13}C_{16:0}$, $\delta^{13}C_{18:0}$, $\Delta^{13}C$ values, lipid composition and classification of residues from potsherds from Gueldaman, Algeria.

Archaeological Unit	Lab Number	Sample No.	Lipid concentration (ug g^{-1})	Total lipid in extract (ug)	$\delta^{13}C_{16:0}$	$\delta^{13}C_{18:0}$	$\Delta^{13}C$	Lipid composition	Classification
UA2	GLD002	653	n/d	n/d	n/d	n/d	n/d	FFA, AL, OH, W, HW	Beeswax
UA3	GLD032	184	255.2	533.3	-27.0	-27.6	-0.5	FFAs (C_{16} - C_{18}), TAGs	Mixed ruminant and non-ruminant adipose
UA2	GLD035	749	11.3	16.2	-27.3	-33.2	-5.9	FFAs (C_{16} - C_{18})	Dairy fat
UA2	GLD038	695	131.3	137.9	-26.7	-31.7	-5.0	FFAs (C_{16} - C_{18})	Dairy fat
UA2	GLD039	614	57.6	109.5	-28.0	-29.8	-1.9	FFAs (C_{16} - C_{18})	Ruminant adipose
UA2	GLD041	616	243.7	638.4	-27.1	-26.8	0.2	FFAs (C_{16} - C_{18})	Non-ruminant adipose
UA2	GLD043	402	134.6	247.7	-28.1	-26.9	1.2	FFAs (C_{14} - C_{18}), DAGs, TAGs	Non-ruminant adipose
UA1	GLD044	834	118.6	240.8	-28.3	-28.4	-0.1	FFAs (C_{16} - C_{18}), TAGs	Mixed ruminant and non-ruminant adipose
UA3	GLD049	207	410.9	653.4	n/d	n/d	n/d	FFAs (C_{16} - C_{18})	n/d
UA1	GLD051	812	37.6	68.8	n/d	n/d	n/d	FFAs (C_{16} - C_{18})	n/d
UA2	GLD056	658	26.7	73.8	-26.3	-30.6	-4.3	FFAs (C_{16} - C_{18})	Dairy fat
UA3	GLD058	205	617.6	2278.9	n/d	n/d	n/d	FFAs (C_{16} - C_{18}), Ketones	n/d
UA2	GLD062	659	16.0	21.7	-27.3	-30.5	-3.2	FFAs (C_{16} - C_{18}), TAGs	Mixed dairy and adipose fat
UA1	GLD072	7	194.0	484.9	-26.9	-27.7	-0.8	FFAs (C_{14} - C_{18})	Ruminant adipose
UA1	GLD075	148	97.0	324.0	-27.6	-28.8	-1.2	FFAs (C_{14} - C_{18}), DAGs, TAGs	Ruminant adipose
UA1	GLD077	6	101.6	378.0	-27.5	-28.8	-1.2	FFAs (C_{16} - C_{18}), DAGs, TAGs	Ruminant adipose
UA1	GLD079	59	195.7	460.0	-27.4	-28.4	-1.0	FFAs (C_{14} - C_{18})	Ruminant adipose
UA1	GLD081	12	5.7	15.0	-26.9	-27.6	-0.7	FFAs (C_{16} - C_{18}), TAGs	Ruminant adipose
UA1	GLD089	348	12.2	41.1	-22.4	-25.5	-3.2	FFAs (C_{16} - C_{20})	Mixed dairy and adipose fat
UA1	GLD091	347	155.4	529.8	-27.8	-28.3	-0.5	FFAs (C_{16} - C_{18}), DAGs, TAGs	Mixed ruminant and non-ruminant adipose
UA1	GLD098	262	6.5	14.1	-25.9	-27.9	-2.0	FFAs (C_{16} - C_{18}), TAGs	Ruminant adipose
UA1	GLD103	106	50.2	152.7	-27.0	-28.7	-1.7	FFAs (C_{16} - C_{18}), DAGs, TAGs	Ruminant adipose
UA1	GLD104	30	65.4	149.2	-26.3	-28.3	-2.0	FFAs (C_{16} - C_{18}), TAGs	Ruminant adipose
UA1	GLD105	46	133.6	179.0	-26.4	-28.1	-1.7	FFAs (C_{16} - C_{18}), DAGs, TAGs	Ruminant adipose
UA1	GLD112	124	43.5	105.3	-27.4	-31.5	-4.1	FFAs (C_{14} - C_{18}), TAGs	Dairy fat
UA1	GLD114	72	80.4	136.7	-25.5	-27.0	-1.5	FFAs (C_{16} - C_{18})	Ruminant adipose
UA1	GLD118	167	600.7	1958.2	-26.7	-28.5	-1.8	FFAs (C_{16} - C_{18}), DAGs, TAGs	Ruminant adipose
UA1	GLD121	180	21.2	67.6	-26.2	-27.4	-1.2	FFAs (C_{16} - C_{18}), DAGs, TAGs	Ruminant adipose
UA1	GLD122	60	286.0	689.2	-26.9	-27.3	-0.4	FFAs (C_{14} - C_{18}), DAGs, TAGs	Mixed ruminant and non-ruminant adipose
UA1	GLD125	103	143.0	326.0	-27.9	-28.9	-1.0	FFAs (C_{16} - C_{18}), DAGs, TAGs	Ruminant adipose
UA1	GLD130	205	32.3	82.5	-25.1	-25.5	-0.4	FFAs (C_{16} - C_{18}), DAGs, TAGs	Mixed ruminant and non-ruminant adipose
UA1	GLD146	169	37.5	78.7	-24.7	-27.3	-2.6	FFAs (C_{16} - C_{18}), DAGs, TAGs	Ruminant adipose

The $\Delta^{13}C$ values show that four TLEs (14%, Table 3) can be unambiguously assigned to a ruminant dairy fat origin, 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_3 diet in Britain and C_3/C_4 diets in Africa (Copley *et al.*, 2003b; Dunne *et al.*, 2012a). Two residues, GLD062 and GLD089, with $\Delta^{13}C$ values of -3.2 and -3.2 ‰ respectively, plot close to the border between ruminant dairy and ruminant adipose values (Fig. 7c and d). Ruminant dairy fats are differentiated from ruminant adipose fats when they display $\Delta^{13}C$ values of less than -3.1 ‰, known as the universal proxy (Dunne *et al.*, 2012a; Salque, 2012). Archaeological

animal fats which plot close to the -3.1 ‰ boundary should not be firmly interpreted as ruminant dairy or adipose fats due to the slight overlap between the observed $\Delta^{13}C$ values of ruminant carcass and milk fats in experimental work (Salque, 2012). Hence, it is likely that some mixing of dairy and adipose fats occurred in these vessels. A further 19 residues (68%) plot within the range for ruminant adipose fat, with another 3 residues plotting in the non-ruminant adipose fat range. Several of these cluster around the borderline between ruminant and non-ruminant adipose fat making attributions difficult, again suggesting some mixing of fats. Consequently, a total of four residues were attributed to a dairy fat origin and 14 were assigned to a ruminant adipose origin (Fig. 7). Two TLE's (GLD041 and GLD043) unambiguously originate from non-ruminant animal fats, suggesting that minor amounts of animal products originating from wild fauna were processed in the vessels. These potsherds originate from levels UA2 and 3 (Fig 7d).
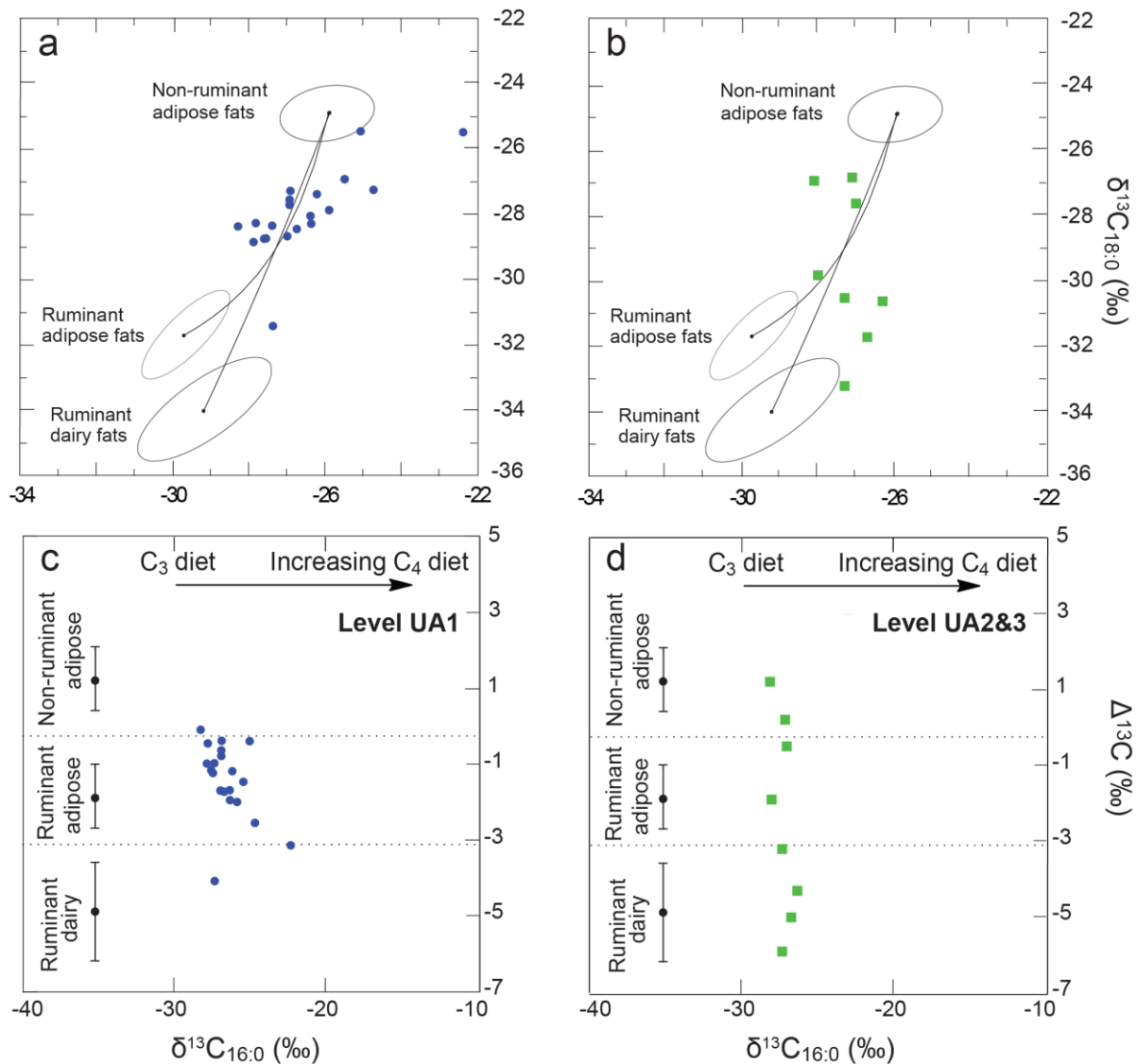
Figure 7. Graphs showing: **a** and **b**. $\delta^{13}C$ values for the $C_{16:0}$ and $C_{18:0}$ fatty acids for archaeological fats extracted from level UA1 GLD1 ceramics (circles) and levels UA2 and 3 (squares). The three fields correspond to the $P = 0.684$ confidence ellipses for animals raised on a strict C_3 diet in Britain (Copley et al., 2003). Each data point represents an individual vessel. **c** and **d** show the $\Delta^{13}C$ ($\delta^{13}C18:0 - \delta^{13}C16:0$) values from the same potsherds. The ranges shown here represent the mean $\pm$ 1 s.d. of the $\Delta^{13}C$ values for a global database comprising modern reference animal fats from Africa (Dunne *et al.*, 2012), UK (animals raised on a pure C3 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.

5. Discussion and conclusion

This project provided a unique opportunity to use the technique of organic residue analysis to address questions regarding the diet and subsistence strategies of Neolithic pastoralists in Mediterranean north Africa and also contribute insights to the spatiotemporal extent of the exploitation of domesticates for their carcass and dairy fats in the region, leading to greater understanding of pathways to food production in Holocene Africa.

Molecular and isotopic analysis of absorbed food residues from vessels from Neolithic Gueldaman Cave site confirms that the majority of animal products processed in the vessels originated from ruminant animals, i.e. cattle, sheep or goat. Most of the residues could be unambiguously assigned to a predominantly ruminant carcass fat origin, although dairy products were also identified, confirming that the exploitation of domesticated animals and their secondary products was taking place at this Mediterranean north African site, dated to the Neolithic, at *c*. 7000 yrs BP (fifth millennium BC). These results confirm that, despite separate pastoral trajectories and different prevailing palaeoecological conditions, the exploitation of milk and milk products occurs contemporaneously (7000 yrs BP, fifth millennium BC) in both Mediterranean and Saharan north Africa. However, whereas sheep and goat seem to appear first at Gueldaman Cave, cattle were the first domesticates in Saharan Africa, followed by sheep and goat once the region became more arid.

Organic residue analysis can be a powerful proxy both for the mobility of past populations and in discerning the past 'isoscapes' they inhabited (West *et al.*, 2010; Dunne *et al.*, 2012a). The foods that animals eat exhibit characteristic isotopic signatures (Gannes *et al.*, 1997) and isotopic analyses ($\delta^{13}C$) of fatty acids extracted from archaeological potsherds are thus a

reflection of the consumed diet and thus can provide information about the environment in which the animals foraged (Copley *et al.*, 2003a; Mukherjee *et al.*, 2005). The analyses of modern reference fats collected from non-ruminant animals (meat) and ruminant animals (meat and milk) has demonstrated the wide range of $\delta^{13}C$ values observed in fatty acids as a result of inhabiting diverse isoscapes (Dunne *et al.*, 2012b). At Gueldaman Cave, the range of $\delta^{13}C_{16:0}$ values exhibited by the fatty acids suggests that the animals producing these fats were subsisting on a diet comprising mainly C_3 plants with a significant C_4 and/or aridity influence on the diets of the animals. This implies that the herders and their animals were possibly living a relatively settled lifestyle, with no long distance transhumant movements, perhaps due to them inhabiting a relatively stable ecosystem. This is in contrast to the Neolithic period in the Sahara, where climatic and environmental conditions are known to have been more unpredictable (e.g. deMenocal *et al.*, 2000), leading to the adoption of transhumant pastoralism, moving seasonally to maximise water availability, as a subsistence strategy. These differing pastoral trajectories reflect the geographically distinct ecological conditions prevailing in the different regions and are also likely influenced by local demographic and cultural influences.

Lipid residue analyses and archaeozoological studies can be used as complementary pieces of evidence regarding dietary practices, herding strategies and, on occasion, species-specificity. For example, a study conducted by Evershed and coworkers (2008), comprising the analyses of more than 2,200 sherds in the Near East and southestern Europe, found milk residues were detected in highest numbers from sites where cattle were the predominant species in the faunal assemblage, implying that cattle were the primary producers of milk. At Gueldaman, in contrast to the Saharan Neolithic, where poor preservation of animal bone precluded herd reconstructions, the identification of lipid residues demonstrating animal products processing can be directly correlated with the archaeozoological assemblage. The faunal assemblage is dominated by sheep and goat (58.5% of NISP), with some cattle (19% of NISP) and pig (16% of NISP) being present. The *Bovini* seem to be wild (aurochs) in early units and domestic specimens in late units, while suids seems to be wild rather than domesticated throughout. As the majority of the residues comprise ruminant carcass and dairy fats, it seems likely that these mostly originate from sheep and goat, the dominant species at the site. It is interesting that small numbers of wild fauna (such as antelope, Barbary sheep) were present in the assemblage, which might suggest hunting remained a (minor) part of the subsistence strategy

of the people. This corresponds well with the lipid residue results, as only two of the vessels sampled were used to process non-ruminant animal fats (7%). These might also originate from wild boar.

The age mortality profile from the first identified level (UA1) suggests that the earliest domesticates were mainly exploited for their meat; with milk production increasingly becoming an important component of their subsistence practices in levels UA2 to UA4, demonstrated by the high abundances of Class A (0-2 months) animals killed. The cull of high numbers of Classes B-D animals in the same period (which provide tender meat) suggests a mixed meat/milk economy. This correlates well with the lipid profiles as, in the earliest level (UA1, Fig. 7c) where a meat economy predominates, the lipid residue analyses reflect an extremely high incidence of ruminant carcass product processing in the vessels. Milk and its products are also processed (5%), suggesting that secondary products are exploited from the earliest introduction of domesticates, albeit in low abundances. Significantly, in levels UA2 and UA3 (Fig. 7d), where a mixed meat/milk economy is well-established, the processing of milk becomes more commonplace, with 38% of the residues being of dairy fat origin. It should also be noted that in two of the samples the $\Delta^{13}C$ values of the fatty acids denote some mixing of carcass fats and milk products in the vessels, making attributions difficult, thus it is also possible that the dairy product processing at the site is under-represented. Interestingly, the $\Delta^{13}C$ values show that the two incidences of wild fauna processing occur in these later levels, perhaps implying a return to some hunting practices after the early adoption (and total reliance) on domesticates as a source of meat.

In conclusion, analysis of organic residues absorbed within the fabric of ceramic vessels, using molecular and isotopic techniques, has unequivocally established the importance of domesticates, both for their carcass and milk products, in Neolithic levels at Gueldaman Cave, Algeria. In addition, this paper helps to further elucidate the development of animal husbandry practises in Holocene north Africa, confirming that pathways to food production in the region were complex, likely depending on varying factors such as local environments and resources.

5300 words

Acknowledgements

Material and Methods

Lipid analysis and interpretations were performed using established protocols described in detail in earlier publications (Dudd and Evershed, 1998; Copley *et al.*, 2003b; Evershed *et al.*, 2008b). Briefly, ~2 g of potsherd were sampled and surfaces cleaned with a modelling drill to remove exogenous lipids. Cleaned sherd fragments were crushed in a solvent-washed mortar and pestle and an internal standard was added (20 µg *n*-tetratriacontane) prior to solvent extraction using 2 x 10 ml $CHCl_3$/MeOH 2:1 *v/v*, *via* sonication (20 min). After centrifugation the solvent was decanted and evaporated under a gentle stream of N_2 to obtain the total lipid extract (TLE). Aliquots of the TLE were filtered through a silica column and treated with *N,O*-bis(trimethylsilyl)trifluoroacetamide (BSTFA, 40 µl, 70° C, 60 min). Excess BSTFA was evaporated under N_2, and the derivatives were dissolved in hexane and analysed via high-temperature gas chromatography using a gas chromatograph (GC) fitted with a high temperature 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 followed by an increase to 350° at a rate of 10° min^{-1} followed by a 10 min isothermal. A procedural blank (no sample) was prepared and analysed alongside every batch of samples.

Further compound identification was accomplished using gas chromatography-mass spectrometry (GC-MS). Aliquots from selected TLEs were hydrolysed (0.5 M NaOH/MeOH; 70°C, 1 h) followed by acidification to pH 3 using 1 M aqueous HCl and the extraction of free fatty acids into 3 x 3 ml $CHCl_3$. Fatty acid methyl esters (FAMEs) were methylated with BF_3-methanol (14% *w/v*, 70°C, 1 h) and extracted (3 x 2 ml $CHCl_3$) and the solvent removed under nitrogen. 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° for 2 min, ramping to 300° at 10° min^{-1}, followed by an isothermal hold at 300° (15 min).

The carbon isotopic composition of individual fatty acids was determined using GC-combustion-isotope ratio MS (GC/C/IRMS). Analyses were performed using a TRACE GC Ultra GC coupled to a Thermo Finnigan DELTA$_{plus}$ XP mass spectrometer via a Thermo Finnigan GC Combustion III interface. Samples were introduced via autosampler or injected via a PTV injector in splitless mode, onto a fused silica capillary column (HP-1, non-polar, 50 m x 0·32 mm i.d., 0.1 µm film thickness). The flow rate of the carrier gas (He) was set at 2 ml min^{-1} and the Cu/Ni/Pt oxidation reactor was maintained at 940°C. The temperature programme consisted of a 2 min isothermal at 50° and then ramped at 10° min^{-1} to 300°C followed by a 10 min isothermal. Results were calibrated against reference CO_2, which was injected directly into the source three times at the beginning and end of the run. All samples were run in duplicate with external standards every four runs.

The $\delta^{13}C$ values were derived according to the following expression and are relative to the international standard vPDB: $\delta^{13}C$ ‰ = ((R sample − R standard)/R standard) × 1000, where R = $^{13}C/^{12}C$ and were corrected for the carbon atoms added during methylation using a mass balance equation (Rieley, 1994).

References

1. Boessneck, J., Müller, H. H. and Teichert, M. (1964). Osteologishe Unterscheidungsmerkmahle zwischen Schaf (Ovis aries Linnaeus) une Ziege (Capra hircus Linnaeus). Kühn Archiv 78(1-2): 1-129.

2. Caneva, I. (1988). El Geili: the history of a Middle Nile environment 7,000 B.C.-A.D. 1,500. Oxford: B.A.R. International Series 424.

3. Copley, M. S., Berstan, R., Dudd, S. N., Docherty, G., Mukherjee, A. J., Straker, V., Payne, S. and Evershed, R. P. (2003). Direct chemical evidence for widespread dairying in Prehistoric Britain. Proceedings of the National Academy of Sciences of the United States of America 100(4): 1524-1529.

4. Craig, O. E., Chapman, J., Heron, C., Willis, L. H., Bartosiewicz, L., Taylor, G., Whittle, A. and Collins, M. (2005). Did the first farmers of central and eastern Europe produce dairy foods? Antiquity 79(306): 882-894.

5. Cramp, L. J. E., Evershed, R. P. and Eckardt, H. (2011). What was a mortarium used for? Organic residues and cultural change in Iron Age and Roman Britain. Antiquity 85: 1339-1352.

6. de Beaumais, A. and Royer, P. (1926). Fouilles de l'Adrar Gueldaman. Bulletin de la Société préhistorique française vol. XXIII: 223-238.

7. deMenocal, P., Ortiz, J., Guilderson, T., Adkins, J., Sarnthein, M., Baker, L. and Yarusinsky, M. (2000). Abrupt onset and termination of the African Humid Period: rapid climate responses to gradual insolation forcing. Quaternary Science Reviews 19(1-5): 347-361.

8. Dudd, S. N. and Evershed, R. P. (1998). Direct demonstration of milk as an element of archaeological economies. Science 282(5393): 1478-1481.

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

10. Dunne, J., Evershed, R. P., Cramp, L. J. E., Bruni, S., Biagetti, S. and di Lernia, S. (2013). The beginnings of dairying as practised by pastoralists in 'Green' Saharan Africa in the 5th millennium BC. Documenta Praehistorica 40: 118-130.

11. Dunne, J., Evershed, R. P., Salque, M., Cramp, L., Bruni, S., Ryan, K., Biagetti, S. and di Lernia, S. (2012). First dairying in green Saharan Africa in the fifth millennium bc. Nature 486(7403): 390-394.

12. Evans, K. and Heron, C. (1993). Glue, disinfectant and chewing gum: natural products chemistry in archaeology. Chemistry & Industry 12: 446-449.

13. Evershed, R. P., Dudd, S. N., Anderson-Stojanovic, V. R. and Gebhard, E. R. (2003). New chemical evidence for the use of combed ware pottery vessels as beehives in Ancient Greece. Journal of Archaeological Science 30(1): 1-12.

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

15. Evershed, R. P., Stott, A. W., Raven, A., Dudd, S. N., Charters, S. and Leyden, A. (1995). Formation of long-chain ketones in ancient pottery vessels by pyrolysis of acyl lipids. Tetrahedron Letters 36(48): 8875-8878.

16. Evershed, R. P., Vaughan, S. J., Dudd, S. N. and Soles, J. S. (1997). Fuel for thought? Beeswax in lamps and conical cups from late Minoan Crete. Antiquity 71(274): 979-985.

17. Farquhar, G. D., Ehleringer, J. R. and Hubick, K. T. (1989). Carbon isotope discrimination and photosynthesis. Annual Review of Plant Physiology and Plant Molecular Biology 40(1): 503-537.

18. Gannes, L. Z., O'Brien, D. M. and Martínez del Rio, C. (1997). Stable isotopes in animal ecology: assumptions, caveats, and a call for more laboratory experiments. Ecology 78(4): 1271-1276.

19. Garcea, E. A. A. (2006). Semi-permanent foragers in semi-arid environments of North Africa. World Archaeology 38(2): 197-219.

20. Gautier, A. (1984). The fauna of the Neolithic site of Kadero (Central Sudan). In Origin and early development of food-producing cultures in North-Eastern Africa, eds. L. Krzyzaniak and M. Kobusiewicz, Poznan: Polish Academy of Sciences and Poznan Archaeological Museum. 317-319

21. Gautier, A. (1987). Prehistoric men and cattle in North Africa: a dearth of data and a surfeit of models. In Prehistory of Arid North Africa, ed. A. E. Close, Dallas: Southern Methodist University Press. 163-187

22. Gifford-Gonzalez, D. (2005). Pastoralism and its consequences. In African Archaeology, ed. A. B. Stahl, Oxford: Blackwell Publishing. 187-224

23. Gregg, M. W., Banning, E. B., Gibbs, K. and Slater, G. F. (2009). Subsistence practices and pottery use in Neolithic Jordan: molecular and isotopic evidence. Journal of Archaeological Science 36(4): 937-946.

24. Haaland, R. (1992). Fish, pots and grain: Early and Mid-Holocene adaptations in the Central Sudan. African Archaeological Review 10(1): 43-64.

25. Haaland, R. (1995). Sedentism, cultivation, and plant domestication in the Holocene Middle Nile Region. Journal of Field Archaeology 22(2): 157-174.

26. Hansel, F. A., Copley, M. S., Madureira, L. A. S. and Evershed, R. P. (2004). Thermally produced [omega]-(o-alkylphenyl)alkanoic acids provide evidence for the processing of marine products in archaeological pottery vessels. Tetrahedron Letters 45(14): 2999-3002.

27. Helmer, D. (1992). La domestication des animaux par les hommes préhistoriques. Paris: Masson.

28. Helmer, D., Gourichon, L. and Vila, E. (2007). The development of the exploitation of products from Capra and Ovis (meat, milk and fleece) from the PPNB to the Early Bronze in the northern Near East (8700 to 2000 BC cal.). Anthropozoologica 42(2): 41-69.

29. Helmer, D. and Vigne, J.-D. (2004). La gestion des cheptels de caprinés au Néolithique dans le midi de la France. In Approches Fonctionnelles en Préhistoire, Actes du XXVème Congrès Préhistorique de France (Nanterre, 2000), eds. P. Bodu and C. Constantin, Paris: Mémoires de la Société Préhistorique Française, Numéro spécial. 397-407

30. Heron, C., Nemcek, N., Bonfield, K. M., Dixon, D. and Ottaway, B. S. (1994). The chemistry of Neolithic beeswax. Naturwissenschaften 81(6): 266-269.

31. Holmes, W. (1970). Animals for food. Proceedings of the Nutritional Society 29(02): 273-281.

26

32. Jackson, L. L. and Blomquist, G. J. (1976). Insect Waxes. In Chemistry and Biochemistry of Natural Waxes, ed. P. E. Kolattukudy, Amsterdam: Elsevier. 201-233

33. Kherbouche, F., Hachi, S., Abdessadok, S., Sehil, N., Merzoug, S., Sari, L., Benchernine, R., Chelli, R., Fontugne, M., Barbaza, M. and Roubet, C. (2014). Preliminary results from excavations at Gueldaman Cave GLD1 (Akbou, Algeria). Quaternary International 320(0): 109-124.

34. Krzyzaniak, L. (1991). Early farming in the Middle Nile Basin: recent discoveries at Kadero (Central Sudan). Antiquity 65(248): 515-532.

35. Linstädter, J. (2008). The Epipalaeolithic-Neolithic-transition in the Mediterranean region of Northwest Africa. Quartär 55: 41-62.

36. Lubell, D., Feathers, J. and Schwenninger, J.-L. (2009). Post-Capsian occupation in the eastern Maghreb: implications of a revised chronological assessment for the adult burial at Aïn Misteheyia. Journal of African Archaeology 7(2): 175-189.

37. Marshall, F. and Hildebrand, E. (2002). Cattle before crops: the beginnings of food production in Africa. Journal of World Prehistory 16(2): 99-143.

38. Mukherjee, A. J., Copley, M. S., Berstan, R., Clark, K. A. and Evershed, R. P. (2005). Interpretation of δ13C values of fatty acids in relation to animal husbandry, food processing and consumption in Prehistory. The Zooarchaeology of Fats, Oils, Milk and Dairying - 9th ICAZ Conference. J. Mulville and A. K. Outram. Durham, Oxbow Books: 77-93.

39. Outram, A. K., Stear, N. A., Bendrey, R., Olsen, S., Kasparov, A., Zaibert, V., Thorpe, N. and Evershed, R. P. (2009). The earliest horse harnessing and milking. Science 323(5919): 1332-1335.

40. Payne, S. (1973). Kill-off patterns in sheep and goats: the mandibles from Aşvan Kale. Anatolian Studies 23: 281-303.

41. Payne, S. (1985). Morphological distinctions between the mandibular teeth of young sheep, Ovis, and Goats, Capra. Journal of Archaeological Science 12: 139-147.

42. Payne, S. (1987). Reference codes for Wear Stages in the mandibular cheek teeth of sheep and goats. Journal of Archaeological Science 1 14: 609-614.

43. Prummel, W. and Frisch, H.-J. (1986). A guide for the distinction of species, sex, and body size of sheep and goat. Journal of Archaeological Science 14: 609-614.

44. Rahmani, N. (2004). Technological and cultural change among the last hunter-gatherers of the Maghreb: The Capsian (10,000–6000 B.P.). Journal of World Prehistory 18(1): 57-105.

45. Raven, A. M., van Bergen, P. F., Stott, A. W., Dudd, S. N. and Evershed, R. P. (1997). Formation of long-chain ketones in archaeological pottery vessels by pyrolysis of acyl lipids. Journal of Analytical and Applied Pyrolysis 40–41(0): 267-285.

46. Regert, M., Colinart, S., Degrand, L. and Decavallas, O. (2001). Chemical alteration and use of beeswax through time: accelerated ageing tests and analysis of archaeological samples from various environmental contexts. Archaeometry 43(4): 549-569.

47. Regert, M., Dudd, S. N., Pétrequin, P. and Evershed, R. P. (1999). Fonction des céramiques et alimentation au Néolithique final sur les sites de Chalain. De nouvelles voies d'étude fondées sur l'analyse chimique des résidus organiques conservés dans les poteries. Revue d'archéométrie 23: 91-99.

48. Rieley, G. (1994). Derivatization of organic compounds prior to gas chromatographic combustion-isotope ratio mass spectrometric analysis: identification of isotope fractionation processes. Analyst 119(5): 915-919.

49. Roubet, C. (2001). Le Néolithique de tradition capsienne sensu stricto. In Encyclopedia of Prehistory. Volume 1: Africa, eds. P. N. Peregrine and M. Ember, New York: Kluwer Academic, Plenum Publishers. 197-219

50. Roubet, C. (2003). Statut de berger: des communautés atlasiques, néolithisées du Maghreb oriental, dès 7000 BP. L'Anthropologie 107(3): 393-442.

51. Salque, M. (2012). Regional and chronological trends in milk use in prehistoric Europe traced through molecular and stable isotope signatures of fatty acyl lipids preserved in pottery vessels. Unpublished PhD Thesis, University of Bristol.

52. Salque, M., Bogucki, P. I., Pyzel, J., Sobkowiak-Tabaka, I., Grygiel, R., Szmyt, M. and Evershed, R. P. (2013). Earliest evidence for cheese making in the sixth millennium BC in northern Europe. Nature 493(7433): 522-525.

53. Sherratt, A. (1983). The secondary exploitation of animals in the Old World. World Archaeology 15(1): 90-104.

54. Sherratt, A. G. (1981). Plough and pastoralism: aspects of the Secondary Products Revolution. In Pattern of the Past eds. I. Hodder, G. Isaac and N. Hammond, Cambridge: Cambridge University Press. 261-306

55. Smith, A. B. (1980). Domesticated cattle in the Sahara and their introduction into West Africa. In The Sahara and the Nile, eds. M. A. J. Williams and H. Faure, Rotterdam: A.A. Balkema. 489-503

56. Spangenberg, J. E., Jacomet, S. and Schibler, J. (2006). Chemical analyses of organic residues in archaeological pottery from Arbon Bleiche 3, Switzerland − evidence for dairying in the late Neolithic. Journal of Archaeological Science 33(1): 1-13.

57. Stern, B., Connan, J., Blakelock, E., Jackman, R., Coningham, R. A. E. and Heron, C. (2008). From Susa to Anuradhapura: Reconstructing aspects of trade and exchange in bitumen-coated ceramic vessels between Iran and Sri Lanka from the Third to the Ninth Centuries AD. Archaeometry 50: 409-428.

58. Stern, B., Heron, C., Corr, L., Serpico, M. and Bourriau, J. (2003). Compositional variations in aged and heated pistacia resin found in Late Bronze Age Canaanite amphorae and bowls from Amarna, Egypt Archaeometry 45(3): 457-469.

59. Tulloch, A. P. (1971). Beeswax: Structure of the esters and their component hydroxy acids and diols. Chemistry and Physics of Lipids 6(3): 235-265.

60. Tulloch, A. P. and Hoffman, L. L. (1972). Canadian beeswax: Analytical values and composition of hydrocarbons, free acids and long chain esters. Journal of the American Oil Chemists Society 49(12): 696-699.

61. Vigne, J.-D. and Helmer, D. (1999). Nouvelles analyses sur les débuts de l'élevage dans le centre et l'ouest méditerranéen. Le Néolithique du Nord-Ouest méditerranéen, Actes du XXIVe congrès préhistorique de France. Carcassonne 2-30 septembre 1994.

62. Vigne, J.-D. and Helmer, D. (2007). Was milk a "secondary product" in the Old World Neolithisation process? Its role in the domestication of cattle, sheep and goats. Anthropozoologica 42(2): 9-40.

63. West, J. B., Bowen, G. J., Dawson, T. E. and Tu, K. P. (2010). Isoscapes: Understanding Movement, Pattern and Process on Earth through Isotope Mapping. New York: Springer.

64. Zeder, A.-M. and Pilaar, S.-E. (2010). Assessing the reliability of criteria used to identify mandibles and mandibular teeth in sheep, Ovis, and goats, Capra. Journal of Archaeological Science 37: 225-242.

31

65. Zeder, M.-A. and Lapham, H.-A. (2010). Assessing the reliability of criteria used to identify postcranial bones in sheep, Ovis, and goats, Capra. Journal of Archaeological Science 37(11): 2887-2905.