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Tracing pottery use and the emergence of secondary product exploitation through lipid residue analysis at Late Neolithic Tell Sabi Abyad (Syria)

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Late Neolithic settlements dating to around 7000 cal. BC are widespread in Upper Mesopotamia, however, the site of Tell Sabi Abyad is unique in the scale and quality of excavation, revealing an extensive architecture, huge numbers of domesticated animal bones, stone tools and potsherds. A previous study reported lipid residues in nearly 300 potsherds as part of a wider investigation of the origins of dairying in the Near East and Southeastern Europe. The aim of this paper is to interpret the organic residue findings in more detail, addressing such factors as the association of lipids in pottery with particular phases, ware types, and the faunal record. Overall, the recovery rate of lipids in sherds is low (14% of the sherds investigated in this study yielded detectable lipids) and the mean lipid concentration for sherds containing lipids is ca. 82 $\mu g\ g^{-1}$. These results are typical of sites from this period and general region (southern Mediterranean and Near East). Our interpretations indicate: (i) the use of specific ceramic categories of vessel for "cooking", (ii) clear evidence of the extensive heating of vessels is deduced from the presence of ketones, formed from the condensation of fatty acids, in some vessels, (iii) strong differences in recovery rates possibly reflecting differences in use between different pottery types, (iv) in particular the Dark-Faced Burnished Ware (DFBW) contained the highest frequency of residues (46% yielded detectable lipids), (v) degraded animal fats were detectable, as evidenced by fatty acids with $C_{18:0}$ in high abundance and in few cases tri-, di- and monoacylglycerols, (vi) the presence of abundant carcass fats is consistent with interpretations based on faunal assemblage of extensive meat exploitation, and (vii) four vessels dated to 6400 to 5900 cal BC yielded milk fat residues.

1. Introduction

Tell Sabi Abyad is located in Upper Mesopotamia on the river Balikh, a perennial tributary of the Euphrates (Fig. 1). It is an extremely valuable Neolithic site because of its long, unbroken sequence of cultural change covering the 7th and early 6th millennia cal. BC. The site is characterised by broad lateral exposures and well-preserved contexts and sequences of prehistoric inhabitation (Akkermans, 1993, 2013; Akkermans (Ed.), 1996; Akkermans et al., 2006, 2014; van der Plicht et al., 2011). The excavations have resulted in a particularly well-dated sequence, with successive culture-historical phases formally based mainly upon changes in the ceramic assemblage (Fig. 2). The stratigraphic sequence is also firmly supported by an extensive program of radiocarbon dating (van der Plicht et al., 2011; Akkermans and van der Plicht, 2014).

The current evidence indicates that people first settled at the site in the late 8th millennium during the Pre-Pottery Neolithic. They inhabited what is today the high, western half of the mound, excavated at Operations III, IV and V (Fig. 3). In the Early Pottery

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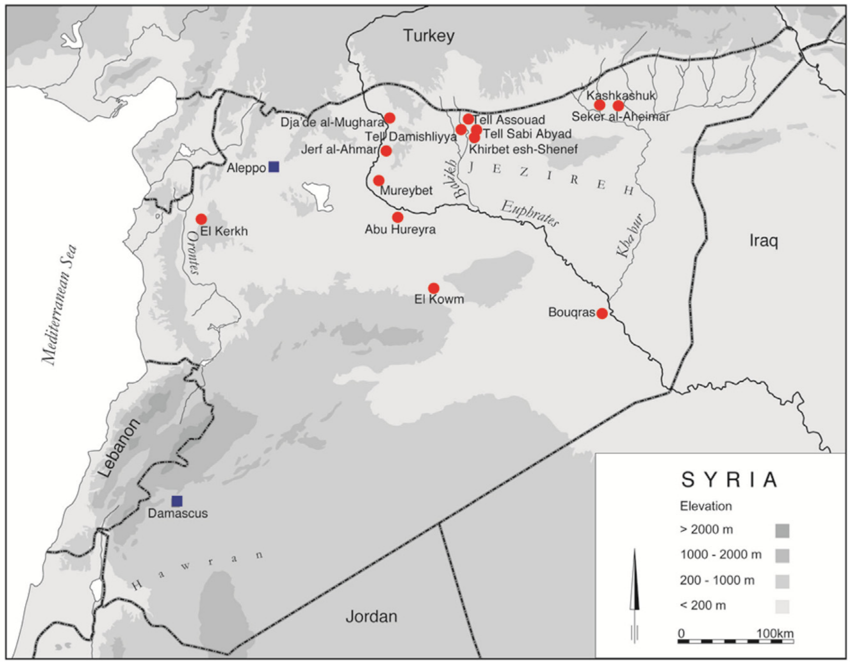

Fig. 1. Map of Upper Mesopotamia showing the location of Tell Sabi Abyad and other prehistoric sites (red dots). (For interpretation of the references to colour in this figure legend, the reader is referred to the web version of this article.)

Neolithic period a small settlement thrived at this location, characterized by a series of mainly rectilinear buildings separated by much open space. Around 6300 cal. BC, with the transition from Early Pottery Neolithic to Pre-Halaf, people moved the locus of the village eastward and constructed new buildings on the eastern slopes and at the foot of the earlier settlement mound, known as Operations I and II (Fig. 3). Characteristic for these later phases was a dense agglomeration of multi-roomed buildings that may have served as collective storage facilities to a partly semi-pastoral population (Akkermans and Verhoeven 1995; Akkermans et al. (Eds.), 2014, Duistermaat, 2013).

The inhabitants of Tell Sabi Abyad kept a range of domestic plants and animals, which initially included mainly pig and ovicaprids, with hunted species also present. Gradual shifts in patterns of animal exploitation were evident (Astruc and Russell, 2013; Cavallo, 2000; Russell, 2010). In particular, throughout the 7th millennium people relied more and more on the control of proto-domesticated herds of cattle. Eventually, at the end of the 7th millennium, in what is known as the Pre-Halaf period, this led to full domestication and a sustained separation of wild and domesticated populations of cattle (Cavallo, 2000; Russell, 2010). At about the same time, shifting cull patterns of ovicaprids and the introduction of spindle whorls (Rooijakkers, 2012) suggest an intensified exploitation of secondary products of wool and dairy products. The iconography of the Pre-Halaf to Early Halaf period shows stylized images of cattle and ovicaprids painted or sculpted on ceramics (Nieuwenhuyse, 2007), reflecting the rising cultural importance of these animals.

It is now accepted that organic residues are widely preserved in archaeological pottery and can provide information on both the use of vessels and wider economic activities, particularly those relating to the procurement of animal products. In relation to this pottery from Tell Sabi Abyad contributed to an extensive investigation, involving more than 2200 vessels from 25 Neolithic sites in the Near East and Southeastern Europe, in which organic residues were used to document the early evolution of milk use by prehistoric farmers (Evershed et al., 2008). The investigation included nearly 300 vessels from Tell Sabi Abyad. Herein, we present the detailed results of the organic residue analyses of these same vessels. The results are interpreted in the context of the functional uses of the pottery, dietary habits and culinary practices, and, specifically, the introduction of secondary products into the Late Neolithic economy (Sherratt, 1981, 1983). The latter are compared to interpretations of animal exploitation and herd management based on faunal analyses at the site. We also begin to investigate the factors contributing to the preservation of prehistoric residues, attempting to move towards contextualizing and interpreting their socio-economic context, and making comparisons with the Neolithic in other regions, *viz*. Northwestern Anatolia (Thissen et al., 2010; Türkekul-Bıyık, 2009; Türkekul-Bıyık and Özbal, 2008), Central Anatolia (Copley et al., 2006; Evershed et al., 2008; Pitter et al., 2013), western Iran (Gregg, 2010) and the southern Levant (Gregg et al., 2009).

2. Changing pottery assemblage

Indeed, a particular importance of Tell Sabi Abyad was its abundant pottery reflecting the fact that Upper Mesopotamia is home to some of the oldest pottery-using societies in the world. Pottery was first introduced in the region around 7000 cal. BC, marking the shift from the aceramic Early Neolithic (Pre-Pottery Neolithic B) to the Late Neolithic, also known as the Pottery Neolithic period (Akkermans, 1993; Arimura et al., 2000; Faura and Le Mière, 1999; Le Mière and Picon, 2003; Miyake, 2005; Nieuwenhuyse, 2006, 2007, 2009; Nieuwenhuyse et al., 2010; Nishiaki and Le Mière, 2005; Özdoğan, 2009; Tsuneki and Miyake, 1996).[1] During the 7th millennium BC pottery production gradually increased and by the end of the millennium ceramic vessels had truly become indispensable. Pottery production

[1] All dates in this paper are absolute calibrated BC.

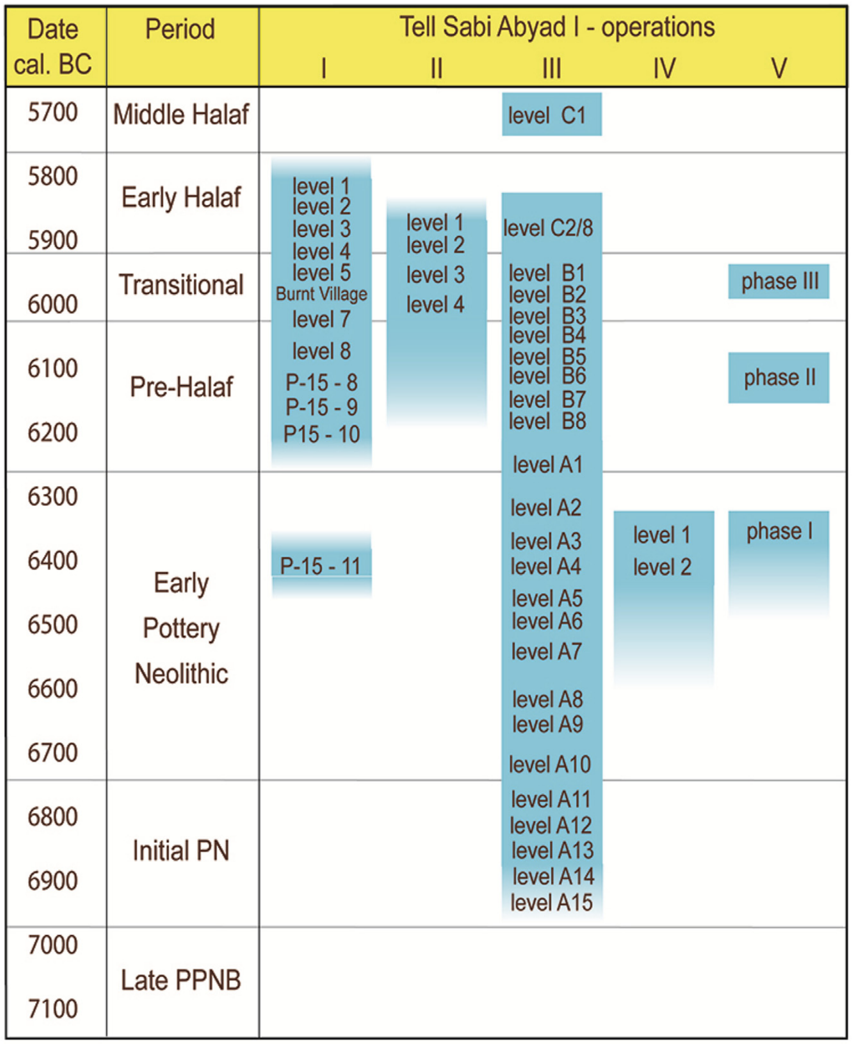

Fig. 2. The culture-history, absolute date and stratigraphy of the excavations at different locations at Tell Sabi Abyad. (after Nieuwenhuyse et al., 2010; van der Plicht et al., 2011).

diversified technologically, morphologically and stylistically, contributing to an expanding array of socio-economic and symbolic activities.

The Tell Sabi Abyad ceramic assemblage comprises a number of distinct wares, each employed for different activities. Wares are defined on the basis of their ceramic-technological properties or "chaîne opératoire" (Le Mière and Nieuwenhuyse, 1996; Nieuwenhuyse, 2007, n.d.). The majority of the pottery was a plant-tempered and coarsely-made known as Standard Ware (SW, Figs. 4.1–11), with simple shapes initially, but in the later Early Pottery Neolithic storage jars developed (Fig. 4.8). During the Pre-Halaf period SW gained new roles possibly for serving and displaying food and drink, suggested by elaborate decorations (Figs. 4.10–11). Throughout its existence SW pottery gained a wide range of uses, but is unclear if vessels were heated by use over fire. Le Mière and Picon (1991, 1999) have proposed that SW was inherently unsuitable for cooking but was nonetheless used occasionally, on an "ad hoc" basis, as a poor-quality cooking ware.

Other ceramic categories were more suited for activities involving heat. So-called Early Fine Ware (EFW) and Grey-Black Ware (GBW) were mineral-tempered burnished wares that emerged during the Early Pottery Neolithic (Figs. 5.15–16). The temper consisted of finely-ground calcite or basalt (Nilhamn et al., n.d.). Together with the regular wall thickness, the frequent lugs, the closed shape and the occasional presence of soot this suggests that they were used for cooking.[2] Mineral Coarse Ware (MCW) was first introduced in the Pre-Halaf period (Figs. 5.13–14). This burnished ware was made with a dense temper of coarsely-ground calcite, and mainly included hole mouth pots, very frequently carrying lugs. These properties almost make it a text-book case of a functionally specialized cooking ware. This interpretation is corroborated by the presence of soot (Le Mière and Picon, 1991; Le Mière and Nieuwenhuyse, 1996: 128). On the other hand, the strong permeability of this pottery, in spite of its strong burnish on either side, would make it less suitable for cooking (Daszkiewicz et al., 2000, 2003).

The so-called Dark-Faced Burnished Ware (DFBW) is an intriguing category appearing during the Pre-Halaf and Transitional periods (Figs. 5.1–12). DFBW was not made locally in the Balikh, and it may have come from the northern Levant (Bader et al., 1994;

[2] In the Halaf period Grey-Black Ware was no longer (exclusively) associated with cooking. This category changed its function and became a serving-display ware alongside the painted Fine Wares (Nieuwenhuyse, 2007).

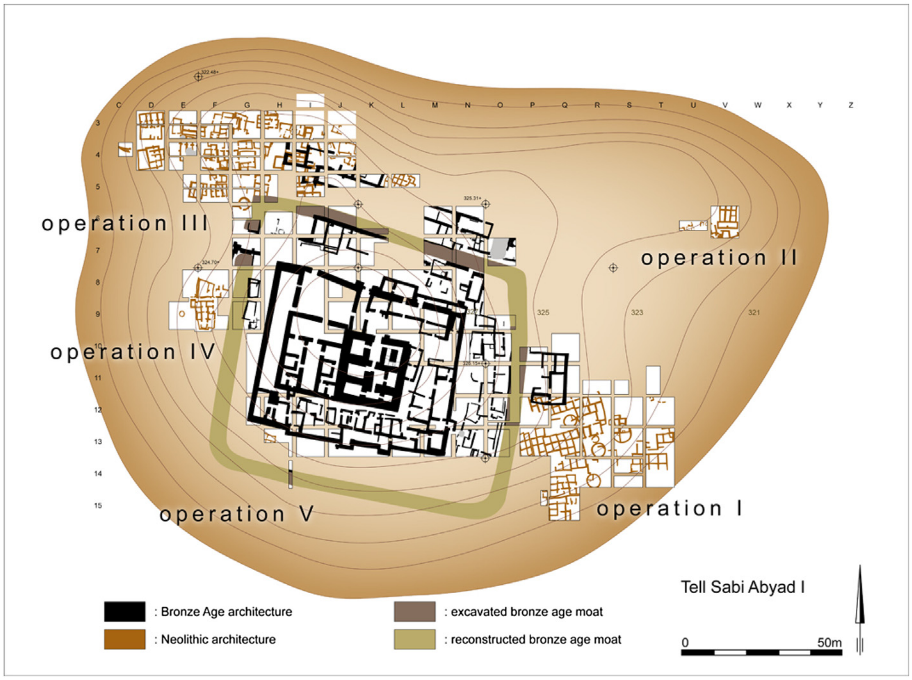

Fig. 3. The mound of Tell Sabi Abyad, showing the locations of Operations I to V.

Le Mière, 1989, 2000, 2001; Le Mière and Nieuwenhuyse, 1996: 126–7; Le Mière and Picon, 1987, 1999) or from southeastern Anatolia (Nieuwenhuyse, 2007: 82–85). Characterized by a strong mineral temper, burnished surfaces, and a compact fabric with reduced porosity, the vessels would have been resistant to both mechanical and thermal stresses. It is clear that DFBW was valued for its properties as superior cooking ware at Tell Sabi Abyad. A common practice for jars involved the careful removal of the neck after which the resulting hole mouth body was re-used as a cooking pot (Figs. 5.1–5). Frequent soot and the occasional preservation of visible residues attest to the frequent deployment of DFBW pots for cooking (Le Miè re and Nieuwenhuyse, 1996: 129; Nieuwenhuyse, 2007: 129–130).

In addition to these utilitarian wares, there were several elaborately-fashioned painted wares that served the consumption of food and drink and social display. The Standard Fine Ware (SFW, Figs. 4.12–17) represented a true novelty at the start of the 6th millennium BC. This pottery was made of a compact, carefully prepared clay (Nieuwenhuyse, 2007). Standard Fine Ware rose to prominence during the Transitional Period and came to dominate the ceramic assemblage by the Early Halaf (when it is termed Halaf Fine Ware). Typical for the Early Halaf period were the so-called "cream bowls": carinated, collared open serving vessels (Mallowan and Cruikshank-Rose, 1935). Alongside Standard Fine Ware, Orange Fine Ware (OFW, Fig. 4.18), technologically somewhat less refined, represented another stylistically-elaborate serving ware (Nieuwenhuyse, 2007).

3. Materials and methods

3.1. Pottery selection

A total of 287 sherds were investigated in this study. Almost half of the ceramic assemblage studied came from Operation II (n = 133), while the rest came from Operations I, III and IV (Table 1A). Operation II is thus over-represented *vis à vis* other excavated parts, but overall the samples are spatially well distributed across the site. However, while the sampling attempted to cover the various periods equally, because the stratigraphic analyses and absolute dating became available only after the sampling, this led to uneven representation of specific periods (see below). The sherds were not selected taking specific pottery forms into account or targeting specific pottery types (Thissen et al., 2010) and were mostly body sherds (Table 1), which could not generally be attributed to a specific vessel type. Only 4.5% of the assemblage samples were rim sherds. The selected sherds included jars, bowls, hole mouth pots and a few Standard Ware husking tray fragments.

The sampling included seven distinct wares (Table 1B). The majority of the samples were coarse, plant-tempered *Standard Ware* (SW, n = 94) and fine, painted *Standard Fine Ware* (SFW, n = 87). Also represented were *Dark-Faced Burnished Ware* (DFBW, n = 28), *Early Fine Ware* (EFW, n = 25), *Grey-Black Ware* (GBW, n = 21) and *Mineral Coarse Ware* (MCW, n = 25). Finally, five sherds of *Orange Fine Ware* (OFW) were included. Unfortunately, two sherds came without adequate description. All together almost the entire spectrum of wares that comprise the total ceramic assemblage during the Early Pottery Neolithic through Pre-Halaf to Transitional Period was represented.

In view of the long inhabitation sequence of Tell Sabi Abyad, two periods are well-represented: the Transitional Period ($n = 172$) and the Early Pottery Neolithic ($n = 102$). The Early Pottery Neolithic samples mainly come from the final stages of that period. Most come from Operation III, levels A2 ($n = 21$) or from mixed level A2/A3 contexts ($n = 12$). The oldest samples are thirteen sherds from Operation III, level A4, dated to 6455–6390 cal. BC. The Pre-Halaf period is severely underrepresented with only four samples (Table 1C). Two sherds came from topsoil contexts and seven were from a mixed Early Pottery Neolithic - Pre Halaf context. To summarize, this study documents the later stages of the Early Pottery Neolithic to the Transitional Period, from ca. 6400 to ca. 5900 cal. BC.

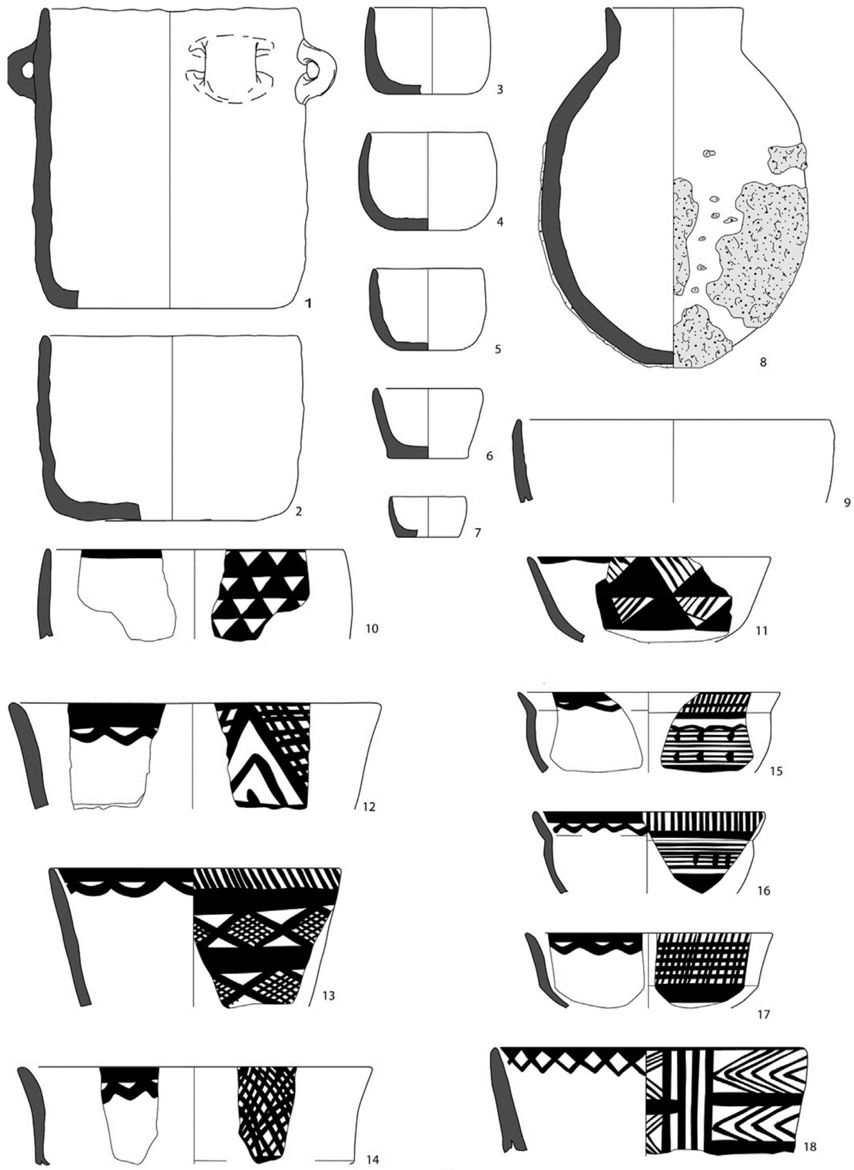

Fig. 4. Characteristic pottery types at Tell Sabi Abyad. 1–11: Standard Ware. 12–17: Standard Fine Ware. 18: Orange Fine Ware. 8: plastered. 10–18: painted (after Akkermans et al., 2006; Nieuwenhuyse, 2007).

3.2. Lipid extraction of pottery

Lipid analyses of potsherds were performed using our established protocol (Copley et al., 2003; Dudd and Evershed, 1998; Evershed et al., 2008), whereby ca. 2 g samples were taken and their surfaces cleaned using a modelling drill to remove any exogenous lipids (e.g. soil or finger lipids due to handling). The samples were then ground to a fine powder, accurately weighed and a known amount (20 μg) of internal standard (*n*-tetratriacontane) added. The lipids were extracted with a mixture of chloroform and methanol (2:1 *v*/*v*). Following separation from the ground potsherd the solvent was evaporated under a gentle stream of nitrogen to obtain the total lipid extract (TLE).

3.3. Gas chromatography (GC) and GC-mass spectrometry (GC–MS) of lipids

Portions (generally one fifth aliquots) of the extracts were then trimethylsilylated using *N*,*O*-bis(trimethylsilyl)trifluoroacetamide (20 mL; 70 °C; 1 h; Sigma–Aldrich Company Ltd., Gillingham, UK) and submitted directly to analysis by high temperature-gas chromatography (HTGC). Where necessary combined gas chromatography-mass spectrometry (GC–MS) analyses were also performed on trimethylsilylated aliquots of the lipid extracts to enable the elucidation of structures of components not identifiable on the basis of GC retention time alone.

HTGC analyses were performed on a Hewlet Packard 5890 series II gas chromatograph coupled to an Opus V PC with HP Chemstation software. The samples were injected into a fused silica capillary column (15 m × 0.32 mm i.d.) coated with a dimethyl polysiloxane stationary phase (J&W Scientific; DB1-HT, 0.1 μm film thickness). The temperature programme comprised a 2 min isothermal period at 50 °C followed by an increase to 350 °C at 10 °C min^{-1}; the temperature was held at 350 °C for 10 min. A FID was used to monitor the column effluent. The carrier gas used was hydrogen with a column head pressure of 10 psi. Quantification was achieved

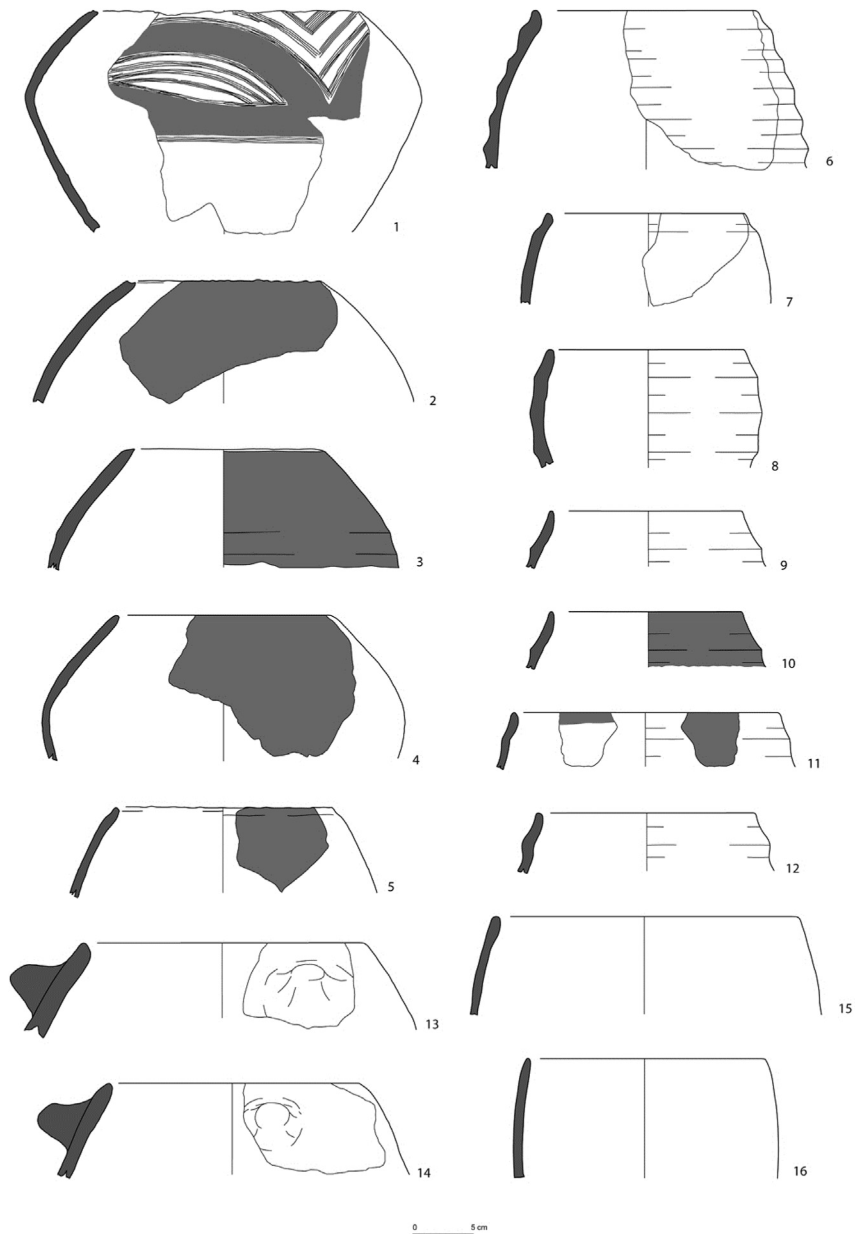

Fig. 5. Characteristic pottery types at Tell Sabi Abyad. 1–12: Dark-Faced Burnished Ware. 13–14: Mineral Coarse Ware. 15–16: Early Fine Ware. 1: painted-and-incised. 2–5, 10–11: red slipped (after Akkermans et al., 2006; Nieuwenhuyse, 2007).

by the internal standards method.

Compound identification was achieved using a Varian 3400 GC, comprising a SPI injector coupled, via a high temperature transfer line, to a triple stage quadrupole MS (TSQ 700, Finnigan MAT). The GC conditions were similar to those used for the HTGC analyses. The temperature of the interface between the GC and MS was held at 350 °C. The MS operating conditions were as follows: ion source 240 °C, filament current 400 μA, electron voltage 70 eV, scanning the range *m/z* 50–850 using q3 at a scan rate of 1 scan s^{-1} employing He as the carrier gas. Data acquisition and processing were carried out using an ICIS data system.

3.4. GC-combustion-isotope ratio-MS

In order to determine $\delta^{13}C$ values of the fatty acids, TLEs were treated with methanolic sodium hydroxide (5% *v/v*, 70 °C, 1 h). Following neutralisation, lipids were extracted into hexane and the solvent reduced by rotary evaporation. Fatty acid methyl esters (FAMEs) were prepared by reaction with BF_3-methanol (14% *w/v*; 2 mL; Sigma–Aldrich, Gillingham, UK, 70 °C, 1 h). The methyl ester derivatives were extracted with diethyl ether and the solvent removed under nitrogen. The FAMEs were re-dissolved into hexane for analysis by GC and gas chromatography-combustion-isotope ratio mass spectrometry (GC-C-IRMS). The majority of GC-C-IRMS analyses of fatty acids from potsherds were performed using a Varian 3400 GC coupled to a Finnigan MAT Delta-S IRMS via an extensively modified Finnigan MAT Type I combustion interface, Cu and Pt wires (0.1 mm o.d.) in an alumina reactor (0.5 mm i.d.). The reactor temperature was maintained at 860 °C and the mass spectrometer source pressure was 6×10^{-6} mbar. Faraday cups were used for the detection of ions of *m/z* 44 ($^{12}C^{16}O_2$), 45 ($^{13}C^{16}O_2$ and $^{12}C^{17}O^{16}O$) and 46 ($^{12}C^{18}O^{16}O$). The GC column used was a silica

Table 1
Summary of Tell Sabi Abyad organic residue analyses in: A. Excavation areas of the site. B. Pottery wares. C. Archaeological periods.

	Potsherds analysed					Residues detected	
	Unknown	Base	Body	Rim	Total	n	%
A. Operation							
I	9	2	20	10	41	11	27%
II	0	8	124	1	133	25	19%
III	0	32	28	1	61	3	5%
IV	0	15	36	1	52	2	4%
Total	9	57	208	13	287	41	14%
B. Ware							
- (no information)	2	0	0	0	2	0	0%
Dark-Faced Burnished Ware	5	0	19	4	28	13	46%
Early Fine Ware	0	5	20	0	25	1	4%
Grey-Black Ware	0	3	17	1	21	2	9%
Mineral Coarse Ware	1	1	23	0	25	6	24%
Orange Fine Ware	0	0	5	0	5	2	40%
Standard Fine Ware	1	1	83	2	87	14	16%
Standard Ware	0	47	41	6	94	3	3%
Total	9	57	208	13	287	41	14%
C. Period							
Topsoil	0	2	0	0	2	0	0%
Transitional	8	10	143	11	172	34	20%
Pre-Halaf	1	0	3	0	4	2	50%
Early Pottery Neolithic/Pre-Halaf	0	3	4	0	7	0	0%
Early Pottery Neolithic	0	42	58	2	102	5	5%
Total	9	57	208	13	287	41	14%

capillary column (50 m × 0.32 i.d.) coated with a polyethylene glycol stationary phase (CP Wax-52 CB, 0.25 μm film thickness). The temperature programme comprised a 1 min isothermal period at 50 °C followed by an increase to 150 °C at 15 °C min^{-1} followed by an increase to 220 °C at 4 °C min^{-1} then a further increase to 240 °C at 15 °C min^{-1} with an isothermal period of 10 min.

4. Results and discussion

4.1. General assessment of the organic residues

Of the 287 sherds submitted to solvent extraction 41 were shown by HTGC to contain detectable lipids that could be confidently interpreted to be of archaeological origin (Table 2). Compared to north-western Europe this recovery rate (14%) is low but considering that no targeted sampling was adopted, the figure is comparable with other Mediterranean Neolithic sites. For example, at Çatalhöyük initial investigations revealed lipids in 18% of the sherds, rising to 36% with targeted sampling of cooking vessels (Copley et al., 2006; Pitter et al., 2013). Additionally, Late Neolithic pottery sherds from Barcın Höyük in northwestern Turkey showed 24% of sherds to contain detectable lipid residues (Thissen et al., 2010: 166).

The lipid profiles from the Tell Sabi Abyad sherds showed a remarkable constancy being dominated by free fatty acids, sometimes as the sole components (Fig. 6A), in other cases accompanied by low abundances of monoacylglycerols, diacylglycerols and triacylglycerols (Fig. 6B–C). A third class of lipids seen in 9 sherds contains a range of mid-chain ketones eluting in the retention time range of the internal standard (Fig. 6B–C). The presence of the aforementioned classes of lipid is typical of degraded animal fats particularly on account of the high abundance of the $C_{18:0}$ fatty acid (Evershed et al., 2002; Evershed, 2008). The low abundance of $C_{18:1}$ fatty acid, the main fatty acid in fresh animal fats, is consistent with the sensitivity of such compounds to oxidative loss during vessel use and burial; indeed the low abundance of unsaturated fatty acids provides an important quality control criterion in confirming the indigeneity of archaeological animal fat residues. The odd-carbon numbered ketones ranging from C_{31} to C_{35} are a diagnostic group of compounds forming by heating fats above ca. 300 °C (Evershed et al., 1995; Raven et al., 1997; Evershed, 2008). The presence of mid-chain ketones is a common feature of Neolithic pottery in Europe, the Near East and Eurasia and points to the importance of processed animal products in a variety of dietary and quasi-industrial roles in the Neolithic life. No biomarkers characteristic of plant material have been detected in any of the sherds. However, low concentrations of lipids in plants compared to animal products can preclude the identification of plant material when mixed with animal products.

The added internal standard allows quantification of the lipid recoveries from the pottery from Tell Sabi Abyad (Table 2, Fig. 7). The concentrations range from none detected in the vast majority of vessels to 580 μg g^{-1}, although the latter is exceptional for this assemblage. The average concentration of lipids in sherds containing significant concentration of lipids (>5 μg g^{-1}) is 82 μg g^{-1}. These concentrations and rates of recovery of lipids are typical for the wider region (cf. Çatalhöyük in Central Anatolia). The lipid concentrations will be discussed in more detail in relation to different wares and pottery type in the section below.

4.2. Distribution of lipid concentrations

The sherds containing detectable lipids are far from equally distributed across the site. The percentage of lipid residues is highest in Operation I (27%), followed by Operation II (19%). Operations III and IV produced significantly lower recovery rates (respectively, 5% and 4%; Table 1A; Operations I/II and III/IV, one-sample χ^2 test, $p < 0.001$). The nature of the depositional setting likely plays a role in the preservation of lipids. For example, the sherds from Operation I and II were recovered from layers excavated below a thick horizon of later deposition. In contrast, the sherds from Operation III and, in particular, Operation IV were recovered from depositions lying closer to the surface of the mound, probably contributing to lower lipid recovery rates. However, these Operations exposed different archaeological periods which may be more important in explaining the differences in lipid recovery rates from the sherds (Table 1B). Most significant was the higher recovery rate of *ca.* 20% in body sherds from the Transitional

Table 2
Tell Sabi Abyad residue analysis: pottery samples with detectable lipid residues.

Lab number	Operation	Provenance	Level	Period	Date (cal. BC)	Ware	Shape	Lipids (mg g^{-1})	Lipids detected	$\delta^{13}C_{16:0}$ (‰)	$\delta^{13}C_{18:0}$ (‰)	$\Delta^{13}C$ (‰)	Commodity identified
SAB1	I	T14(144) 125:31	6/7a	Transitional	6000–5900	DFBW	Rim	0.04	FA	−24.4	−26.8	−2.4	Ruminant carcass
SAB2	I	T12 (46) 96:8	LB/6	Transitional	6000–5900	DFBW	Body	0.18	FA	−24.8	−29.2	−4.4	Ruminant dairy
SAB6	I	R12(114)219:2	8	Pre-Halaf	6225–6000	DFBW	Body	0.58	FA, TAG(tr)	−27.2	−29.1	−1.9	Ruminant carcass
SAB9	I	Q13 (139) 375:1	6	Transitional	6000–5900	OFW	Body	0.08	FA	−25.9	−29.0	−3.1	Ruminant carcass
SAB11	I	S14 (108) 299:1	6	Transitional	6000–5900	OFW	Body	0.14	FA	−25.8	−28.4	−2.6	Ruminant carcass
SAB21	I	R12 (94) 156:100	8	Pre-Halaf	6225–6000	DFBW	–	0.03	FA	−26.4	−27.1	−0.7	Ruminant carcass
SAB27	I	R13 (146) 353:100	6/7b	Transitional	6000–5900	DFBW	–	0.31	FA, MAG, DAG, TAG	−26.6	−28.1	−1.5	Ruminant carcass
SAB28	I	R13 (169) 373:100	6/7b	Transitional	6000–5900	DFBW	–	0.04	FA	−26.3	−27.7	−1.4	Ruminant carcass
SAB29	I	R13 (146) 353:101	6/7b	Transitional	6000–5900	DFBW	–	0.01	FA, K(tr)	−26.4	−27.8	−1.4	Ruminant carcass
SAB31	I	R13 (204) 494:1	6	Transitional	6000–5900	DFBW	Body	0.02	FA	−23.4	−26.5	−3.1	Ruminant carcass
SAB35	I	Q13 (139) 380:6	6	Transitional	6000–5900	MCW	–	0.01	FA(tr), K, TAG(tr)	−25.5	−27.3	−1.8	Ruminant carcass
SAB54	II	V6 (41) 106:1	3/4	Transitional	6000–5900	MCW	Body	0.01	FA(tr), TAG(tr)				
SAB55	II	V6 (35) 100:3	?	Transitional	6000–5900	MCW	Body	0.02	FA(tr), K(tr), TAG(tr)	−25.0	−27.0	−2.0	Ruminant carcass
SAB56	II	V6 (49) 153:1	3/4	Transitional	6000–5900	MCW	Body	0.03	FA(tr), TAG	−22.1	−25.3	−3.2	Ruminant carcass
SAB57	II	V6 (52) 119:2	3/4	Transitional	6000–5900	SFW	Body	0.01	FA(tr)				
SAB62	II	V6 (35) 91:1	?	Transitional	6000–5900	DFBW	Body	0.40	FA	−25.5	−24.4	1.1	Non-ruminant
SAB63	II	V6 (57) 127:1	3/4	Transitional	6000–5900	MCW	Body	0.12	FA	−26.4	−27.1	−0.7	Ruminant carcass
SAB65	II	V6 (49) 153:2	3/4	Transitional	6000–5900	DFBW	Rim	0.01	FA(tr), K(tr), TAG(tr)	−22.5	−24.5	−2.0	Ruminant carcass
SAB94	II	V6 (21) 59:4	4	Transitional	6000–5900	DFBW	Body	0.29	FA	−25.9	−27.6	−1.6	Ruminant carcass
SAB112	II	V6 (19) 55:2	4	Transitional	6000–5900	SFW	Body	0.11	FA	−26.4	−29.8	−3.4	Ruminant dairy?
SAB113	II	V6 (21) 59:9	4	Transitional	6000–5900	SFW	Body	0.01	FA	−26.0	−28.4	−2.3	Ruminant carcass
SAB122	II	V6 (34) 72:9	4	Transitional	6000–5900	SFW	Body	0.04	FA	−25.4	−28.8	−3.5	Ruminant dairy?
SAB127	II	V6 (28) 69:1	4?	Transitional	6000–5900	MCW	Body	0.01	FA(tr), TAG(tr)	−22.7	−24.7	−2.0	Ruminant carcass
SAB154	II	V6 (7) 20:7	4	Transitional	6000–5900	SFW	Body	0.02	FA	−24.7	−25.2	−0.5	Ruminant carcass
SAB158	III	G3 (19) 89:1	A4	Early Pottery Neolithic	6455–6390	SW	Rim	0.02	FA, TAG(tr)	−25.1	−27.0	−2.0	Ruminant carcass
SAB173	II	V6 (23) 48:1	4	Transitional	6000–5900	DFBW	Body	0.45	FA, TAG(tr)	−24.8	−28.6	−3.9	Ruminant dairy
SAB175	II	V6 (18) 47:6	4	Transitional	6000–5900	SFW	Body	0.08	FA				
SAB189	II	V6 (18) 42:4	3	Transitional	6000–5900	SFW	Body	0.01	FA(tr)				
SAB190	II	V6 (23) 48:4	4	Transitional	6000–5900	DFBW	Body	0.04	FA	−26.0	−26.2	−0.2	Non-ruminant
SAB193	III	G3 (17) 107:2	A2/A3	Early Pottery Neolithic	6395–6330	SW	Body	0.01	FA				
SAB209	II	V6 (4) 10:6	3	Transitional	6000–5900	SFW	Body	0.04	FA	−24.8	−27.2	−2.4	Ruminant carcass
SAB214	II	V6 (10) 17:8	4	Transitional	6000–5900	SFW	Body	0.02	FA	−27.1	−31.4	−4.3	Ruminant dairy
SAB219	II	V6 (4) 10:8	3	Transitional	6000–5900	SFW	Body	0.01	FA(tr)				
SAB220	II	V6 (21) 59:5	4	Transitional	6000–5900	GBW	Base	0.01	FA(tr)				
SAB221	II	V6 (7) 16:8	4	Transitional	6000–5900	SFW	Body	0.01	FA(tr)				
SAB223	II	V6 (8) 18:16	4	Transitional	6000–5900	SFW	Body	0.01	FA(tr)				
SAB225	II	V6 (8) 18:17	4	Transitional	6000–5900	SFW	Body	0.01	FA	−24.2	−27.1	−2.9	Ruminant carcass
SAB229	II	V6 (3) 14:7	3	Transitional	6000–5900	SFW	Body	0.06	FA(tr)				
SAB252	III	G3 (17) 51:1	A2/A3	Early Pottery Neolithic	6395–6330	SW	Base	0.04	FA, K, TAG(tr)	−24.5	−23.4	1.1	Non-ruminant
SAB263	IV	E9 (11) 32:14	–	Early Pottery Neolithic	6400–6330	GBW	Body	0.01	FA(tr), K(tr), TAG(tr)	−25.9	−31.9	−6.0	Ruminant dairy
SAB270	IV	E9 (15) 44:7	–	Early Pottery Neolithic	6400–6330	EFW	Body	0.01	FA(tr), K(tr), TAG(tr)				

Key: FA fatty acids, MAG monoacylglycerols, DAG diacylglycerols, TAG triacylglycerols, K ketones, (tr) traces.

period from Operations I and II, ($n = 143$) compared to the Early Pottery Neolithic, exposed in Operations III and IV, where only 5% yielded detectable lipids (3 residues extracted from 58 sherds; one-sample χ^2 test, $p < 0.05$). These differences in lipid concentrations are shown in Fig. 7A.

While the age of the pottery might be the most obvious influence on lipid preservation, the major factor affecting lipid concentrations appears to be the type of pottery investigated, more specifically the ware type (Table 1C, Table 3, Fig. 7B). Around 46% of all DFBW sherds (13/28 sherds) produced significant lipid residues with high lipid concentrations also being highest in this ware type (Fig. 7B). These findings corroborate the interpretations from earlier work that this pottery type was used for cooking. This is further supported by the detection of ketones in two of the 28 DFBW sherds studied indicating that these sherds were heated above ca. 300° C during their lifetime of use. Rim sherds and body sherds yield a higher detection rate than bases (one-sample χ^2 test, $p < 0.05$; Charters et al., 1993). Table 4.

Above average lipid concentrations for the assemblage were detected in a high proportion (24%, 6/25 sherds) of the sherds of the other main cooking ware, MCW (Fig. 7B). These findings appear to confirm the influence of pottery use, in this case cooking, on the

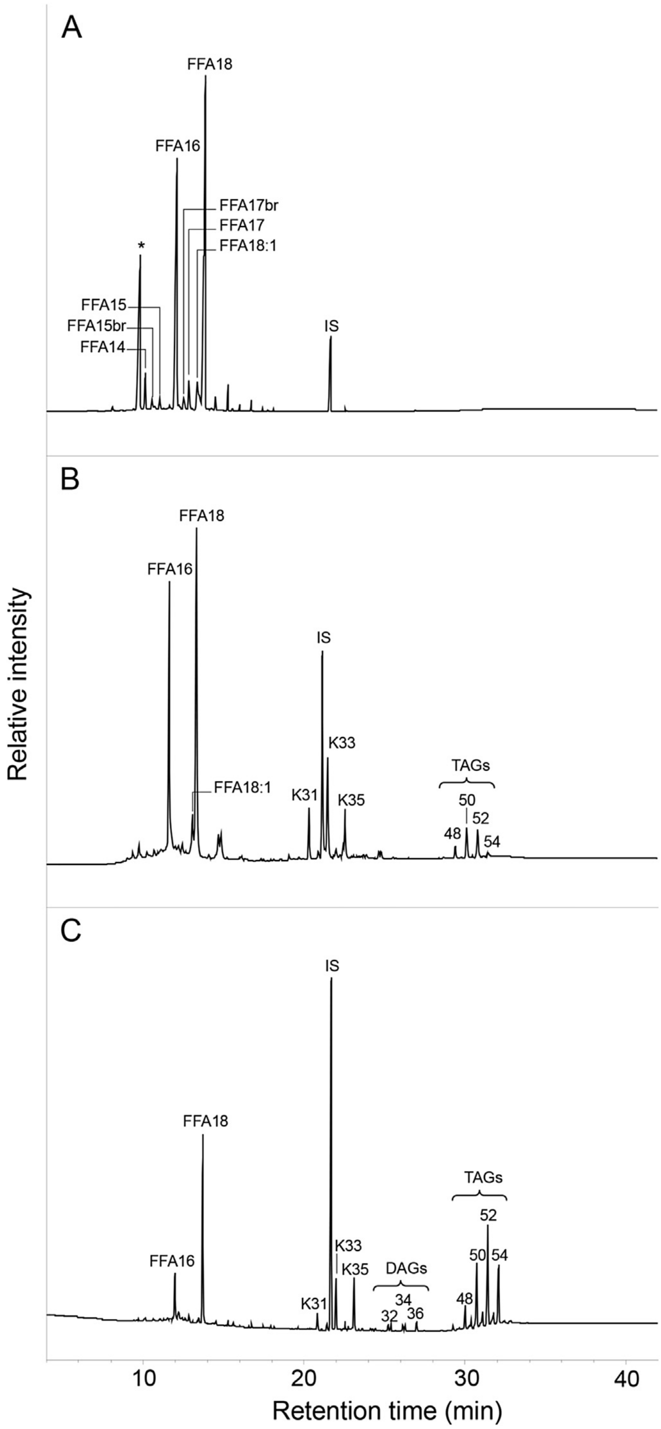

Fig. 6. Partial gas chromatograms of TLEs from pottery from Tell Sabi Abyad typical of (A) a degraded ruminant animal fat (SAB2) and (B)/(C) well preserved animal fat (SAB252 and SAB55, respectively). Abbreviations: FFA N:i, free fatty acids with N carbon atoms and i unsaturation; br, branched; K, mid-chain ketones with 31, 33 and 35 carbon atoms; MAG, monoacylglycerols; DAG, diacylglycerols; TAG, triacylglycerols, with M, acyl carbon number; IS, internal standard (*n*-tetratriacontane); * plasticizer.

absorption of lipids. The recovery rate of lipids from MCW sherds comparable to that of DFBW (one-sample χ^2 test, $p > 0.05$). In contrast, the two mineral-tempered wares typical for the Early Pottery Neolithic, GBW and EFW, exhibited appreciably lower recovery rates at <10% and 4%, respectively (DFBW/MCW and GBW/EFW, one-sample χ^2 test, $p < 0.01$).

Only 3% (3/94 sherds) of coarse plant-tempered SW sherds yielded detectable lipids (Fig. 7B). This recovery rate is very similar to what was observed at Çatalhöyük, where lipid concentrations in organic-tempered wares were much lower than in mineral-tempered wares (Pitter et al., 2013: 198). The low recovery rate in SW sherds corroborates the hypothesis that this pottery type was occasionally used as 'ad hoc' cooking ware, in spite of disadvantageous performance properties (Le Mière and Picon, 1991, 1999). The only three SW sherds yielding detectable lipids all came from the final stages of the Early Pottery Neolithic, the same period that also yielded the two EFW and GBW sherds with detectable residues. The presence of lipid residues in those Early Pottery Neolithic sherds suggest that people apparently already cooked with pottery containers, but perhaps not yet as often as they would during the Transitional stage. Interestingly, the two painted serving wares from the Transitional period, SFW and OFW, also produced a relatively high recovery rate of 17% (16/92 sherds in total). Petrographic studies have demonstrated that the fine and compact SFW does not transfer heat efficiently and that SFW pots would break if used as cooking vessels (Nieuwenhuyse, 2007). Hence, the presence of lipid residues in those SFW sherds suggests a cold liquid contact (serving or storing) rather that than a hot organic liquid processing (cooking).

4.3. Lipid residues versus archaeozoological record

The stable carbon isotope compositions (δ^{13}C values) of the predominant fatty acids ($C_{16:0}$ and $C_{18:0}$) of residues identified as animal fats and extracted from pottery vessels were determined to allow classification to commodity group (e.g. non-ruminant fat, ruminant adipose fat and ruminant dairy fat). δ^{13}C values were compared to a global modern reference animal fat database assembled from animals from Africa (Dunne et al., 2012), the UK (Copley et al., 2003; animals raised on a pure C_3 diet), Kazakhstan (Outram et al., 2009), Switzerland (Spangenberg et al., 2006) and the Near East (Gregg et al., 2009). The $\Delta^{13}C$ ($= \delta^{13}C_{18:0} - \delta^{13}C_{16:0}$) value was used to suppress the influence of varying abundances of C_3/C_4 plants in the animals' diets and aridity effects (Copley et al., 2003; Dunne et al., 2012). The δ^{13}C values recorded for the $C_{16:0}$ fatty acid (−27.2 to −22.1‰) are more depleted by > 4‰ than those from ruminant animal fats raised on a pure C_3 diet (−31.2 to −27.8‰) pointing to a significant C_4 and/or aridity influence on the diets of the animals (Fig. 8).

Table 2 and Fig. 8 summarise the animal fat classifications based on the δ^{13}C values, revealing the dominance of ruminant animal fats, with ruminant carcass fats being by far the most abundant class of lipid residue, accounting for 20 of the 41 residues detected. Four of the degraded fats were dairy fat residues with two others lying on the borderline of the carcass and dairy fat ranges, which may indicate they are mixture (Table 3). Hence, ca. 10% of all the animal fat residues detected were determined to be dairy fats. Two of these dairy fats were attested in DFBW (samples SAB2 and SAB173) and one in GBW (sample SAB263) – pottery wares suitable for cooking (Le Miè re and Nieuwenhuyse, 1996: 129; Nieuwenhuyse, 2007: 129–130). In these heavy-duty vessels dairy products were apparently processed involving heat. But the analyses also yielded one example of a SFW sherd yielding ruminant dairy lipid residue (sample SAB214) suggesting that this fragile vessel was used for storing/serving dairy products. The earliest evidence for milk in Sabi Abyad comes from a GBW bodysherd recovered from Operation IV and dating to the final stages of the Early Pottery Neolithic, ca. 6400–6330 BC. The DFBW sherds from the Transitional Period are dated to ca. 6000–5900 BC. Presently these constitute the earliest evidence for milk use attested in Upper Mesopotamia, possibly in the Middle East as a whole.

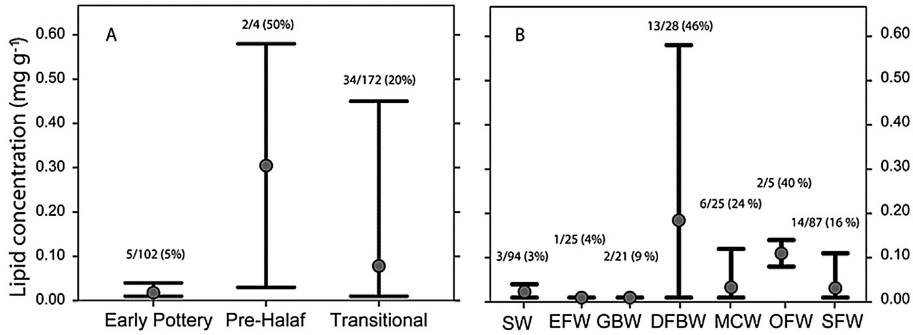

Fig. 7. Lipid concentrations (mg g^{-1}), numbers of residues detected and recovery rates by (A) archaeological period and (B) pottery type (ware).

Table 3
Tell Sabi Abyad residue analysis. Detectable residue types by ceramic ware.

	Ware							
	DFBW	EFW	GBW	MCW	OFW	SFW	SW	Total
Unidentified	.	1	1	1	.	7	1	11
Non-ruminant	3	.	.	1	.	1	1	6
Ruminant carcass	8	.	.	4	2	3	1	18
Ruminant dairy	2	.	1	.	.	1	.	4
Mixture ruminant fats?	.	.	.	.	.	2	.	2
Total	13	1	2	6	2	14	3	41

Table 4
Tell Sabi Abyad residue analysis. Results by fragment type.

	None detected		Residues detected		Total	
- (no information)	4	44%	5	56%	9	100%
Base	55	97%	2	3%	57	100%
Body	177	85%	31	15%	208	100%
Rim	10	77%	3	23%	13	100%
Total	246	86%	41	14%	287	100%

These findings are consistent with the work carried out by Vigne and Helmer (2007) pointing at even earlier evidence for dairying practices based upon slaughtering profiles. In fact, ovicaprids were used for a mixed milk and meat production in the Middle East already during the Late and the Early PPNB (8th millennium BC). Only few kill-off patterns for cattle are available in the Near East for the Middle and Late PPNB, but they also suggest that cattle were exploited for milk at the time. Archaeozoological studies are indeed a powerful means of unravelling herding practices and milk exploitation at archaeological sites. They allow the contextualization of the foodstuffs present as lipid residues in sherds and, as lipid residue analyses are not species-specific, a tentative identification of animal species represented in the residues.

The faunal analyses at Tell Sabi Abyad cover a timespan from ca. 6900 to ca. 5900 cal BC, from the Initial Pottery Neolithic into the Early Halaf period (Cavallo, 2000; Russell, 2010). The faunal investigations suggest continuous, gradual shifts in animal exploitation over the 7th and early 6th millennia. These sequential changes have been grouped into eight ‘animal exploitation phases’ (AEP), each phase characterized by a specific species composition and exploitation pattern (Russell, 2010: 239–240, 274). Sherds analysed in this study were sampled from the AEP IV to VII (Fig. 9), allowing investigations of the shift in the exploitation of ovicaprids (domestic sheep and goats) and the domestication of cattle.

Ovicaprids dominated the faunal assemblage throughout. Both postcranial fusion and mandibular tooth wear show rather similar mortality profiles for each animal exploitation phase, yet with subtle changes through time. AEP I to IV show a culling age of approximately two years of age with very few animals over three

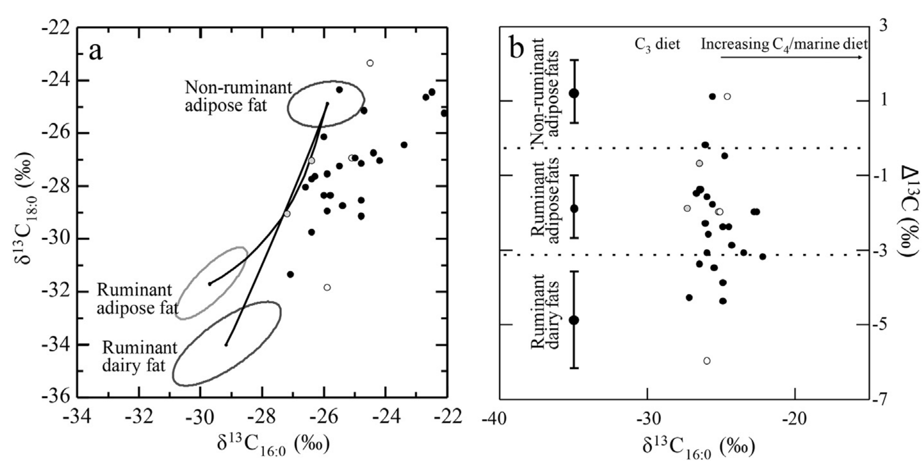

Fig. 8. Graphs showing: a. $\delta^{13}C$ values for the $C_{16:0}$ and $C_{18:0}$ fatty acids prepared from animal fat residues extracted from sherds from the Early Pottery Neolithic (white dots), Pre-Halaf (grey dots) and Transitional (black dots) periods. The three fields correspond to the $P = 0.684$ confidence ellipses for animals raised on a strict C_3 diet in Britain (Copley et al., 2003). Each data points represent an individual vessel. The analytical error (±0.3‰) is approximately the size of the points on the graph. b. $\Delta^{13}C$ values from the same potsherds. Ranges show the mean ± 1 s.d. for a global database comprising modern reference animal fats from United Kingdom (animals raised on a pure C_3 diet), Africa, Kazakhstan, Switzerland and the Near East (Dunne et al., 2012).

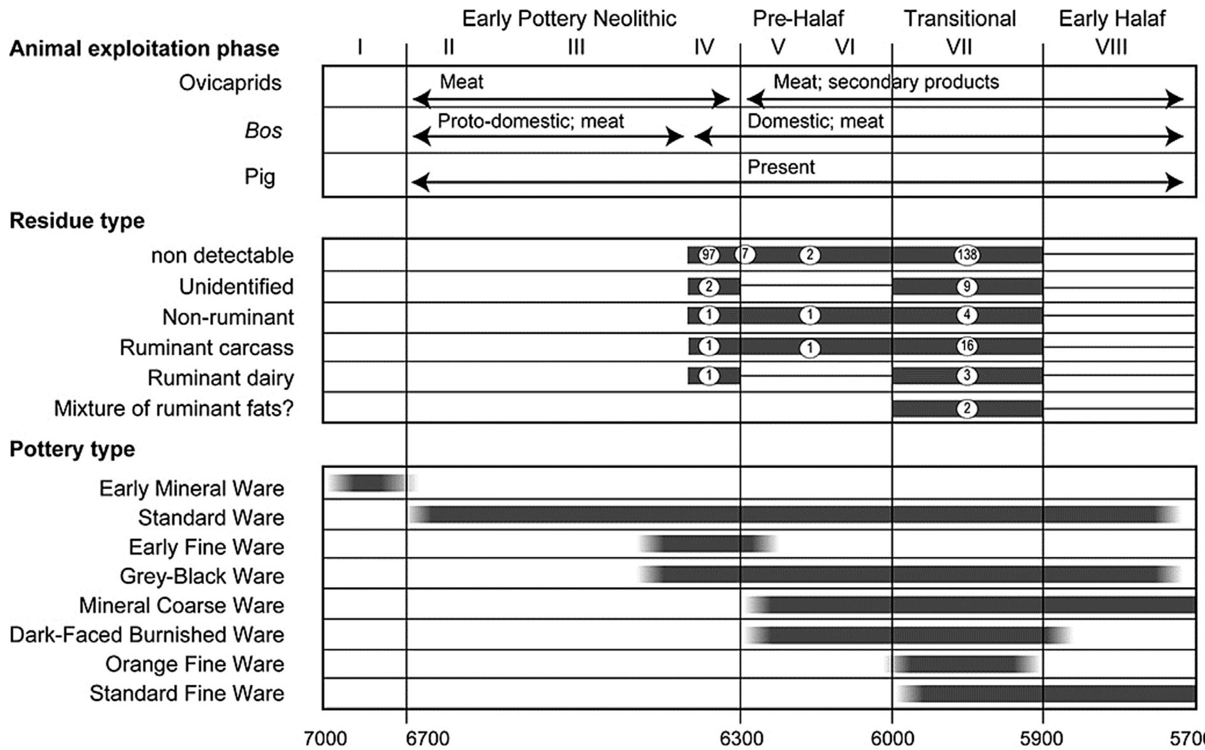

Fig. 9. Synchronizing presence/absence of pottery types at Tell Sabi Abyad (Wares; lower), organic residue results (center) and main trends in the exploitation of ovicaprids and cattle (upper).

years of age present. By AEP V–VI in the Pre-Halaf period, the main culling age shifts by one year to around three years of age with more animals living up to four years of age and older. In AEP VII–VIII, the Transitional and Early Halaf periods, this trend continues with even more animals living over four years of age. These mortality profiles suggest that in the oldest levels ovicaprid husbandry was geared primarily towards meat production, with the majority of animals being culled at the prime meat age of two years and only a small number of breeding stock maintained. Through time this shifted to a mixed economy of both meat and secondary product production.

The archaeozoological studies from Sabi Abyad then suggest a move from primarily meat to meat plus milk and fibre production during AEP V at around 6225 BC, synchronizing well with the first lipid residues in the pottery. In the Pre-Halaf period levels (Operation III levels A1-B4, AEP V–VI), faunal analyses points to an increased emphasis on the production of milk. The lamb (or kid) would have been kept during the lactation period and some females culled because of decreased milk yield or lamb production at two to four years of age. Apart from dairy products the hair or fleeces of ovicaprids appear to have become more important as the numbers of older animals in the faunal assemblage increased as well. The emphasis on wool is also suggested by the synchronous introduction of spindle whorls in the Pre-Halaf period (Rooijakkers, 2012). In the Early Halaf period (AEP VIII) the faunal signal for intensified secondary product production becomes even more pronounced with milk and fleece production perhaps taking priority over meat production in sheep and goats.

Bos remains were found in all levels, cattle being the second most common taxon in the levels contemporaneous with the sherds where lipid residue analyses were carried out (AEP IV to VII). The sequence documents a long-term process of gradually-increasing control over aurochs, starting in AEP IV and eventually resulting in the complete domestication of this animal in AEP V (Russell, 2010). In the Initial Pottery Neolithic and Early Pottery Neolithic (AEP I-III) the mortality profiles do not reflect that of a wild and hunted population but rather a culturally controlled or proto-domestic cattle population. Herd security and meat production seem to have been the main foci of *Bos* husbandry (Balasse, 2003). This form of animal management continued through AEP IV-V but by this time the animals can be considered fully domestic. Meat production becomes more intensive by AEP VI, so as in Transitional (AEP VII) and Early Halaf (AEP VIII) periods. Cattle at Tell Sabi Abyad, then, appear to have been kept primarily for their meat. There is no evidence of their use for traction.

Thus, faunal analyses suggest that ovicaprids were the milk producers during the final stages of the Early Pottery Neolithic, during which milk fats are first attested at Tell Sabi Abyad, through the Pre-Halaf-Transitional into the Early Halaf period. The role of cattle remains less clear. In the time period concerned domestic cattle appear to have been primarily exploited for meat at Tell Sabi Abyad but the occasional use of their milk cannot be ruled out. A total disregard of this valuable resource would be contrary to what would be expected, particularly when considering the importance of this animal in Halaf iconography.

5. Concluding remarks

The lipid residue analyses presented herein further emphasise the importance and the feasibility of studying residue residues on pottery sherds from the Late Neolithic in Upper Mesopotamia (Evershed et al., 2008). The results obtained provide a valuable perspective on the practical uses of Late Neolithic ceramic containers in Upper Mesopotamia. Traditionally pottery use is modelled on the basis of the shape, size, and performance properties of the containers; the investigation of lipid residues allows us to move one important step further, scrutinizing how vessels were actually used. Thus, this study supports earlier interpretations of

Mineral Coarse Ware, Early Fine Ware, Grey-Black Ware and in particular Dark-Faced Burnished Ware as 'cooking' wares. The DFBW especially gave an unequivocal signal that the vessels were used frequently subjected to direct heating. Together with the unique performance properties of this ceramic type this shows that DFBW was the preferred 'cooking' ware during the period investigated in this study.

In contrast, the recovery rates from the coarsely-made, plant-tempered Standard Ware that constituted the bulk of the ceramic assemblage were far lower (3%). Usage of this early pottery remains unresolved. Storing or serving cold liquid lipid rich foods would likely have resulted in increased recovery rates, and thus dry goods storage is a more likely function. Cooking was in this case not the primary activity in which this ware category was involved. In contrast to Çatalhöyük in Central Anatolia, no 'clay balls' that might have been used for indirect boiling (Copley et al., 2006) have so far been attested at Tell Sabi Abyad or at any other Late Neolithic site in Upper Mesopotamia. If we put this into a broader perspective, it appears that in this region cooking foodstuffs in ceramic pots did not play any significant role prior to the final stages of the Early Pottery Neolithic. A sustained shift to cooked food, then, was *not* synchronous with the first adoption of pottery, which in Upper Mesopotamia took place already between ca. 7000–6700 cal. BC (Nieuwenhuyse et al., 2010). Only from ca. 6400–6300 cal. BC onwards can cooking foods and beverages in pottery vessels unequivocally be documented as part of Late Neolithic culinary practices. This was a profound cultural change that must have had far-reaching socio-economic repercussions, which so far have been barely investigated.

The lipid residues extracted from sherds from Tell Sabi Abyad are among the oldest currently known in the ancient Near East, together with those from Çatalhöyük in Central Anatolia (Copley et al., 2006; Evershed et al., 2008; Pitter et al., 2013). They predate by several centuries those from Tell el-Kerkh dated to the Early Halaf period (Shimoyama and Ichikawa, 2000) and they are considerably earlier that the ones from Late Neolithic al-Basatîn in the Wadi Ziqlab in northern Jordan (Gregg et al., 2009). Thus, they provide a unique insight into the late 7th millennium BC animal exploitation in the region. This study has thus brought forward evidence for the consumption of milk in the centuries immediately preceding the Halaf period and during the Early Halaf period. This is evidenced some 80 years after Max Mallowan introduced the term 'cream bowl' for the characteristic carinated collared bowls from the Halaf period based on ethnographic examples when observing his workmen drinking milk from carinated metal plates (Mallowan and Rose, 1935). Complementary studies of age-at-death of animals at the site and construction of kill-off patterns demonstrate that the exploitation of ovicaprids for meat during the Early Pottery period shifted towards a mixed exploitation for meat and milk from the Pre-Halaf period. Cattle however seem to have been kept primarily for meat.

Evidently, while the residue evidence by itself is rather consistent with archaeozoological studies performed on the Tell Sabi Abyad assemblages, the overall signal of early dairy production in the Upper Mesopotamian Late Neolithic remains rather weak. In this study only four sherds gave unequivocal evidence of ruminant dairy lipids. Although the number of sherds containing dairy fat residues is low they do point to early secondary product exploitation. However, the small size of the current assemblage means caution should be exercised in its contextualization and interpretation. Further samples from a broader range of time periods are required to strengthen, confirm or refute the patterns that begin to emerge. Notwithstanding this, if the different strands of evidence are brought together – pottery use, material culture, village layout, animal exploitation and dairy residues – they suggest far-reaching social and economic changes transforming Upper Mesopotamian societies at the end of the seventh millennium.

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