The beginnings of dairying as practised by pastoralists in 'green' Saharan Africa in the 5th millennium BC

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ABSTRACT – *Previous research has identified the antiquity and chronology of dairying practices as beginning in the Near East and its subsequent spread across Europe. In the Libyan Sahara, archaeological evidence, confirmed by the remarkable rock art depicting cattle herding, together with faunal evidence, also suggests an early inception of dairying practices in North Africa and the formation of an independent 'secondary products' economy by mobile pastoral groups. In this paper, we elaborate on the first unequivocal chemical evidence, based on the $\delta^{13}C$ and $\Delta^{13}C$ values of the major fatty acids of milk fat, for the adoption of dairying practices by prehistoric Saharan African people in the fifth millennium BC.*

IZVLEČEK – *Predhodne raziskave so pokazale, da je najstarejši začetek uporabe mleka vezan na območje Bližnjega Vzhoda, od koder se je potem širila v Evropo. Arheološki podatki iz libijske Sahare, podprti z izjemnimi upodobitvami pastirjev z govedom v skalah in z živalskimi ostanki na najdiščih, kažejo na zgodnjo uporabo mleka v Severni Afriki in na neodvisno gospodarstvo 'sekundarnih produktov', ki so ga oblikovale mobilne pastirske skupine. V članku predstavljamo prve nedvoumne kemijske dokaze, ki temeljijo na vrednostih $\delta^{13}C$ in $\Delta^{13}C$ glavnih mlečno-maščobnih kislin, o usvojitvi mlečno-gospodarskih praks prazgodovinskih ljudi v saharski Afriki v petem tisočletju pr. n. št.*

KEY WORDS – *dairying; North Africa; Sahara; Tadrart Acacus; pottery; hunter-gathers; herders; cattle; rock art; stable carbon isotopes; fatty acids*

Introduction

In African prehistory, it is widely accepted that the existence of pastoralism (using domesticated cattle, sheep and goats) as an established and widespread way of life emerged long before plant domestication (*Marshall, Hildebrand 2002*). This is in contrast to the process of 'Neolithisation' beginning in the Near East, characterised by the transition from a mobile hunter-gatherer lifestyle to an increasingly settled, agricultural way of life. With the domestication of plants and animals such as cattle, sheep, goats and pigs, and the adoption of these new subsistence practices in the Near East, this sedentary, farming way of life spread outwards across Europe and into the British Isles.

In Saharan Africa, extremely favourable climatic and environmental conditions prevailed during the Holocene African Humid Period which began around 10000 years ago (*e.g., de Menocal* et al. *2000; Gasse 2000; Cremaschi* et al. *2010*). The strengthening of northern hemisphere summer insolation due to earth orbital changes resulted in a shifting of the African monsoon hundreds of kilometres to the north, leading to an intensive recharge of large water bo-

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dies such as Lake Mega-Chad and Lake Mega-Fazzan. Rock art and faunal remains confirm the presence of abundant savannah and lake margin fauna such as elephant, hippopotamus, giraffe and crocodile at this time, inhabiting a mosaic of savannah and woodland vegetation (*Brooks* et al. *2005; Mercuri 2008; Cremaschi, Zerboni 2009; Cremaschi* et al. *2010; Drake* et al. *2011*).

During these prevailing favourable climatic conditions in the Early Holocene, prehistoric peoples in the Libyan Sahara lived as primarily sedentary and pottery-producing hunters, fishers and gatherers, exploiting different environments and resources. Towards the end of the Early Holocene period, we see the advent of food production (throughout the Sahara) with the adoption of bovids and ovicaprids (*Cremaschi, di Lernia 1999; Garcea 2004; Brooks* et al. *2005; Cremaschi, Zerboni 2009; di Lernia 2013*).

Fig. 1. Rock art image of domesticated cattle, between 5000 and 8000 years old, from Tagg'n Tort, in the Tadrart Acacus Mountains, Libyan Sahara. (Photo by Filippo Gallino; The Archaeological Mission in the Sahara, Sapienza University of Rome).

The development of African subsistence strategies would likely have been heavily shaped by the drying conditions beginning in the Middle Holocene, which led to the unstable, marginal environments that Saharan hunter-gatherers inhabited. As conditions deteriorated and aridification took hold (*Brooks* et al. *2005; Cremaschi, Zerboni 2009; Cremaschi* et al. *2010*), the previously semi-sedentary prehistoric groups became nomadic cattle herders (*di Lernia 2002*) moving seasonally in response to climatic conditions. Then, predictable access to resources would surely have been their major concern, rather than the intensification of yield more applicable to early farmers in the Levant (*Marshall, Hildebrand 2002*).

Secondary products and the dairying revolution

Some thirty years ago, Andrew Sherratt (*1983*) argued that, several millennia after the beginning of the development of animal husbandry in the Near East, another innovation in animal exploitation occurred, that of the intensive use of secondary products, such as milk, blood, wool and traction, which can be repeatedly extracted from an animal throughout its lifetime. It is now generally agreed that the exploitation of secondary products probably began earlier, likely during the first spread of farming (*Sherratt 2006*), although it is increasingly becoming clear that the adoption and development of dairying as part of a subsistence strategy may have been a piecemeal process, developing in varying ways depending on local environmental conditions and different cultural groups (*Evershed* et al. *2008*). Direct evidence for the practice of dairying, beginning in the 7th millennium BC in northwestern Anatolia (*Evershed* et al. *2008*), appearing in the 6th millennium BC in eastern Europe (*Craig* et al. *2005*) and reaching Britain in the 4th millennium BC (*Copley* et al. *2003; Dudd, Evershed 1998*) has been established through the compound-specific stable carbon isotope analysis of animal fat residues preserved in archaeological pottery. Significantly, this research on the antiquity of dairying practices has largely been confined to Europe, the Near East and Eurasia, with no attempt yet being made to identify the inception of dairying practices in the African continent. In the Libyan Sahara, however, the rock art and faunal evidence (*e.g., Mori 1965; Cremaschi, di Lernia 1998; di Lernia, Zampetti 2008*) suggests that the inception of dairying practices in north Africa and an early and independent 'secondary products' economy (*Sherratt 1983*) seems plausible given what we now know of the first appearance of milking in the Near East (*Evershed* et al. *2008*).

Rock art and cattle

Compelling evidence (not seen in European contexts) of prehistoric cattle herding in northern Africa comes from the remarkable rock paintings and engravings of the Sahara (Fig. 1), possibly one of the world's highest concentrations of prehistoric art, and long known for their rich and vivid portrayal of astonishing scenes from everyday life. The extensive rock art, probably originating from *c.* 10 000 years ago, demonstrates that cattle clearly played a significant part in the lives and ideology of ancient human groups living in this region during the Holocene. The most widespread representations, with hundreds of decorated sites having been identified throughout the Sahara, are of domestic cattle and it is thought this 'style' (with many internal regional variants), generally associated with Pastoral Neolithic groups, may have persisted for a very long period, from around 7000–4000 uncal BP (*e.g., di Lernia, Gallinaro 2010; Gallinaro* et al. *2008; Le Quellec 1998; Lutz, Lutz 1995*). This pictorial record contains countless scenes with representations of cattle, some emphasising the female's full udders and, in a few cases, depictions of the actual milking of a cow, such as at Wadi Teshuinat II (*Gallinaro* et al. *2008*) in the Acacus or Wadi Tiksatin in the Messak (*Lutz, Lutz 1995*). However, reliable dates for this rock art can rarely be ascertained (*di Lernia, Gallinaro 2010*) and thus, although highly suggestive of the existence of dairying practices by prehistoric herders in the region, cannot provide an accurate chronology of its uptake.

Faunal remains from securely dated Saharan contexts also indicate that domesticated animals (cattle, sheep or goats) were present in the area from the end of the 8th millennium BP (*di Lernia 2013*), becoming much more common in the 5th millennium BC (*Gautier 2002; Gifford-Gonzalez, Hanotte 2011*). In European contexts, the reconstruction of the age profile of domesticated animals (which reflects herding strategies) excavated from archaeological sites enables the identification of kill-off patterns (*Payne 1973; Vigne, Helmer 2007*). Unfortunately, in the Libyan Sahara, these remains are very poorly preserved and highly fragmented, precluding such herd reconstructions; thus even indirect evidence of dairying is missing (*Gautier 2002*).

The archaeological evidence comprising the rock art, faunal remains and ceramic assemblages suggests that we might hypothesise that these prehistoric herders were exploiting their cattle for their secondary products. Thus, a biomolecular approach, where lipid residues are extracted from Saharan ceramics (Fig. 2), has the potential to ascertain the chronology and location of the inception of dairying practices beginning in northern Africa.

Biomolecular archaeology and lipids

The field of biomolecular archaeology can be defined as the study of biological molecules surviving from antiquity which yield information relating to past human activity (*Evershed 1993; 2008*). Over the last decades, analytical methodologies have been developed to investigate and identify 'archaeological biomarkers' in biomolecules such as ancient DNA, protein, carbohydrates, pigments and lipids. Lipids, the organic solvent soluble components of living organisms, *i.e.* the fats, waxes and resins of the natural world, are the most frequently recovered compounds from archaeological contexts (*Evershed 1993; 2008*). They are also resistant to decay and are likely to endure at their site of deposition because of their inherent hydrophobicity, making them excellent candidates for use as biomarkers in archaeological research (*Evershed 1993; 2008*).

Pottery has become one of the most extensively studied materials for organic residue analysis (*Mukherjee* et al. *2005*) as ceramics, once made, are virtually indestructible and therefore among the most common artefacts recovered from archaeological sites from the Neolithic period onwards (*Tite 2008*). These residues survive in three ways; rarely, actual contents are preserved in situ (*e.g., Charrie-Duhaut* et al. *2007*) or, more commonly, as surface residues (*Evershed 2008*). The most common form is that of absorbed residues preserved within the vessel wall, which have been found to survive in >80% of dome-

Fig. 2. Middle Pastoral restored vessels from the Murzuq dunefield (*c. *6000–5000 uncal BP).

stic cooking pottery assemblages worldwide (*Evershed 2008*).

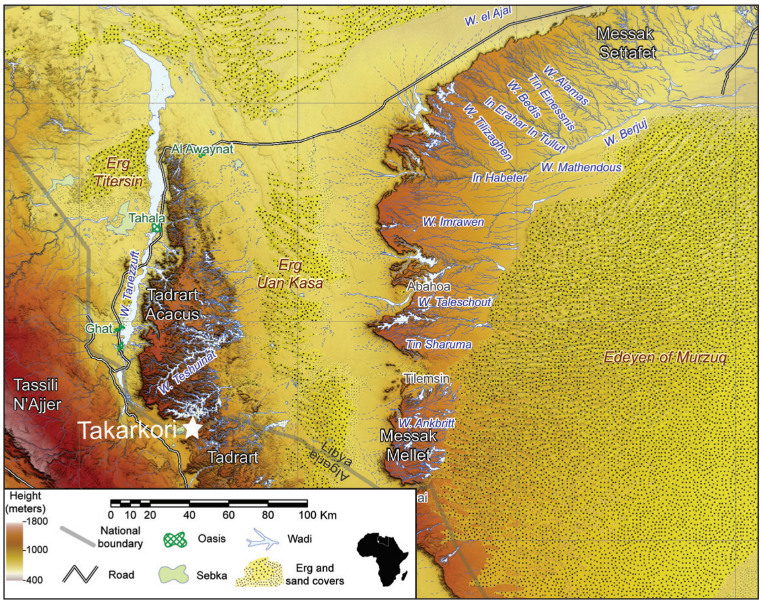

Fig. 3. The Takarkori rockshelter shown in the region of the Messak plateau and surroundings (modified, from* di Lernia *et al.* 2013*).

The analysis of lipid components of visible or absorbed organic residues found in archaeological pottery has to date allowed the identification of a considerable range of substances such as terrestrial animal fats (*Evershed* et al. *1997a; Mottram* et al. *1999*), marine animal fats (*Copley* et al. *2004; Craig* et al. *2007*), plant waxes (*Evershed* et al, *1991*), beeswax (*Evershed* et al. *1997b*), birch bark tar (*Charters* et al. *1993a; Urem-Kotsou* et al. *2002*) and palm kernel oil (*Copley* et al. *2001*). However, preserved animal fats are by far the most commonly observed constituents of lipid residues recovered from archaeological ceramics. A range of chemical criteria, including saturated fatty acid compositions, double bond positions, triacylglycerol distributions and $\delta^{13}C$ have been used to assign the origin of ancient animal fats to domesticated animals such as cattle, pigs and sheep and goats (*Copley* et al. *2003; Dudd, Evershed 1998; Mottram* et al. *1999*). These techniques have allowed the fats from ruminant and non-ruminant animals to be distinguished, but it was the establishment of a stable carbon isotope approach which allowed the identification of ruminant adipose and dairy fats resting on differences in the $\delta^{13}C$ values of the principal fatty acids (C_{16} and C_{18}) that are present in all animal fats, and this technique has therefore been of considerable value in answering many archaeological questions regarding ancient economies. This method is based on the fact that the differential routing of dietary carbon and fatty acids during the synthesis of adipose and dairy fats in ruminant animals results in different $\delta^{13}C$ values for the two saturated fatty acids produced (*Dudd, Evershed 1998*).

Site and samples

The Takarkori site is a large rock shelter some 80m long and approx. 10 to 15m deep, lying 100m above a dry river valley. It is located in the Tadrart Acacus massif, which is situated in the Central Sahara, in the south-western corner of Libya, close to the Algerian border (Fig 3.) (*Biagetti, di Lernia 2007; Biagetti* et al. *2004; Biagetti, di Lernia 2013*).

The Tadrart Acacus is a sandstone mountain range traversed by several wadis that run from west to east. Several rock shelters serving as human occupations since the late Pleistocene are located along the wadis (*Barich 1987; Cremaschi, di Lernia 1999*).

This region (see Fig. 3) has been licensed to the 'Italian-Libyan Archaeological Mission in the Acacus and Messak' under the control of the University of Rome La Sapienza and the Department of Antiquities, Tripoli. Research began here in 1955, originally focused on rock art, but later expanding into excavations of single stratified rock shelters such as Ti-n-Torha, Uan Muhuggiag, Uan Tabu and Uan Telecat (*Mori 1965; Barich 1987; Cremaschi, di Lernia 1998*). In the 1990s, a landscape approach was adopted, using systematic extensive surveys, which has gathered a large dataset relating to landscape exploitation, settlement patterns, rock art, genetics and burial customs within Holocene cultural trajectories, all of which have led to crucial insights into the prehistory of this part of the Sahara. Additional excavations also took place at stratified shelters in the Acacus, such as Uan Afuda, Uan Tabu and Takarkori (*Cremaschi, di Lernia 1998; di Lernia 1999; Garcea 2001; Biagetti, di Lernia 2013*).

This long period of extensive research in the region has provided an understanding of the development of human subsistence strategies within the region and led to the development of a model of cultural trajectories (*e.g., Cremaschi, di Lernia 1999*). The two cultural horizons within the Early Holocene based on extractive economies are named Early and Late Acacus. Early Acacus hunter-gatherers specialised in hunting Barbary sheep from multi-activity base camps within the Acacus and small site hunting stations near lakes in the lowlands (*Barich 1987; di Lernia 1996; Cremaschi, di Lernia 1998; Garcea 2001*). The Late Acacus was marked by a more sedentary lifestyle, with a broader subsistence base largely founded on plant resources, and the introduction of grinding equipment and ceramic technology (*Barich 1987; di Lernia 1999; Garcea 2001; Biagetti, di Lernia 2007*). Some animal management (corralling and delayed use) of Barbary sheep also appears to take place at this time (*di Lernia 2001*).

The subsequent 'Pastoral' or Neolithic horizon consists of four phases, Early, Middle, Late and Final Pastoral (*di Lernia 1999; di Lernia, Merighi 2006*). These stages cover the Holocene and denote the adoption of cattle and sheep/goats, together with intensive exploitation of wild cereals (*Mercuri 2008*). In the Early Pastoral period, sedentism (in the mountains) remained high, with the settlement pattern in the Middle Pastoral being characterised by the seasonal exploitation of different environments, with the herding of cattle through a transhumant lifestyle through summer semi-residential sites in the lowlands to winter sites in the mountains (*Cremaschi, di Lernia 1999; di Lernia 2002*). In the Late and Final Pastoral period (5th–4th millennium BP), increasingly arid conditions denote a move to long-distance specialised nomadism with sheep and goats (*di Lernia 1999; di Lernia, Merighi 2006*).

The Takakori rock shelter was chosen for excavation because of the remarkable preservation of its archaeological deposit (about 1.6m in depth) over a large area. Fieldwork identified evidence of a long and protracted human occupation covering the Late Acacus (Mesolithic) period, together with Early, Middle and Late Pastoral (Neolithic) remains. Radiocarbon dating fixes the layers between 8800 to 4200 years uncal BP, demonstrating more than 4000 years of human occupation (*Biagetti, di Lernia 2007; 2013*).

Sherd number	Lab Code	Period	Lipid concentration (ug/g)	$\delta^{13}C_{16.0}$	$\delta^{13}C_{18.0}$	$\Delta 13C$	Classification
21	TAK21A	Middle Pastoral	5830.6	−14.7	−20.5	−5.8	Dairy Fat
26	TAK1	Middle Pastoral	760.7	−14.2	−15.0	−0.9	Ruminant adipose
45	TAK45	Middle Pastoral	639.8	−21.9	−24.1	−2.1	Ruminant adipose
120	TAK120	Middle Pastoral	5592.7	−15.2	−18.7	−3.5	Dairy Fat
124	TAK124	Middle Pastoral	1615.5	−18.1	−20.1	−2.0	Ruminant adipose
197	TAK197	Middle Pastoral	151.5	−20.9	−21.1	−0.2	Non-ruminant adipose
420	TAK420	Middle Pastoral	1119.3	−18.3	−21.5	−3.2	Dairy Fat
443	TAK443	Middle Pastoral	17217.6	−16.9	−23.7	−6.8	Dairy Fat
576	TAK6	Middle Pastoral	800.2	−22.0	−21.7	0.3	Non-ruminant adipose
748	TAK9	Middle Pastoral	5650.5	−13.7	−19.0	−5.2	Dairy Fat
824	TAK11	Late Pastoral	4994.2	−20.5	−24.9	−4.4	Dairy Fat
873	TAK873	Middle Pastoral	71.8	−18.5	−17.7	0.8	Non-ruminant adipose
896	TAK896	Middle Pastoral	218.0	−23.6	−25.0	−1.5	Ruminant adipose
987	TAK987	Middle Pastoral	4442.6	−13.6	−19.3	−5.7	Dairy Fat
997	TAK15	Middle Pastoral	1117.4	−13.3	−17.4	−4.1	Dairy Fat
1009	TAK1009	Middle Pastoral	1555.7	−11.0	−11.0	0.0	Non-ruminant adipose
1012	TAK1012	Middle Pastoral	3591.2	−14.9	−16.5	−1.7	Ruminant adipose
1572	TAK1572	Middle Pastoral	3148.5	−23.7	−28.2	−4.5	Dairy Fat
1693	TAK21	Late Acacus	20.1	−23.1	−19.8	3.3	Non-ruminant adipose
1797	TAK24	Early Pastoral	1674.6	−21.9	−21.0	0.9	Non-ruminant adipose
1846	TAK25	Middle Pastoral	819.2	−15.6	−19.7	−4.1	Dairy Fat
1863	TAK26	Middle Pastoral	175.0	−22.3	−26.2	−4.0	Dairy Fat
1903	TAK27	Early Pastoral	308.5	−22.8	−21.7	1.1	Non-ruminant adipose
2028	TAK2028	Middle Pastoral	1931.0	−24.5	−28.9	−4.4	Dairy Fat
2251	TAK28	Middle Pastoral	96.9	−21.5	−24.0	−2.5	Ruminant adipose
2523	TAK29	Late Pastoral	445.6	−18.5	−19.7	−1.2	Ruminant adipose
2588	TAK30	Late Acacus	823.3	−13.9	−13.8	0.1	Non-ruminant adipose
2817	TAK32	Late Acacus	6882.8	−19.3	−17.5	1.8	Non-ruminant adipose
2857	TAK35	Middle Pastoral	238.3	−20.1	−22.9	−2.8	Ruminant adipose

Tab. 1. Subset of sherds selected for isotopic analyses showing period, lipid concentrations and fatty acid $\delta^{13}C$ and $\Delta^{13}C$ values.

This site presented a remarkable opportunity to utilise the archaeological biomarker approach to identify which foodstuffs were processed in the ceramic assemblages from each cultural horizon and thus ascertain the subsistence strategies practised by the prehistoric peoples living at the site together with their changes through time.

Material and methods

A total of 81 potsherds covering a wide range of decoration techniques and motives commonly found on Saharan ceramics (*Biagetti* et al. *2004; Caneva 1987*) were sampled from the Takarkori rock shelter, of which 56 were excavated from the Middle Pastoral period (*c.* 5200–3800 BC), and the remainder originating from the Late Acacus (n = 8), and Early (n = 14) and Late Pastoral (n = 3) periods (Tab. 1). The samples were analysed by GC and GC–MS in order to identify and quantify the extracted compounds. Only those residues unambiguously assigned as degraded animal fats, *i.e.* those with exceptionally high $C_{16:0}$ and $C_{18:0}$ values, were selected for gas chromatography-combustion-isotope ratio mass spectrometry (GC–C–IRMS) analysis to determine the $\delta^{13}C$ values for the individual $C_{16:0}$ and $C_{18:0}$ carboxylic acids, with the aim of classifying the fats. Of the 29 samples selected for GC–C–IRMS analysis, 18 displayed clear evidence of pure animal fat origin, with the remaining 11 comprising lipid profiles suggestive of the mixing of animal and plant fats.

Lipid analysis and interpretations were performed by means of established protocols described in detail in earlier publications (*e.g., Evershed* et al. *2008*). Briefly, ~2g of potsherd were sampled and surfaces cleaned with a modelling drill to remove any exogenous lipids. The sherds were then ground to a powder, an internal standard added and solvent extracted by ultrasonication (chloroform/methanol 2:1 ν/ν, 2x10ml). The solvent was evaporated under a gentle stream of nitrogen to obtain the total lipid extract (TLE). Aliquots of the TLE were trimethylsilylated (*N,O*-bis(trimethylsilyl) trifluoroacetamide 80µl, 70°C, 60min; Sigma-Aldrich Company Ltd, Gillingham, UK), and submitted to analysis by GC and GC–MS. Further aliquots of the TLE were treated with NaOH/H_2O (9:1 ω/ν) in methanol (5% ν/ν, 70°C, 1h). Following neutralisation, lipids were extracted into chloroform and the excess solvent evaporated under a gentle stream of nitrogen. Fatty acid methyl esters (FAMEs) were prepared by reaction with BF_3-methanol (14% ω/ν, 70°C, 1h; Sigma-Aldrich Company Ltd, Gillingham, UK). The FAMEs were extracted with chloroform, and the solvent removed under nitrogen. The FAMEs were re-dissolved into hexane for analysis by GC and GC–C–IRMS. FAMEs of freeze-dried reference fats (typically using 5mg of TLEs) were prepared exactly as above.

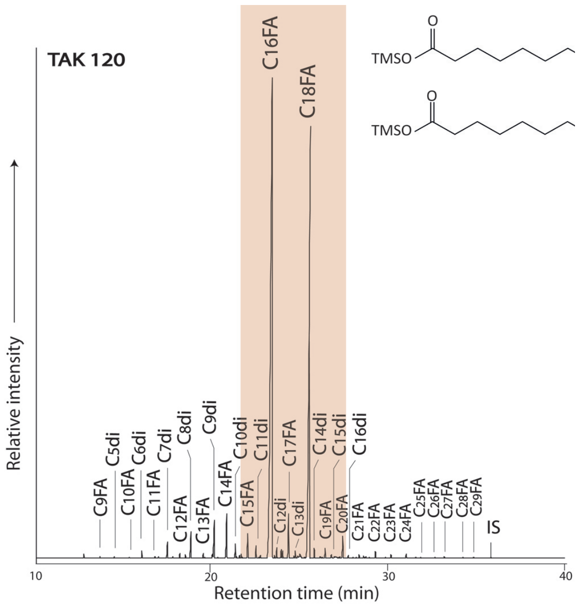

Fig. 4. Partial gas chromatogram displaying the typical trimethylsilylated lipid extract from potsherd TAK120 excavated from Middle Pastoral levels in the Takarkori rock shelter. C_x indicates a fatty acid, where x is the carbon chain length, and $C_x$diFA, α,ω-dicarboxylic acids, IS, Internal Standard, C_{34} n-tetratriacontane. The distribution is characteristic of a degraded animal fat origin.

Results and discussion

GC analysis of the total lipid extracts (TLEs) showed that many of the 81 potsherds demonstrated extraordinary preservation of lipids, containing concentrations of up to 6mg/g (mean 1.2mg/g), with one particular sherd (TAK 443) displaying a concentration of 17mg/g. It is noteworthy that lipids were observed in every sherd, in contrast to European archaeological sites, where generally <40% of sherds contain extractable lipids with mean concentrations of *c.* 0.1mg/g (*Charters* et al. *1993b; Dudd, Evershed 1998*). This remarkable preservation is likely to be related to the arid conditions prevailing in the region.

Lipid biomarker analyses by GC–MS showed residues fall into 3 broad categories. The most common distribution (Fig. 4) was dominated by high abundances of the $C_{16:0}$ and $C_{18:0}$ fatty acids, which derive from degraded animal fats. Also abundant were branched chain fatty acids, C_{13} to C_{18}, components of bacterial origin diagnostic of ruminant animal fats (*Christie 1981*). The two other types of residues identified in these ceramics reflect the processing of plants and mixtures of plant and animal products in the vessels and will not be discussed further in this paper.

With regard to the 81 potsherds, only those residues unambiguously assigned as degraded animal fats (Tab. 1), *i.e.* those dominated by $C_{16:0}$ and $C_{18:0}$ (*e.g.*, Fig. 3), were selected for GC–C–IRMS analysis to determine the $\delta^{13}C$ values for the individual $C_{16:0}$ and $C_{18:0}$ carboxylic acids, with the aim of establishing whether they originate from a ruminant or non-ruminant dairy or adipose fats origin. It has been demonstrated that differences occur in the $\delta^{13}C$ values of these major *n*-alkanoic acids, palmitic ($C_{16:0}$) and stearic ($C_{18:0}$), due to the differential routing of dietary carbon and fatty acids during the synthesis of adipose and dairy fats in ruminant animals, thus allowing ruminant milk fatty acids to be distinguished from carcass fats by calculating $\Delta^{13}C$ values ($\delta^{13}C_{18:0}$–$\delta^{13}C_{16:0}$) and plotting them against the $\delta^{13}C$ value of the $C_{16:0}$ fatty acid. Previous research has shown that by plotting $\Delta^{13}C$ values, variations in C_3 versus C_4 plant consumption are removed, thereby emphasising the biosynthetic and metabolic characteristics of the fat source (*Dudd, Evershed 1998; Copley* et al. *2003*). This is now confirmed by the stable carbon isotope analyses of a new reference collection of modern ruminant animal fats from Africa collected to encompass the range of carbon isoscapes (*West* et al. *2010*) likely to have been encountered by early Saharan pastoralists (*Dunne* et al. *2012*).

Of the 29 animal fats residues selected for GC–C–IRMS analyses, 22 originate from Middle Pastoral levels, 3 from the Late Acacus, 2 from the Early Pastoral and the remaining 2 from the Late Pastoral period (Tab. 1). The comparison of the $\Delta^{13}C$ values of the residues from the archaeological pottery from the Middle Pastoral period (*c.* 5200–3800 BC) with those of modern reference animal fats collected from Libya and Kenya (*Dunne* et al. *2012*) show that 50% of these plot within, or on the edge of, the isotopic ranges for dairy fats, with a further 33% falling within the range for ruminant adipose fats and the remainder corresponding to non-ruminant carcass fats (Figs. 5 and 6). Significantly, the residues originating from the Late Acacus phase, where archaeological levels do not yield faunal remains from domesticated animals, and Early Pastoral periods, where they are rare and badly preserved, do not contain dairy fats, and plot in the non-ruminant fat range, probably derived from wild fauna found locally. The unambiguous conclusion is that the appearance of dairying fats in pottery correlates with the more abundant presence of cattle bones in the cave deposits during the Middle Pastoral period, suggesting a full pastoral economy, as the cattle were intensively exploited for

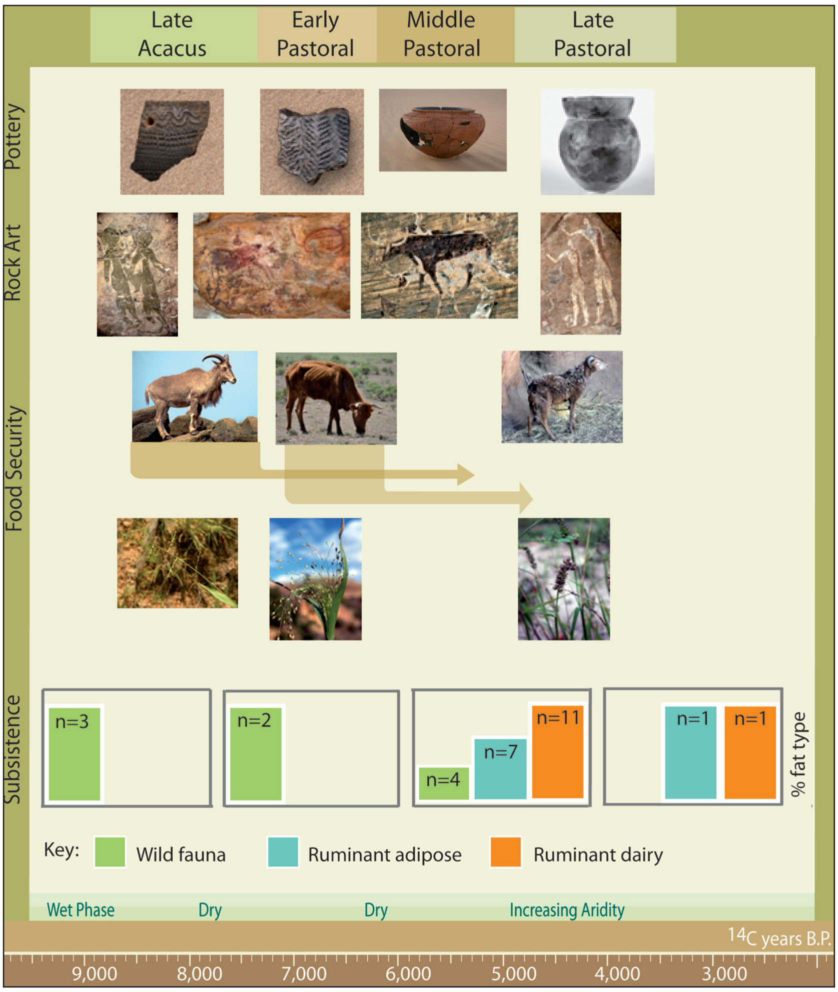

Fig. 5. Showing the cultural sequence in the Tadrart Acacus region, pottery, rock art and food security. Histograms demonstrate the prevalence of dairy fat, ruminant and wild fauna adipose fats within ceramics excavated from the Takarkori rock shelter (modified, after* Dunne et al. *2012).

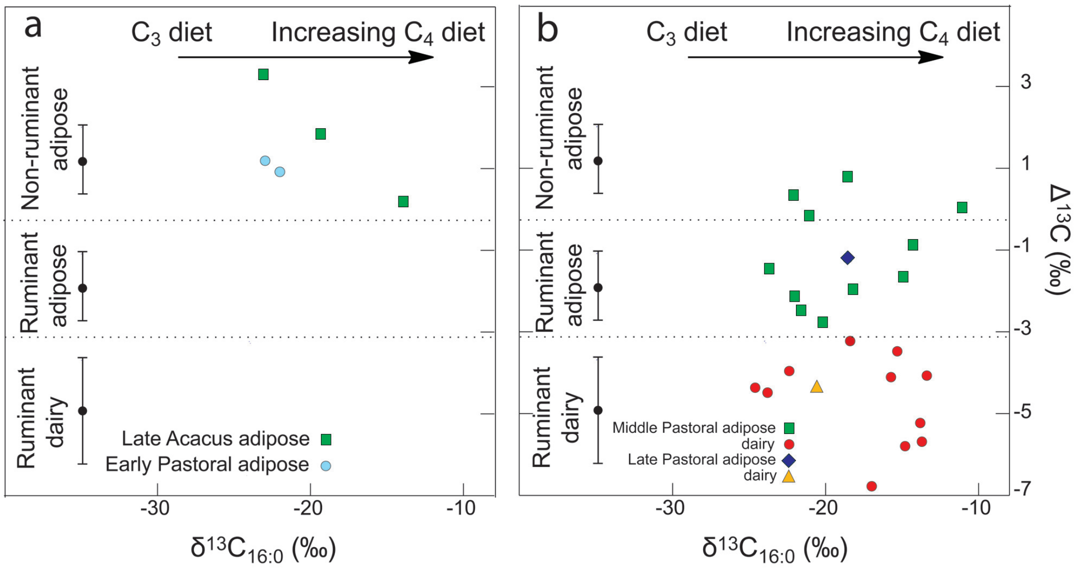

Fig. 6. Plot of the $\Delta^{13}C$ values for the archaeological fat residues (Late Acacus/Early Pastoral and Middle/Late Pastoral). Significantly, the residues originating from the Late Acacus and Early Pastoral periods (Plot a) do not contain dairy fats, and plot in the non-ruminant range, probably deriving from wild fauna. Plot b clearly demonstrates that extensive processing of dairy products in pottery vessels from this region begins in the Middle Pastoral period* c. *5200–3800 BC. The ranges shown here represent the mean ± 1 s.d. of the $\Delta^{13}C$ values for a global database comprising modern reference animal fats and fats from Africa, UK (animals raised on a pure C_3 diet) (*Dudd, Evershed 1998*), Kazakhstan (*Outram *et al.* 2009*), Switzerland (*Spangenberg *et al.* 2006*) and the Near East (*Gregg *et al.* 2009*), published elsewhere.

their secondary products. Of the two samples originating from the Late Pastoral period, one has a dairy fat source and the other a ruminant adipose fat origin.

Of particular note is the wide range of $\delta^{13}C$ values exhibited by the fatty acids, plotting across the range –25‰ to –10‰, perhaps suggesting that the animals giving rise to these fats had subsisted on an extensive range of different forages either comprised entirely of C_3 plants, varying combinations of C_3 and C_4 plants to a diet comprising wholly C_4 plants. This wide range of $\delta^{13}C$ values for these African potsherds is unprecedented, and points to differing pastoral modes of subsistence, such as vertical transhumance, by these prehistoric Saharan groups, as suggested by their settlement pattern based on summer sites in the lowland sand seas and winter sites (such as Takarkori) in the mountains (*di Lernia 2002*), probably in response to seasonal weather patterns. This pattern is confirmed by the recent isotope studies on human and faunal remains from Takakori rock shelter (*di Lernia, Tafuri 2013*) as well on cattle remains from Middle Pastoral ceremonial sites in the Messak plateau and surroundings (*di Lernia* et al. *2013*).

It is noteworthy that the remarkable preservation of organic material excavated from Takarkori rock shelter in the Libyan Sahara is also mirrored at the molecular level, with organic residues being identified in 80 out of the 81 potsherds analysed, sometimes in extremely high concentrations. The interpretation of 80 diagnostic absorbed organic residues from the ceramics demonstrates that these vessels were used frequently or intensively to contain or process commodities of animal and plant origin. In the Middle Pastoral Period (*c.* 5200–3800 BC) the animal products appeared to derive predominantly from dairy products (50%), although carcass fats (33%) were also extensively processed. Significantly, the residues originating from the Late Acacus and Early Pastoral periods (n = 5), where archaeological levels either do not contain faunal remains from domesticated animals (Late Acacus) or are quite rare (Early Pastoral), plot in the non-ruminant fat range, probably deriving from wild fauna found locally. The appearance of dairying fats in pottery correlates with the more abundant presence of cattle bones in cave deposits during the Middle Pastoral period and confirms the indirect evidence of dairying provided by the remarkable rock art of the region.

Conclusion

In summary, our findings corroborate the evidence of dairying practices by prehistoric Saharan herders as seen in the rock art of the region. The first appearance of domesticated fauna in the Tadrart Acacus archaeological record, radiocarbon dated from around 7400 uncal bp (*di Lernia 2013*), seems not to be paralleled at Takarkori by a developed exploitation of secondary products. Significantly, the chemical evidence for extensive processing of dairy products in pottery vessels in the Libyan Sahara dates to the Middle Pastoral period (*c.* 5200–3800 BC), confirming that milk and its secondary products played a significant part in the diet of these prehistoric pastoral people. The finding of dairy fat residues in pottery is consistent with milk from the domesticated animals being processed, thereby explaining why, in spite of lactose intolerance, milk products could be consumed by these people. It is noteworthy that stable carbon isotope analyses demonstrate that the animal sources of the fats processed in the ceramic vessels excavated from the Takarkori rock shelter subsisted on an extensive range of different forages comprised either completely of C_3 plants, varying combinations of C_3 and C_4 plants or a diet comprising wholly C_4 plants. This wide range of $\delta^{13}C$ values for these African potsherds is unprecedented and points to differing pastoral modes of subsistence and mobility (likely vertical transhumance) by these prehistoric Saharan groups, as suggested by archaeological evidence confirming their settlement pattern based on summer sites in the lowland sand seas and winter sites (such as Takarkori) in the mountains (*di Lernia 2002*). This would probably have been in response to seasonal weather patterns, although it is also possible that these animals may have originated from non-local regions, as shown in the nearby area of the Messak plateau and Edeyen of Murzuq (*di Lernia* et al. *2013*).

These results also confirm that domesticated cattle used as part of a dairying economy were present in north Africa during the 5th millennium BC, thus supporting the idea of an earlier ingression into the central Sahara (*Gifford-Gonzalez, Hanotte 2011; Marshall, Hildebrand 2002; di Lernia 2002*), possibly suggesting a local process of pastoral development, also based on the exploitation of secondary products. This is also consistent with the finding of the –13 910*T allele, associated with the LP trait in Europeans, across some Central African groups, such as the Fulbe of northern Cameroon (*Mulcare* et al. *2004*), supporting arguments for some movement of people, with their cattle, from the Near East into eastern Africa in the early-middle Holocene.

These new data provide a tantalising, yet emphatic, glimpse of the emergence of the dairy complex as a component of pastoralism in Africa. The stage is now set for a wider, continental scale, investigation using lipid biomarkers in pottery.

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