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## ORIGINAL ARTICLE



# Wild Food: Plants, Fish and Small Animals on the Menu for Early Holocene Populations at al-Khiday, Central Sudan

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**Abstract** Al-Khiday, located on the bank of the White Nile in Sudan, offers an exceptionally preserved stratigraphic sequence, providing a unique opportunity to use organic residue analysis to investigate diet and subsistence during the Khartoum Mesolithic and the Early Neolithic, a period of nearly 3500 years (7000–4500 cal BC). While the vast and diverse Mesolithic fish assemblage indicates a strong reliance on products from aquatic habitats,

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Laboratory of Biodiversity and Evolutionary Genomics, Royal Belgian Institute of Natural Sciences, KU Leuven, Brussels, Belgium floodplains, vegetated marshes, and open water, results from the lipid residue analysis suggest that the fish were not cooked in ceramic pots, but consumed in other ways. Rather, pots were more specialized in processing plants, including wild grasses, leafy plants, and sedges. These results, confirmed by experimental analysis, provide, for the first time, direct chemical evidence for plant exploitation in the Khartoum Mesolithic. Non-ruminant fauna (e.g., warthog) and low lipid-yielding reptiles (e.g., Adanson's mud turtle and Nile monitor lizard), found in significant numbers at al-Khiday, were likely also cooked in pots. There is little evidence for the processing of wild ruminants in the Mesolithic pots, suggesting either that ruminant species were not routinely hunted or that large wild fauna may have been cooked in different ways, possibly grilled over fires. These data suggest sophisticated economic strategies by sedentary people exploiting their ecological niche to the fullest. Pottery use changed considerably in the Early Neolithic, with ruminant products being more routinely processed in pots, and while the exploitation of domesticates cannot be confirmed by a small faunal assemblage, some dairying took place. The results provide valuable information on Early and Middle Holocene lifeways in central Sudan.

**Resumé** Al-Khiday, située sur la rive du Nil Blanc, présente une séquence stratigraphique exceptionnellement préservée qui fournit une opportunité unique d'utiliser l'analyze des résidus organiques pour étudier le régime alimentaire et la subsistance pendant tout le Mésolithique de Khartoum et le Néolithique ancien, une période d'environ de 3500 ans (7000-4500 cal BC). Alors que le grand assemblage diversifié d'os de poisson du Mésolithique montre une forte dépendance aux produits des habitats aquatiques, des plaines alluviales, des marais végétalisés et de la rivière, les résultats de l'analyze des résidus lipidiques suggèrent que les poissons n'étaient pas cuits dans les marmites en céramique, mais, probablement, consommés dans d'autres façons. Ou plutôt, les pots étaient utilisés pour la transformation des plantes, des herbes sauvages, des feuillues et des carex. Ce résultat, confirmé par l'analyze expérimentale, offre une preuve chimique directe de l'exploitation des plantes dans le Mésolithique de Khartoum. La faune non-ruminante, comme le phacochère, et les reptiles à faible rendement en lipides, comme la tortue d'Adanson ou le varan du Nil, retrouvés en quantités importantes à al-Khiday, était également probablement cuits dans des pots. Il y a peu de preuves de la transformation de ruminants sauvages dans les pots en céramique, ce qui suggère que les espèces des ruminants n'étaient pas systématiquement chassées, ou que la grande faune sauvage pouvait avoir été cuite de différentes manières, probablement grillée. Ces données suggèrent que les populations sédentarisées avaient des stratégies économiques sophistiquées, tirant probablement la meilleure part de leur niche écologique. L'utilization de la poterie a considérablement changé au Néolithique ancien, ou les produits des ruminants sont plus régulièrement transformés avec cuisson dans de pots, et bien que l'exploitation des animaux domestiqués ne puisse être confirmée par un petit assemblage faunique, une certaine production laitière a lieu. En résumé, les résultats fournissent des informations utiles sur les modes de vie de l'Holocène ancien et moyen dans le centre du Soudan.

**Keywords** Organic residues · Al-Khiday · Khartoum Mesolithic · Neolithic · Isotopes · Plant processing

### Introduction

The Mesolithic of Sudan, or Khartoum Mesolithic, first characterized by Arkell (1949) during his excavations at the Khartoum Hospital site (Arkell, 1949) and Shaheinab (Arkell, 1953), lasted at least 3000 years (Salvatori, 2012). It is marked by a wide distribution of sites along the banks and hinterlands of the Blue and White Nile and other early Holocene rivers such as Wadi Muqaddam and Wadi Howar (Hosfield et al., 2015; Jesse, 2000; Smith, 1998). These sites include Saggai I (Caneva, 1983), Umm Marrahi (Elamin & Mohammed-Ali, 2004), Abu Darbein, Aneibis and El Damer (Haaland & Magid, 1995), Umm Singid (Caneva, et al., 1993), Jebel Sabaloka (Varadzinová et al., 2022), el-Barga (Honegger, 2005; Honegger & Bastien, 2009), and El Shaqadud (Marks & Mohammed-Ali, 1991), among others (e.g., Ali Hakem & Khabir, 1989; Caneva, 1988; Khabir, 1987; Salvatori & Usai, 2006, 2009; Salvatori, et al., 2011; Usai & Salvatori, 2002, 2005). Such sites were inhabited by communities of mainly sedentary pottery-using hunter-gatherer-fisherfolk (HGF), who produced high-quality decorated ceramics, grinding stones, and retouched stone and bone tools and instruments.

Notably, these sites are generally marked by a lack of stratified deposits, largely due to natural and anthropogenic post-depositional disturbances, thereby limiting the potential for understanding the chronology, extent, and social and economic organization of the Sudanese Mesolithic (Salvatori, 2012; Usai, 2014). However, the site of al-Khiday provides a detailed, stratigraphically integral record of Mesolithic deposits (Salvatori, 2012; Salvatori et al., 2014), producing the first cultural sequence of the Khartoum Mesolithic that covers a period of over 1000 years (Maritan, et al., 2018; Usai & Salvatori, 2019), and the transition to the Early Neolithic (Table 1). Here, the Mesolithic is divided into three phases, Early (7000-6650 cal BC), Middle (6650-6000 cal BC), and Late Mesolithic (6000-5800 cal BC). The Middle Mesolithic can be further broken down into three sub-phases: Middle Mesolithic A (6650-6500 cal BC), B (6500-6250 cal BC) and C (6200-6000 cal BC). To date, these deposits have provided a unique opportunity to investigate subsistence strategies, burial practices, material production, and social organization at a very detailed level (e.g., Buckley et al., 2014; Dal Sasso et al., 2014, 2016; Linseele, 2020; Maritan et al., 2018; Salvatori et al., 2014; Usai & Salvatori, 2019; Usai et al., 2014, 2017; Zerboni et al., 2018). The neolithization of the region appears rooted in the Mesolithic HGF groups, although some external influences cannot be ruled out (Usai, 2016). Neolithic habitation sites tend to be situated on the alluvial plain close

Table 1 Calibrated radiocarbon dates from al-Khiday sites

Lab No	Site and Feature	Material	C14 date bp	Cal. 1o BP*	Cal. 2 <sub>o</sub> BC	Cultural period
Caserta DSH9624	16-D-3 SU16	Charcoal	7986±33	8998-8651	7049–6702	Early Mesolithic
Beta-376245	16-D-3 SU9	Charcoal	$7980 \pm 50$	8999–8645	7050–6696	Early Mesolithic
Beta-201728	16-D-5 SU 6	Charcoal	$7980 \pm 40$	8996-8649	7047-6700	Early Mesolithic
Beta-279538	16-D-5 SU 250	Org.Sed.	$7960 \pm 40$	8990-8646	7041–6697	Early Mesolithic
Beta-239622	16-D-5 SU 455a	Charcoal	$7940 \pm 40$	8985-8639	7036–6690	Early Mesolithic
Beta-213892	16-D-5 SU 48	Charcoal	$7870 \pm 40$	8975-8547	7026-6598	Early Mesolithic
Beta-239621	16-D-5 SU 455b	Shell	$7830 \pm 40$	8770-8476	6821-6527	Early Mesolithic
Caserta DSH9297	16-D-3 SU19 Est	Charcoal	$7738 \pm 47$	8595-8419	6646-6470	Middle-Mesolithic A
Beta-385158	16-D-4 Pit 126d	Shell	$7770 \pm 30$	8600-8449	6651-6500	Middle-Mesolithic A
Beta-239620	16-D-4 Pit 29	Shell	$7770 \pm 40$	8630-8430	6681–6481	Middle-Mesolithic A
Beta-239619	16-D-4 Pit 6a	Shell	$7760 \pm 90$	8973-8377	7024-6428	Middle-Mesolithic A
Beta-257255	16-D-5 Peat	Org.Sed.	$7740 \pm 50$	8598-8416	6649–6467	Middle-Mesolithic A
Beta-213891	16-D-5 SU 37	Charcoal	$7710 \pm 40$	8589-8411	6640-6462	Middle-Mesolithic A
Beta-279537	16-D-4 Pit 75	Shell	$7640 \pm 110$	8645-8184	6696-6235	Middle-Mesolithic B
Caserta DSH9263	16-D-3 SU12F	Charcoal	$7513 \pm 59$	8406-8191	6457-6242	Middle-Mesolithic B
Beta-257258	16-D-4 Pit 52	Shell	$7620\pm50$	8537-8350	6588-6401	Middle-Mesolithic B
Beta-279536	16-D-4 Pit 74	Shell	$7600 \pm 90$	8588-8195	6639–6246	Middle-Mesolithic B
Caserta DSH9608	16D4 SE F2	Shell	$7572 \pm 36$	8425-8226	6476-6277	Middle-Mesolithic B
Beta-413247	16-D-4B Pit 9	Shell	$7540 \pm 30$	8410-8217	6461-6268	Middle-Mesolithic B
Beta-257257	16-D-4B Pit 6	Charcoal	$7540 \pm 50$	8419-8200	6470-6251	Middle-Mesolithic B
Beta-279535	16-D-4 Pit 73	Shell	$7530 \pm 100$	8545-8043	6596-6094	Middle-Mesolithic B
Caserta DSH9261	16-D-3 SU12B	Charcoal	$7359 \pm 32$	8310-8031	6361-6082	Middle-Mesolithic B
Beta-318869	16-D-4 Pit 97	Shell	$7510 \pm 40$	8394-8195	6445-6246	Middle-Mesolithic B
Beta-318868	16-D-4 Pit 108	Shell	$7430 \pm 40$	8357-8176	6408-6227	Middle-Mesolithic B
Beta-376244	16-D-3 SU1	Shell	$7300 \pm 30$	8175-8027	6226-6078	Middle-Mesolithic C
Caserta DSH9033	16-D-3 SU1 Upper	Shell	$6763 \pm 73$	7749–7479	5800-5530	Late Mesolithic A
Beta-376246	16-D-4 SU147	Org.Sed.	$6820 \pm 40$	7725–7580	5776-5631	Late Mesolithic A
Beta-201726	10-W-4 SU12	Charcoal	$6490 \pm 40$	7483-7312	5534-5363	Late Mesolithic B
Beta-361822	16-D-4 Grave 158	Shell	$5690 \pm 30$	6558–6399	4609-4450	Early Neolithic
Beta-302091	16-D-4 Grave 103	Shell	$5550 \pm 80$	6535–6191	4586-4242	Early Neolithic
Beta-213890	16-D-5 SU5	Shell	$5470 \pm 50$	6394–6124	4445-4175	Early Neolithic
Beta-302092	16-D-6 SU1	Shell	$5360 \pm 80$	6295–5941	4346-3992	Early Neolithic
Beta-318870	TREISGPS31	Shell	$4560 \pm 30$	5437-5052	3488-3103	Late Neolithic
Beta-361821	16-D-4 Grave 58	Wood	$2030 \pm 30$	2096-1882	147BC69AD	Late Meroitic
Beta-257256	16-D-4 SU61	Charcoal	$1940 \pm 40$	1983–1743	34BC-208AD	Late Meroitic
Beta-239618	16-D-4 Grave 47	Charcoal	$1900 \pm 50$	1941–1708	10BC-243AD	Late Meroitic

Dates calibrated with OxCal 4.4 (IntCal20, Reimer et al., 2020). *SU* stratigraphic unit; shells are all freshwater and mostly of Pila species. Beta Lab dates were published in Maritan et al., 2018, Caserta DSH Lab dates have not been previously published Pila shell is not subject to a reservoir effect (Macri, 2019)

to then-extant Nile channels, which offered reliable, fertile resources. Agro-pastoralists likely utilized large permanent camps such as Esh Shaheinab and Geili, and domesticates, mainly cattle (*Bos taurus*), were the dominant mammal at sites such as Kadero (4600–3800 cal BC) and Ghaba (5200–4500 cal BC), where wild plants

were also intensively exploited (Caneva, 1988; Gautier, 1984; Haaland, 1992, 1995; Krzyzaniak, 1991; Marshall & Hildebrand, 2002; Out et al., 2016). Here, we use an exceptional long-term stratigraphic sequence, in combination with organic residue analysis, faunal and archaeobotanical remains, to investigate the diet and subsistence

strategies of Khartoum Mesolithic and Early Neolithic White Nile populations, across a period of nearly 3500 years.

### Al-Khiday Site Complex, Sudan

The al-Khiday complex is located along the western bank of the White Nile, 25 km south of the city of Omdurman (central Sudan) and 3 km west of the present-day Nile (Fig. 1). The group of sites at al-Khiday are situated on a sandy ridge rising around 4 m above the surrounding plain (Salvatori, 2012; Salvatori et al., 2011, 2014; Usai & Salvatori, 2005; Usai et al., 2010; Williams et al., 2015; Zerboni, 2011), similar to other sites located above the White Nile floodplain, occupied from the early Holocene (Adamson et al., 1982; Clark, 1989; Zerboni, 2011).

The group of sites comprises:

- (i) 16-D-4, a multi-phase site with a cemetery containing 235 inhumation burials of Pre-Mesolithic, Neolithic, and Classic/Late Meroitic Age; a Mesolithic functional area comprising 104 pits; and a near complete Mesolithic circular hut.
- (ii) 16-D-4B, a second Mesolithic functional area with a large number of garbage pits, 32 of which were excavated.
- (iii) 16-D-6, a Neolithic site, probably seasonal.
- (iv) 16-D-3 and 16-D-5 are Mesolithic settlements.
   16-D-5 also contains a poorly preserved Neolithic layer, partly disturbed by post-Meroitic tumuli.

### Mesolithic al-Khiday

Settlements 16-D-3 and 16-D-5, the settlement occupation in the cemetery area (16-D-4), and the functional area (16-D-4B) cover a period of more than one thousand years, between the Early and Late Mesolithic (Salvatori, 2012; Salvatori et al., 2011). The first occupation levels at sites 16-D-3 and 16-D-5 date to the Late Early Mesolithic (Table 1) at *ca*. 7000 to 6750 cal BC and are characterized by postholes suggesting the presence of huts, likely inhabited by small groups of hunter-gatherer-fisherfolk exploiting the rich riverine environment on a

seasonal basis (Linseele, 2020; Linseele & Zerboni, 2018; Maritan et al., 2018). Middle Mesolithic occupation levels are seen at sites 16-D-3, 16-D-4, 16-D-4B, and 16-D-5. This period has three distinct phases (Middle Mesolithic A, B and C; for dates, see Table 1) and is marked by a significant transformation in house architecture, with semi-subterranean mud-walled huts (with fireplaces) being arranged either in a beehive pattern (Salvatori et al., 2014) or isolated (Salvatori et al., 2018). Over 104 pits (Usai, 2014), with diverse fillings, were excavated at 16-D-4 (Usai & Salvatori, 2019). Those filled with ashes, burnt pebbles, (scarce) mollusk shells, and mammal and fish bones (often partially in anatomical connection) are thought to be fireplace pits. Most of the remaining features, including a concentration of 32 pits at 16-D-4B, appear to be large refuse dumps containing significant amounts of faunal remains. All are connected with the main settlement (16-D-5) in a spatial arrangement that suggests an organized use of the space and implies a more sedentary lifestyle (Usai & Salvatori, 2019).

During this period, the climate in the lower White Nile valley was significantly wetter due to high stands of the White Nile floods between ca. 9700 and 9100 cal BP (Adamson et al., 1980; Williams, 2009). The deposits at Mesolithic al-Khiday suggest a local swamp or wetland environment, seasonally flooded by the White Nile (Williams, 2009; Williams & Adamson, 1980; Williams et al., 2015; Zerboni, 2011). Locally wet areas were interspersed with dry thorn savanna and scrub, including species of Acacia, sometimes in association with Balanites aegyptiaca, and grasses such as Hyparrhenia anthistirriodes, Cymbopogon nervatus, and Sorghum spp. (Wickens, 1982). Extensive papyrus swamps probably extended along the White Nile and likely included several varieties of sedges, as suggested by phytoliths from Early and Middle Mesolithic C deposits 16-D-3 (Usai & Salvatori, 2019). The wetlands would have been a source of abundant fish and edible Pila snails, with drier areas providing favorable conditions for species such as kob (Kobus kob), elephant (Loxodonta), and buffalo (Syncerus caffer), as detected in the faunal remains. Thus, during the Mesolithic, the al-Khiday sites were situated in a rich and productive location, providing a sustainable environment over a chronological span of ca. 1200 years.

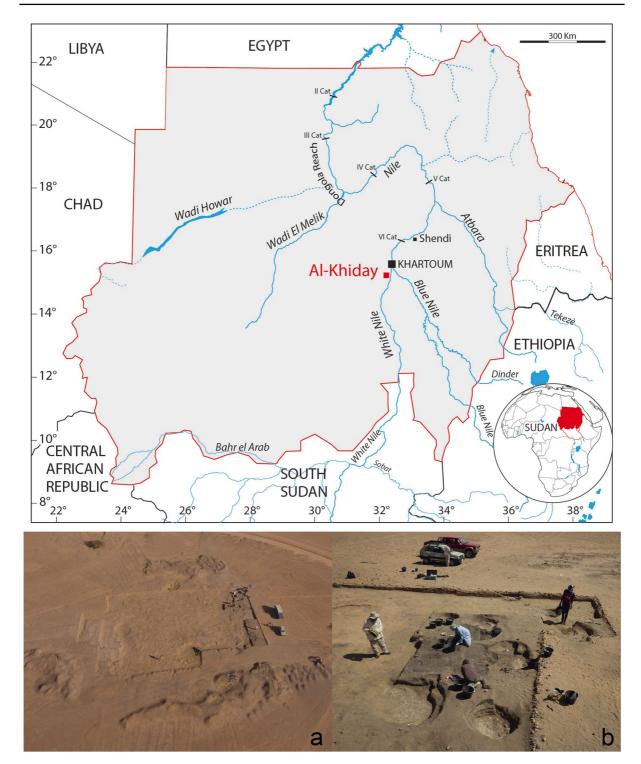


Fig. 1 Map of Sudan with the location of al-Khiday sites: **a** kite view of site al-Khiday 2 (16-D-4); **b** kite view of a portion of the site under excavation ( $\mathbb{C}$  CSSeS)

### Neolithic al-Khiday

The Neolithic evidence at al-Khiday originates from 16-D-5 and 16-D-6 sites. The occupation layer at 16-D-5 comprises a 'carpet' of fragmented pottery, rhyolite and quartzite lithic tools, *Chambardia* shells, grinding stones, and numerous animal bones (Salvatori, 2012; Salvatori et al., 2011). At site 16-D-6, a single occupation layer was recognized, comprising post-holes and a small fireplace (Salvatori et al., 2018). A complete burial of an individual in a contracted position was found, suggesting the presence of a small cemetery associated with the settlement (Salvatori et al., 2018, Fig. 14).

Although detailed information on the climate during the Neolithic occupation at al-Khiday is missing, general data support drying conditions, denoted by a fall in the level of the Nile and a retreat of vegetation (Wickens, 1982; Williams et al., 2015). This is confirmed by oxygen isotope analysis of al-Khiday Neolithic human remains (Iacumin et al., 2016), whose values indicate that the population experienced much drier conditions, although not dry enough to limit the hunting of diverse wild animals.

### **Al-Khiday Pottery**

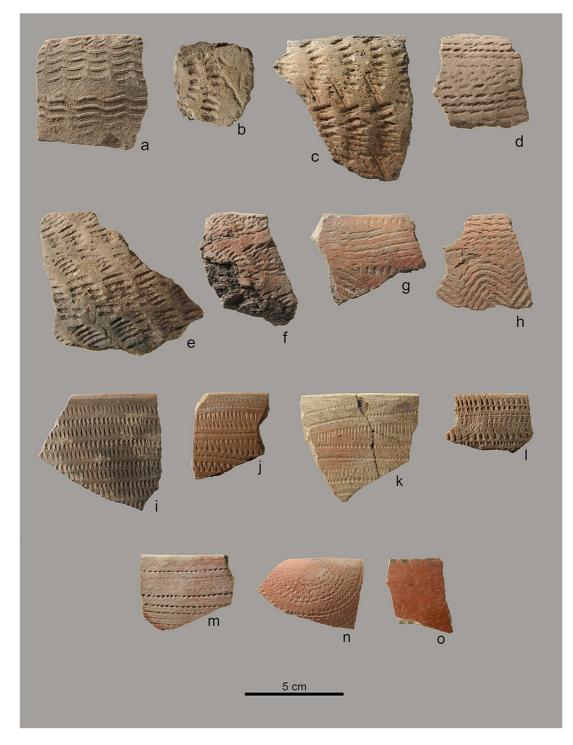
The Mesolithic pottery assemblage comprised vertical and open bowls and (mainly) globular jars. Incised and impressed decorations cover most of the pots' surfaces, including the rims. They often occur on the inner surface (Fig. 2a-h). A range of decoration types (see Fig. 2) was used, including incised wavy line, dotted wavy line, alternately pivoting stamp, rocker stamp drops, and rocker stamp dotted zigzag (Dal Sasso et al., 2014; Salvatori, 2012). The Mesolithic pottery comprises two main fabrics (Dal Sasso et al., 2014): one characterized by abundant grains of sand-sized quartz with a sub-rounded shape (Fig. 3a), and the other rich in feldspar (Fig. 3b). The latter is derived from the grinding of an alkaline granite or syenite and is only found in pottery with certain types of decoration, in particular, incised wavy line (Fig. 2a), rocker stamp dotted zigzag packed (Fig. 2d), and alternately pivoting stamp (Fig. 2g).

Neolithic pottery (Fig. 2i-o) is characterized by new decoration types (fish scale, incised straight lines). However, some Mesolithic styles remain in use (alternately pivoting stamp, rocker stamp dotted zigzag and scraped). The Neolithic pottery from 16-D-5 includes globular, hemispherical, and conical bowls, and globular and ovoid jars decorated with a large range of motifs/techniques. Evenly spaced rocker stamp and packed dotted zig-zag rocker stamp are the most common (Fig. 2i-l). Concentric dotted semicircles produced by an alternating pivoting stamp technique and, as seen on complete pots found at the site, usually organized in symmetric panels, are also very common (Fig. 2g, m). Pottery without impressions is also common, generally with red-slipped polished surfaces and a series of black semicircles at the rim (Fig. 20). The Neolithic pottery is produced using a quartz-rich paste, with smaller inclusions (fine sand), which are sub-rounded/sub-angular in shape (Fig. 3c) denoting a technological change from Mesolithic production (Dal Sasso et al., 2014; Salvatori, 2012; Fig. 3c).

### Lipid Residue Analysis and Results

A total of 152 potsherds, from several al-Khiday sites, including 16-D-3, 16-D-4, 16-D-4B, 16-D-5, and 16-D-6, were investigated according to well-established analytical procedures described in Online Supplementary Material 1 (Correa-Ascencio & Evershed, 2014), with 37 sherds yielding interpretable lipid profiles (Mesolithic 22, Neolithic 15), i.e., containing sufficient concentrations (> 5  $\mu$ g g<sup>-1</sup>) of lipids that can be reliably interpreted (Evershed, 2008). Overall, the lipid recovery rate was 24%, although it varied significantly between the Mesolithic phases and the Early Neolithic (Table 2). The mean lipid concentration of the sherds was 55.0  $\mu$ g g<sup>-1</sup>, with a maximum lipid concentration of 459.6  $\mu$ g g<sup>-1</sup> (Table 3). Notably, lipid concentration for 81% of the interpretable residues (n=23) was less than 50 µg g<sup>-1</sup>, with 59% containing less than 30  $\mu$ g g<sup>-1</sup>. These low lipid concentrations strongly suggest that the commodities processed in most vessels are likely of a low lipidbearing origin, either plants or low lipid-yielding animal products.

The analysis of the fatty acid methyl esters (FAMEs) extracted from the al-Khiday potsherds shows lipid profiles dominated by free fatty acids,



**Fig. 2** Selection of lipid-yielding Mesolithic and Neolithic pottery fragments. Decoration is described by means of technique plus design. Early Mesolithic: **a** Incised Wavy line; **b** Rocker stamp drops deep. Middle Mesolithic A: **c** Rocker stamp drops fan, deep. Middle Mesolithic C: **d** Rocker stamp drops, horizontal; **e** Rocker stamp drops oblique and fan, deep; **f** Rocker stamp drops band; **g** Alternately Pivoting Stamp atypical. Late Mesolithic A: **h** Laqiya. Early Neolithic: **i**, **j** Rocker stamp dotted dots; **k** Rocker stamp dotted zig-zag spaced; **l** Rocker stamp dotted zig-zag; **m** Alternately Pivoting Stamp; **n** Rocker stamp dotted concentric semicircles; **o** Burnished undecorated

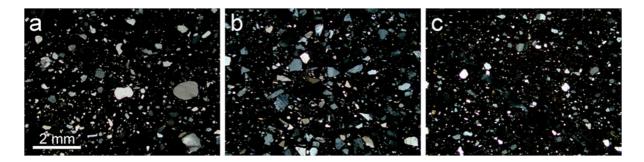


Fig. 3 Photomicrographs of the three main fabrics observed among the Mesolithic and Neolithic pottery at al-Khiday: a fabric rich in sand-sized rounded quartz grains; b fabric rich

palmitic (C<sub>16</sub>) and stearic (C<sub>18</sub>) (Fig. 4a). Also present in the majority of the Mesolithic lipid residues (73%, n=22) are a series of even-numbered longchain fatty acids (LCFAs) in the range up to  $C_{30}$ , often in high abundance (Table 3 and Fig. 4b-d). These are strongly indicative either of an origin in leaf or stem epicuticular waxes (Bianchi, 1995; Kolattukudy et al., 1976; Kunst & Samuels, 2003; Tulloch, 1976) or suberin (Kolattukudy, 1980, 1981; Pollard et al., 2008; Walton, 1990), particularly given the low abundance of lipids in the majority of the vessels. Notably, of these plant lipid profiles, 21 (57% of the assemblage, 95% of Mesolithic sherds) were found in Mesolithic potsherds, with only six originating from the Neolithic vessels (16% of the assemblage, 40% of Neolithic vessels).

Several lipid profiles (n=6, Fig. 4b-d) also include a series of long-chain  $\alpha$ ,  $\omega$ -dicarboxylic acids (diacids), often present in high abundance. These major components of plant polymeric compounds, such as cutin and suberin, are highly unusual in

in alkali-feldspar; c fabric rich in sub-angular fine sand-sized quartz inclusions. Images taken at the same magnification and in crossed-polarized light

plants. Those with chain lengths of  $C_{16}$  to  $C_{26}$  are considered diagnostic for the presence of suberin, found in the outer part of underground plants (Dembitsky et al., 2002; Holloway, 1984; Kolattukudy, 1980, 1981; Pollard et al., 2008; Walton, 1990). The  $\alpha$ ,  $\omega$ -dicarboxylic acids are present at < 5% (apart from in the Arabidopsis) in cutin, so this is highly unlikely to be the source of the diacids within the lipid profiles.

It should be noted that while the detection of abundant fatty acids and diacids is consistent with an origin in plant polyesters, such as suberin or cutin, the absence of hydroxy fatty acids is surprising. Since sterols can be lost in organic residues by a range of degradative pathways (Hammann et al., 2018), it is possible that hydroxylated aliphatic components, such as hydroxy fatty acids, are vulnerable to the same fate and, thus, are not seen in lipid extracts. Nonetheless, the presence of diacids indicate that underground storage organs, i.e., tubers, were likely processed in some al-Khiday vessels.

Table 2 Number of sherds analyzed, interpretable lipid recovered, and % recovery by period

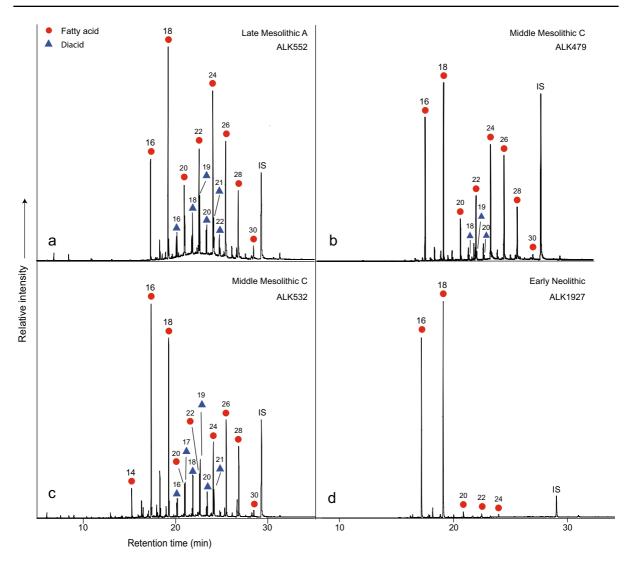
Phase	Date	Number of sherds analyzed	Interpretable lipid recovered	% recovery
Early Mesolithic	7000–6650 cal BC	16	2	12
Middle Mesolithic A	6650–6500 cal BC	12	2	17
Middle Mesolithic B	6500–6250 cal BC	16	2	12
Middle Mesolithic C	6200-6000 cal BC	59	12	20
Late Mesolithic A	6000–5800 cal BC	24	4	17
Late Mesolithic B	5500-5300 cal BC	3	0	0
Early Neolithic	4500-4300 cal BC	22	15	68
		152	37	

Laboratory number	Decoration	Figure number	Site	Strat unit	Phase	Fabric	Lipid con- centration	$\delta^{13} C_{16:0}$	$\delta^{13}C_{18:0}$	$\Delta^{13}$ C	Attribution	LCFA (x) and/or
							( 8 8H)					present
ALK452	Laqiya	I	16-D-3	US IB	MMC	-	6.5	-21.7	-21.6	0.1	Plant/non- ruminant/ ruminant adinose	×
ALK453	APS atyp	Figure 2g	16-D-3	US 1B	MMC	7	20.0	-21.9	- 22.8	6.0-	Plant/ ruminant adipose	0X
ALK466	Rs dr bands	Rs dr bands Figure 2f	16-D-3	US 1	MMC	7	24.4	-22.4	-21.6	0.8	Non- ruminant adipose	×
ALK479	Rs dr hor+fan	I	16-D-3	US 1D	MMC	7	28.1	-22.1	-20.4	1.7	Plant	XO
ALK480	Rs dr bands	I	16-D-3	US ID	MMB	7	41.8	- 23.8	- 24.4	-0.6	Plant/non- ruminant/ ruminant adipose	×
ALK491	Rs dr ob + fan	Figure <mark>2e</mark>	16-D-3	US 1D	MMC	7	2.1	-18.9	-17.2	1.6	Plant	x
ALK492	DWL atyp	I	16-D-3	6 SN	EM	7	15.2	- 18.9	- 18.8	0.0	Plant/non- ruminant/ ruminant adipose	×
ALK498	Rs dr hor	I	16-D-4	US 189	MMB	2	12.8	- 19.3	-17.3	2.1	Plant	x
ALK504	Rs dr hor	I	16-D-3	US 1C	MMC	2	32.9	- 19.4	-18.2	1.3	Plant	XO
ALK515	Rs dr ob + hor	I	16-D-3	US 5	MMA	7	49.0	- 24.3	-21.1	3.2	Plant	x
ALK516	Rs d zz spaced	Figure 2k	16-D-6	US1/1b	EN	ŝ	120.9	-21.2	-24.7	-3.6	Dairy fat/ plant	x
ALK517	Rs d zz packed	I	16-D-6	US1/1b	EN	ŝ	19.0	-21.7	-20.5	1.3	Non- ruminant	x

ry Decoration Figure Rs d zz - packed - Rs dr fan Figure 2c deep Rs dr hor Figure 2d Rs dr hor - IWL atyp Figure 2a Rs d con Figure 2a Rs d con Figure 2a Rs d con Figure 2a Rs d con Figure 2a Rs d zz - packed - Laqiya - Rs pl zz - Rs pl zz - Laqiya -	Table 3 (continued)	ontinued)											
Re d $z_{c}$ -         16-D-6         USI/1b         EV         3         14.4         -190         -179           packed         Edm         Fgure 2c         16-D-4         US 153         MMA         2         459.6         -22.3         -23.3           Ra thor         -         16-D-3         US 1E         MMC         2         13.1         -23.0         -22.4           Ra thor         -         16-D-3         US 1E         MMC         2         20.7         -23.3         -23.4           Ra thor         -         16-D-3         US 1E         MMC         2         20.7         -20.7         -19.2           Ra thor         -         16-D-3         US 1E         MMC         2         20.7         -20.7         -19.2           Ra too         Figure 2n         16-D-3         US 1E         MMC         2         20.7         -19.2         -19.2           Ra too         Figure 2n         16-D-3         US 1E         MMC         2         20.7         -19.2         -19.2           Ra too         Figure 2n         16-D-3         US 1E         MMC         2         20.7         -20.4         -21.9           Ra toro	Laboratory number			Site	Strat unit	Phase	Fabric	Lipid con- centration (µg g <sup>-1</sup> )	$\delta^{13}C_{16:0}$	8 <sup>13</sup> C <sub>18:0</sub>	$\Delta^{13}$ C	Attribution	LCFA (x) and/or diacids (o) present
Rs dr fan         Figure 2 $[6-D-4]$ US 153         MMA         2         459.6 $-2.2.3$ $-23.3$ Rs dr hor         -         -         16-D-3         US 1E         MMC         2         13.1 $-2.07$ $-2.44$ Rs dr hor         -         -         16-D-3         US 1E         MMC         2 $-2.07$ $-2.34$ $-2.34$ Rs dr hor         -         -         16-D-3         US 1E         MMC         2 $-0.77$ $-2.34$ $-2.34$ Rs dr hor         -         -         16-D-3         US 1E         MMC         2 $20.7$ $-2.07$ $-19.2$ Rs dron         Figure 2n         16-D-5         US 5         EN         3 $15.6$ $-19.2$ $-19.2$ Rs dron         Figure 2n         16-D-5         US 5         EN         3 $95.9$ $-19.2$ $-21.9$ Rs dron         Figure 2n         16-D-5         US 5         EN $3$ $15.6$ $-21.8$ $-21.9$ Rs dron         Figure 2n         16-D-5         US 5         S	ALK518	Rs d zz packed	1	16-D-6	US1/1b	EN	ε	14.4	- 19.0	-17.9	1.0	Non- ruminant adipose	×
Rs dr hor       Figure 2d       16-D-3       US IE       MMC       2       13.1       -23.0       -23.4         Rs dr hor       -       16-D-3       US IE       MMC       2       13.1       -20.7       -18.4         IWL atyp       Figure 2a       16-D-3       US IE       MMC       2       20.7       -20.7       -18.4         Rs dr hor       -       16-D-3       US IE       MMC       2       20.0       -19.2       -19.2         Rs dron       Figure 2n       16-D-5       US 5       EN       3       15.6       -18.5       -19.2         Rs dron       Figure 2n       16-D-5       US 5       EN       3       95.9       -19.2       -21.9         Rs dron       Figure 2n       16-D-5       US 5       EN       3       95.9       -19.2       -21.9         Rs drz       -       16-D-3       US 1       MMC       1       45.8       -20.4       -21.8         Rs dron       Figure 2n       16-D-3       US 1       MMC       1       42.2       -21.9       -21.9         Iaqiya       -       16-D-3       US 1       MMC       2       12.0       -21.8       -21.8	ALK521	Rs dr fan deep	Figure 2c	16-D-4	US 153	MMA	7	459.6	- 22.3	-23.3	-1.0	Ruminant adipose	×
Rs dr hor         -         16-D-3         US IE         MMC         2         20.7         -20.7         -184           IWL ayp         Figure 2a         16-D-3         US IE         MMC         1         2.6         -23.8         -23.4           Rs dr hor         -         16-D-3         US IE         MMC         2         20.7         -19.2         -19.2           Rs dron         Figure 2n         16-D-5         US 5         EN         3         15.6         -18.5         -19.2           Rs dron         Figure 2n         16-D-5         US 5         EN         3         95.9         -19.2         -21.9           APS         Figure 2n         16-D-5         US 5         EN         3         45.8         -20.4         -21.8           APS         Figure 2n         16-D-3         US 1         MMC         1         42.2         -22.3         -23.9           Laqiya         -         16-D-3         US 1         MMC         1         45.8         -20.4         -21.8           Laqiya         -         16-D-3         US 1         MMC         2         -22.3         -23.9           Laqiya         -         16-D-3	ALK523	Rs dr hor	Figure 2d	16-D-3	US 1E	MMC	2	13.1	-23.0	-22.4	0.6	Plant	x
IWL atyp         Figure 2a         16-D-3         US IE         MMC         1         2.6         -23.8         -23.4           Rs dr hor         -         16-D-3         US IE         MMC         2         200.8         -19.2         -19.2           Rs         -         16-D-3         US IE         MMC         2         200.8         -19.2         -19.2           Rs         -         16-D-5         US 5         EN         3         15.6         -18.5         -19.2           Rs dcon         Figure 2n         16-D-5         US 5         EN         3         95.9         -19.2         -21.9           APS         Figure 2n         16-D-5         US 5         EN         3         95.9         -21.8         -21.8           APS         Figure 2n         16-D-3         US 1         MMC         1         42.2         -22.8         -23.9           Laqiya         -         16-D-3         US 1         MMC         2         10.7         -21.4         -20.8           Laqiya         -         16-D-3         US 1         MMC         2         12.0         -26.4         -21.8           Laqiya         -         16-D-3	ALK524	Rs dr hor	I	16-D-3	US 1E	MMC	2	20.7	-20.7	-18.4	2.3	Plant	х
Rs dr hor       -       16-D-3       US IE       MMC       2       200.8       -19.2       -19.2         Rs       -       16-D-5       US 5       EN       3       15.6       -18.5       -19.3         Rs       d con       Figure 2n       16-D-5       US 5       EN       3       95.9       -19.2       -21.9         APS       Figure 2n       16-D-5       US 5       EN       3       95.9       -19.2       -21.9         APS       Figure 2n       16-D-5       US 5       EN       3       95.9       -19.2       -21.9         APS       Figure 2n       16-D-3       US 1       MMC       1       42.2       -22.3       -23.9         Ladiya       -       16-D-3       US 1       MMC       2       12.0       -26.4       -21.8         Ladiya       -       16-D-3       US 1       MMC       2       12.0       -20.4       -21.8         Ladiya       -       16-D-3       US 1       MMC       2       12.0       -20.4       -21.8         Ladiya       -       16-D-3       US 1       MMC       2       12.0       -20.4       -21.8 <t< td=""><td>ALK525</td><td>IWL atyp</td><td>Figure <mark>2a</mark></td><td>16-D-3</td><td>US 1E</td><td>MMC</td><td>1</td><td>2.6</td><td>-23.8</td><td>-23.4</td><td>0.5</td><td>Non-</td><td>x</td></t<>	ALK525	IWL atyp	Figure <mark>2a</mark>	16-D-3	US 1E	MMC	1	2.6	-23.8	-23.4	0.5	Non-	x
Rs dr hor       -       16-D-3       US IE       MMC       2       200.8       -19.2       -19.2         Rs       -       16-D-5       US 5       EN       3       15.6       -18.5       -19.3         Rs       -       16-D-5       US 5       EN       3       95.9       -19.2       -19.3         Rs d con       Figue 2n       16-D-5       US 5       EN       3       95.9       -19.2       -21.9         Rs d zz       -       16-D-5       US 5       EN       3       95.9       -19.2       -21.9         Rs d zz       -       16-D-5       US 5       EN       3       95.9       -19.2       -21.6         Laqiya       -       16-D-3       US 1       MMC       1       42.2       -22.3       -23.9         Laqiya       -       16-D-3       US 1       MMC       2       12.7       -21.8         Rs pt zz       -       16-D-3       US 1       MMC       2       20.4       -21.8         Laqiya       -       16-D-3       US 1       MMC       2       22.0       -21.4       -20.8         Laqiya       -       16-D-3       Colluvium B												ruminant adipose	
Rs       -       16-D-5       US 5       EN       3       15.6       -18.5       -19.3         Rs d con       Figure 2n       16-D-5       US 5       EN       3       95.9       -19.2       -21.9         Rs d con       Figure 2n       16-D-5       US 5       EN       3       95.9       -19.2       -21.9         APS       Figure 2n       16-D-5       US 5       EN       3       45.8       -20.4       -21.8         Packed       -       16-D-3       US 1       MMC       1       42.2       -22.8       -23.9         Laqiya       -       16-D-3       US 1       MMC       2       12.0       -26.4       -21.8         Laqiya       -       16-D-3       US 1       MMC       2       12.0       -26.4       -21.8         Laqiya       -       16-D-3       US 1       MMC       2       12.0       -26.4       -21.8         Laqiya       -       16-D-3       US 1       MMC       2       12.0       -26.4       -21.8         Laqiya       -       16-D-3       Colluvium B       LMM       2       12.0       -26.4       -21.8         Laqiya	ALK526	Rs dr hor	I	16-D-3	US IE	MMC	7	200.8	- 19.2	- 19.2	0.0	Ruminant/ non- ruminant	x
Rs       -       16-D-5       US 5       EN       3       15.6       -18.5       -19.3         Rs d con       Figure 2n       16-D-5       US 5       EN       3       95.9       -19.2       -21.9         APS       Figure 2n       16-D-5       US 5       EN       3       95.9       -19.2       -21.9         APS       Figure 2n       16-D-5       US 5       EN       3       95.9       -10.0       -21.5         Rs d zz       -       16-D-3       US 1       MMC       1       42.2       -20.4       -21.8         Laqiya       -       16-D-3       US 1       MMC       1       42.2       -22.28       -23.9         Laqiya       -       16-D-3       US 1       MMC       2       12.0       -26.4       -21.8         Rs pl zz       -       16-D-3       Colluvium B       LMA       2       12.0       -26.4       -21.8         Laqiya       -       16-D-3       Colluvium B       LMA       2       12.0       -26.4       -21.8         Laqiya       -       16-D-3       Colluvium B       LMA       2       13.7       -21.4       -20.6 <t< td=""><td></td><td></td><td></td><td></td><td></td><td></td><td></td><td></td><td></td><td></td><td></td><td>adipose</td><td></td></t<>												adipose	
Rs d con       Figure 2n       16-D-5       US 5       EN       3       95.9       -19.2       -21.9         APS       Figure 2n       16-D-5       US 5       EN       3       95.9       -19.2       -21.9         APS       Figure 2n       16-D-5       US 5       EN       3       95.8       -16.0       -21.5         Rs d zz       -       16-D-3       US 1       MMC       1       45.8       -20.4       -21.8         packed       -       16-D-3       US 1       MMC       1       42.2       -22.8       -23.9         Laqiya       -       16-D-3       US 1       MMC       2       12.0       -26.4       -21.8         Rs pl zz       -       16-D-3       US 1       MMC       2       12.0       -26.4       -21.8         Laqiya       -       16-D-3       Colluvium B       LMA       2       12.0       -26.4       -21.8         Laqiya       -       16-D-3       Colluvium B       LMA       2       13.7       -21.4       -20.8         Laqiya       -       16-D-3       Colluvium B       LMA       2       13.4       -20.6       -19.6	ALK527	Rs	I	16-D-5	US 5	EN	ю	15.6	- 18.5	- 19.3	-0.8	Ruminant adipose	x
APS       Figue 2m       16-D-5       US 5       EN       3       18.3       -16.0       -21.5         packed       -       16-D-5       US 5       EN       3       45.8       -20.4       -21.8         packed       -       16-D-3       US 1       MMC       1       42.2       -22.8       -23.9         Laqiya       -       16-D-3       US 1       MMC       2       12.0       -26.4       -21.8         Laqiya       -       16-D-3       US 1       MMC       2       12.0       -26.4       -21.8         Rs pl zz       -       16-D-3       Colluvium B       LMA       2       1.7       -21.4       -20.8         Laqiya       -       16-D-3       Colluvium B       LMA       2       1.7       -21.4       -20.6         Laqiya       -       16-D-3       Colluvium B       LMA       2       1.7       -21.4       -20.6         Laqiya       -       16-D-3       Colluvium B       LMA       2       18.4       -20.6       -24.0         Laqiya       -       16-D-3       Colluvium B       LMA       2       81.1       -20.6       -19.6 <td< td=""><td>ALK529</td><td>Rs d con circles</td><td>Figure 2n</td><td>16-D-5</td><td>US 5</td><td>EN</td><td>ю</td><td>95.9</td><td>- 19.2</td><td>-21.9</td><td>-2.7</td><td>Ruminant adipose</td><td>ı</td></td<>	ALK529	Rs d con circles	Figure 2n	16-D-5	US 5	EN	ю	95.9	- 19.2	-21.9	-2.7	Ruminant adipose	ı
Rs d zz       -       16-D-5       US 5       EN       3       45.8       -20.4       -21.8         packed       -       16-D-3       US 1       MMC       1       42.2       -22.8       -23.9         Laqiya       -       16-D-3       US 1       MMC       2       12.0       -26.4       -21.8         Laqiya       -       16-D-3       US 1       MMC       2       12.0       -26.4       -21.8         Rs pl zz       -       16-D-3       Colluvium B       LMA       2       1.7       -21.4       -20.8         Laqiya       -       16-D-3       Colluvium B       LMA       2       18.4       -20.6       -24.0         Laqiya       -       16-D-3       Colluvium B       LMA       2       18.4       -20.6       -19.6         Laqiya       -       16-D-3       Colluvium B       LMA       2       81.1       -20.6       -19.6         Laqiya       -       16-D-3       Colluvium A       LMA       2       28.5       -20.2       -20.9	ALK530	APS	Figure 2m	16-D-5	US 5	EN	ŝ	18.3	-16.0	-21.5	-5.5	Dairy fat	
Laqiya       -       16-D-3       US 1       MMC       1       42.2       -22.8       -23.9       -         Laqiya       -       16-D-3       US 1       MMC       2       12.0       -26.4       -21.8         Rs pl zz       -       16-D-3       Colluvium B       LMA       2       1.7       -21.4       -20.8         Laqiya       -       16-D-3       Colluvium B       LMA       2       1.7       -21.4       -20.8         Laqiya       -       16-D-3       Colluvium B       LMA       2       18.4       -20.6       -24.0       -         Laqiya       -       16-D-3       Colluvium B       LMA       2       81.1       -20.6       -19.6         Laqiya       Figure 2h       16-D-3       Colluvium A       LMA       2       28.5       -20.2       -20.9	ALK531	Rs d zz packed	I	16-D-5	US 5	EN	С	45.8	-20.4	-21.8	-1.4	Ruminant adipose	ı
Laqiya       -       16-D-3       US 1       MMC       2       12.0       -26.4       -21.8         Rs pl zz       -       16-D-3       Colluvium B       LMA       2       1.7       -21.4       -20.8         Laqiya       -       16-D-3       Colluvium B       LMA       2       18.4       -20.6       -24.0       -         Laqiya       -       16-D-3       Colluvium B       LMA       2       81.1       -20.6       -19.6         Laqiya       -       16-D-3       Colluvium B       LMA       2       81.1       -20.6       -19.6         Laqiya       Figure 2h       16-D-3       Colluvium A       LMA       2       28.5       -20.2       -20.9	ALK532	Laqiya	I	16-D-3	US 1	MMC	1	42.2	-22.8	-23.9	-1.1	Plant/	хо
Laqiya       -       16-D-3       US 1       MMC       2       12.0       -26.4       -21.8         Rs pl zz       -       16-D-3       Colluvium B       LMA       2       1.7       -21.4       -20.8         Laqiya       -       16-D-3       Colluvium B       LMA       2       18.4       -20.6       -24.0       -         Laqiya       -       16-D-3       Colluvium B       LMA       2       81.1       -20.6       -19.6         Laqiya       -       16-D-3       Colluvium B       LMA       2       81.1       -20.6       -19.6         Laqiya       Figure 2h       16-D-3       Colluvium A       LMA       2       28.5       -20.2       -20.9												ruminant adipose	
Rs pl zz       -       16-D-3       Colluvium B       LMA       2       1.7       -21.4       -20.8         Laqiya       -       16-D-3       Colluvium B       LMA       2       18.4       -20.6       -24.0       -         Laqiya       -       16-D-3       Colluvium B       LMA       2       81.1       -20.6       -19.6         Laqiya       -       16-D-3       Colluvium B       LMA       2       81.1       -20.6       -19.6         Laqiya       Figure 2h       16-D-3       Colluvium A       LMA       2       28.5       -20.2       -20.9       -	ALK534	Laqiya	I	16-D-3	US 1	MMC	2	12.0	-26.4	-21.8	4.6	Plant	х
Laqiya – 16-D-3 Colluvium B LMA 2 18.4 –20.6 –24.0 – Laqiya – 16-D-3 Colluvium B LMA 2 81.1 –20.6 –19.6 Laqiya Figure 2h 16-D-3 Colluvium A LMA 2 28.5 –20.2 –20.9 –	ALK539	Rs pl zz	I	16-D-3	Colluvium B		2	1.7	-21.4	-20.8	9.0	Non-	х
Laqiya         -         16-D-3         Colluvium B         LMA         2         18.4         -20.6         -24.0         -           Laqiya         -         16-D-3         Colluvium B         LMA         2         81.1         -20.6         -19.6           Laqiya         -         16-D-3         Colluvium A         LMA         2         28.5         -20.2         -20.9         -												ruminant adipose/ plant	
Laqiya         -         16-D-3         Colluvium B         LMA         2         81.1         -20.6         -19.6           Laqiya         Figure 2h         16-D-3         Colluvium A         LMA         2         28.5         -20.2         -20.9	ALK542	Laqiya	I	16-D-3	Colluvium B		2	18.4	-20.6	-24.0	-3.3	Dairy fat/ plant	×
Ladiya Figure 2h 16-D-3 Colluvium A LMA 2 28.5 –20.2 –20.9	ALK545	Laqiya	I	16-D-3	Colluvium B		2	81.1	-20.6	-19.6	1.0	Plant	ХO
6	ALK552	Laqiya	Figure 2h	16-D-3	Colluvium A		2	28.5	-20.2	-20.9	-0.7	Plant	хо

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Table 3 (continued)	ontinued)											
Laboratory number	Decoration	Figure number	Site	Strat unit	Phase	Fabric	Lipid con- centration (µg g <sup>-1</sup> )	δ <sup>13</sup> C <sub>16:0</sub>	$\delta^{13}C_{18:0}$	$\Delta^{13}$ C	Attribution	LCFA (x) and/or diacids (o) present
ALK627B	Burnished undec	Figure 20	16-D-5	USS	EN	.6	85.4	- 22.0	-21.1	0.8	Non- ruminant adipose/ plant	x
ALK630	Rs d zz	I	16-D-5	SUUS5	EN	ю	28.1	-24.2	-25.0	-0.8	Plant/ ruminant adipose	×
ALK634	Rs dr deep	Figure 2b	16-D-5	US452	EM	7	7.6	- 23.1	-23.6	- 0.4	Ruminant/ non- ruminant adipose	
ALK1927	Rs d unev spaced	Figure <mark>2</mark> j	16-D-5	US5	EN	3	132.5	-15.5	- 18.2	-2.7	Ruminant adipose	ı
ALK1929	Rs d unev spaced	I	16-D-5	US5	EN	3	60.0	- 16.0	-17.9	-1.9	Ruminant adipose	ı
ALK1931	R sd ev spaced	I	16-D-5	US5	EN	б	123.7	-21.1	- 20.9	0.2	Ruminant/ non-rumi- nant	·
ALK1932	R sd ev spaced	I	16-D-5	US5	EN	3	97.6	-20.8	- 22.8	-2.0	Ruminant adipose	ı
ALK1933	Rs d unev spaced	Figure 2i	16-D-5	US5	EN	б	159.9	- 18.3	- 18.2	0.1	Ruminant/ non-rumi- nant	ı
ALK1934	Rs d zz	Figure 2k	16-D-5	US5	EN	6	46.9	- 19.4	- 19.2	0.3	Ruminant/ non-rumi- nant	T



**Fig. 4 a-d** Partial gas chromatograms of acid-extracted FAMEs showing a-c: typical plant lipid profiles (ALK479, ALK532 and ALK552, Mesolithic sherds); d: typical degraded animal fat lipid profile (ALK1927, Early Neolithic sherd). Cir-

cles, *n*-alkanoic acids (fatty acids, FA), triangles, diacids; IS, internal standard,  $C_{34}$  *n*-tetratriacontane. Number denotes carbon chain length

All lipid-yielding samples were analyzed by gas chromatography-mass spectrometry (GC–MS) in selected ion monitoring (SIM) mode to check for the presence of freshwater biomarkers, such as  $\omega$ -(o-alkylphenyl) alkanoic acids (APAAs) and vicinal dihydroxy acid (DHYAs), which denote the processing of shellfish/crustaceans, fish, waterfowl, and aquatic mammals (see Cramp & Evershed, 2014). No aquatic biomarkers were detectable in the analyzed potsherds, although some aquatic input to the vessels cannot be discounted.

Thirty-seven samples (Mesolithic n=22 and Early Neolithic n=15) underwent gas chromatography–combustion–isotope ratio mass spectrometry (GC-C-IRMS) analyses (Table 3 and Fig. 5) to determine the  $\delta^{13}$ C values of the major fatty acids, C<sub>16:0</sub> and C<sub>18:0</sub>, and ascertain the source of the lipids extracted (Dunne et al., 2012).

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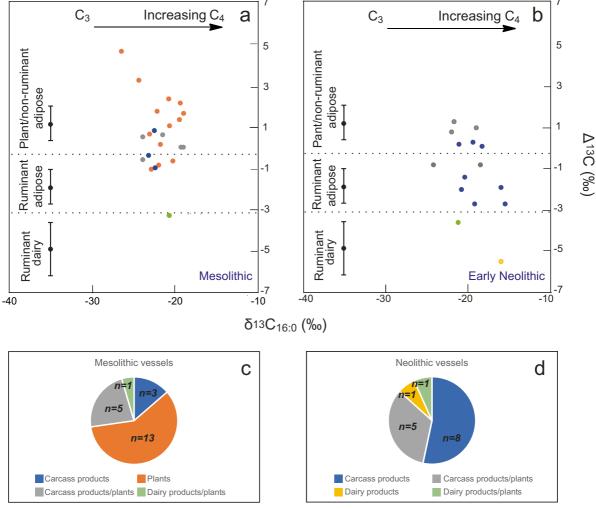


Fig. 5 Graphs showing:  $\Delta^{13}C$  ( $\delta^{13}C_{18:0}$ - $\delta^{13}C_{16:0}$ ) values from a Mesolithic and b Neolithic vessels from al-Khiday; c, d show the types of foods processed in the al-Khiday vessels. Ranges represent the mean  $\pm 1$  s.d. of the  $\Delta$   $^{13}C$  values for a global

### Mesolithic Vessels

Lipid residue results for the Mesolithic period (Fig. 5a, see also annotated Fig. 1a in Online Supplementary Material 2) show that twelve sherds (55%) plot within the non-ruminant/plant region, three sherds plot within the ruminant adipose region (14%), and six sherds plot between the ruminant and non-ruminant/plant region (27%). Significantly, one sherd (ALK542, Late Mesolithic A), with a  $\Delta^{13}$ C value of -3.3 % (Fig. 5a), plots just within

database comprising modern reference animal fats from the UK, Africa, and elsewhere (Dudd & Evershed, 1998; Dunne et al., 2012)

the ruminant dairy region It should be noted that the vessel plots at the upper end of the range, suggest either some mixing of dairy and ruminant carcass products or possibly, that low quantities of dairy products were masked isotopically through mixing with high amounts of non-ruminant fats, although this seems unlikely given its low lipid concentration  $(18.4 \ \mu g \ g^{-1}).$ 

Of the twelve Mesolithic sherds plotting within the non-ruminant/plant region, ten display lipid profiles containing a series of even-numbered long-chain

Sample number	Date	Phase	C <sub>20</sub> FA	C <sub>22</sub> FA	C <sub>24</sub> FA	C <sub>26</sub> FA	C <sub>28</sub> FA
ALK452	6200–6000 cal BC	Middle Mesolithic C	-20.0	-25.5	-25.8	-26.8	_
ALK453	6200-6000 cal BC	Middle Mesolithic C	-22.5	-23.8	-24.8	-27.0	-
ALK479	6200-6000 cal BC	Middle Mesolithic C	-22.5	-25.9	-26.9	-27.7	-
ALK480	6500-6250 cal BC	Middle Mesolithic B	-23.6	-25.4	-26.2	-	-
ALK492	7000–6650 cal BC	Early Mesolithic	- 19.0	-22.5	-21.3	-	-
ALK504	6200-6000 cal BC	Middle Mesolithic C	-18.9	-24.5	-25.9	-26.9	-26.1
ALK515	6650-6500 cal BC	Middle Mesolithic A	-20.0	-23.4	-23.0	-22.1	-18.0
ALK532	6200-6000 cal BC	Middle Mesolithic C	-23.6	-27.0	-28.0	-28.7	-28.2
ALK552	6000–5800 cal BC	Late Mesolithic A	-23.6	-27.9	-28.4	-28.8	-28.7

Table 4 Sample number, date, phase, and  $\delta^{13}$ C values of measurable LCFAs, indicative of plant processing, extracted from al-Khiday potsherds

fatty acids (LCFAs) up to C<sub>30</sub> (see Tables 3 and 4, and Fig. 5a), often in high abundance. Of these, seven have lipid concentrations of less than 30  $\mu$ g g<sup>-1</sup>, with two at less than 50  $\mu$ g g<sup>-1</sup> and one at 81.1  $\mu$ g g<sup>-1</sup>. Three of these sherds also contain diacids. The combination of LCFAs, diacids, and low lipid concentrations suggests these vessels were either used to process solely plants or plants with small amounts of carcass products. However, care must be taken in their interpretation as fatty acids derived from plant processing can contribute more depleted  $\delta^{13}C$ values to the overall fatty acid signature of the  $C_{16:0}$ and C<sub>180</sub> fatty acids. Similarly, of the three Mesolithic potsherds which plot in the ruminant adipose region, two include a series of LCFAs (up to  $C_{30}$ ) and diacids together with lipid concentrations of less than 50  $\mu$ g g<sup>-1</sup>. Thus, they were also likely to have been used to process combinations of plant and animal products or, possibly, solely plants (see below on experimental processing). The other vessel with a ruminant signal (ALK521) has a much higher lipid concentration at 459.6  $\mu g g^{-1}$  and was most likely used to process ruminant carcass products (Online Supplementary Material, Fig. 1a and Table 3). Six vessels yielded  $\Delta^{13}$ C values plotting between the ruminant and non-ruminant/plant regions. Of these, vessel ALK634 did not include LCFAs, but had a low lipid concentration at 7.6  $\mu$ g g<sup>-1</sup> and thus was more likely to have been used solely to process plants, as were the remaining four vessels, with lipid concentrations of less than 50  $\mu$ g g<sup>-1</sup>, and series of LCFAs (and diacids in vessel ALK552). The remaining vessel, ALK526, which did include LCFAs, but with a lipid concentration of 200.8  $\mu$ g g<sup>-1</sup>, may have been used to process both plants and animal carcass products, or denote multi-use of the vessel.

### Early Neolithic Vessels

In the Early Neolithic, seven vessels (47%) plot within the ruminant adipose region (Fig. 5b, see also annotated Fig. 1b in Online Supplementary Material), confirming they were used to process carcass products from either domesticated cattle, sheep (Ovis aries)/goat (Capra hircus), or hunted wild ruminant mammals such as antelope. Of these, the lipid profile from one vessel (ALK630) also includes a series of LCFAs (up to  $C_{28}$ ), suggesting that, although this one vessel was likely used to process animal products and plants, the majority (n=6) were used solely to process ruminant carcass products. Three vessels, ALK517, ALK518, and ALK627B, plot in the non-ruminant/plant region and were likely used to process non-ruminant/plant products (with two also including sequences of LCFAs). Three further vessels, plotting between the ruminant and non-ruminant region, were used to process mixtures of ruminant and non-ruminant carcass products. Two sherds (ALK516 and ALK530) plot within the ruminant dairy region with  $\Delta^{13}$ C values of -3.6and - 5.5 % (Fig. 5b), suggesting some minor exploitation of ruminant animals for their dairy products.

# δ<sup>13</sup>C Values

Organic residue analysis can be a powerful proxy for investigating animal management strategies, such as transhumance (Dunne et al., 2012), and in discerning the past isoscapes in which prehistoric groups lived (West et al., 2010). 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 therefore a reflection of the consumed diet, providing information about the environment in which the animals foraged (Copley et al., 2003; Mukherjee et al., 2005). In the case of the Mesolithic vessels, the majority appear to have been used to process plants, and thus the  $\delta^{13}$ C values will reflect their C<sub>3</sub>/C<sub>4</sub> origin.

The  $\delta^{13}C_{16:0}$  values of the fatty acids extracted from the al-Khiday potsherds range from -26.4 to -16.0%, and the  $\delta^{13}C_{18:0}$  values range from -25.0 to -17.9% (Table 3). The  $\delta^{13}C_{16:0}$  values (Table 3) suggest either that the plants processed in the vessels were mainly of  $C_4$  origin or that the animals giving rise to these fats had subsisted on a range of different forages composed of mainly C<sub>4</sub> plants or C<sub>4</sub> plants with some contribution from C<sub>3</sub> plants. Where possible, the  $\delta^{13}$ C values of the long-chain fatty acids (denoting plant processing) were measured to provide information on their possible origin (i.e.,  $C_3$  or  $C_4,$  Table 4). The  $\delta^{13}C$ values fall mostly within the known  $\delta^{13}$ C values for C<sub>3</sub> bulk plant lipids, although often at the low end of the range, which suggests that the plants processed in the vessels were generally of C<sub>3</sub> origin, with some possible minor C<sub>4</sub> input. It should be noted that these plants may have routinely been both prepared and mixed (and sequentially processed) within the vessels.

While the more enriched (C<sub>4</sub>)  $\delta^{13}$ C<sub>16:0</sub> and  $\delta^{13}$ C<sub>18:0</sub> values suggest the processing of either C<sub>4</sub> wild grasses (such as sorghum and millet) or C<sub>4</sub> tubers (such as the tuberous sedge plant, *Cyperus rotundus*), both common in al-Khiday phytolith remains, the LCFA  $\delta^{13}$ C values suggest a more C<sub>3</sub> origin. The presence of LCFAs denotes the processing of leafy plants, candidates for which include jute mallow, also known as bush okra (*Corchorus olitorius*), widely used as a leafy vegetable and potherb in Africa and found close to the Nile today in abundance. The presence of the diacids in some vessels confirms the likely processing of sedges (although these were not present in enough quantity to be measured isotopically).

To help shed light on possible commodities cooked in the pots, we carried out in situ experimental cooking of the  $C_4$  grasses—sorghum (*Sorghum* sp.) and millet (*Panicum miliaceum*)—and tuberous sedge plants (*Cyperus rotundus*), in individual local, handmade cooking pots, to make comparisons between the  $\delta^{13}C_{16:0}$  and  $\delta^{13}C_{18:0}$  values obtained from the separate cooking of each plant and those obtained from the archaeological vessels.

# Organic Residue Analysis of the Experimental Cooking Vessels

Full details of the production of pots and the cooking experiments can be found in Online Supplementary Material (OSM 3 and Fig. 6). Rims and bases from these experimental vessels, together with 'burnt-on' residues from the millet and *Cyperus* cooking events, were analyzed using well-established analytical procedures described in OSM 1 (Correa-Ascencio & Evershed, 2014). Lipid recoveries from the vessels were generally low (Table 5) but higher from the surface residue, the 'remains' of the cooking events, for example, lipid concentration of the surface residue from the millet pot was high at 5437.5  $\mu$ g g<sup>-1</sup>.

Where possible, the  $\delta^{13}$ C values of the major fatty acids, C<sub>16:0</sub> and C<sub>18:0</sub>, were measured using GC-C-IRMS (Table 5 and Fig. 7). The  $\delta^{13}$ C values of both the sherd and the surface residue from the vessel used to process millet suggest a C4 origin as does the vessel used to cook sorghum. Both the sherd and surface residue from the millet cooking plot in the plant/ non-ruminant region with  $\Delta^{13}$ C values of 0.4 and 0.3% (Fig. 7), as does the surface residue from the cyperus-processing vessel, at 0.2%. Interestingly, the sherd from the sorghum vessel plotted between the ruminant and plant/non-ruminant ranges, although closer to the ruminant, demonstrating that vessels used to process plants can plot at the furthest extent of the plant range. As seen in Fig. 7, the  $\Delta^{13}$ C values from the experimental pots are similar to those from the archaeological pots used to process plants and confirm that the plant lipid profiles could originate from C<sub>4</sub> wild grass (millet and sorghum) and/or tuber (Cyperus?) processing. These values, together with the  $\delta^{13}$ C values from the LCFAs, which denote the addition/mixing of C<sub>3</sub> leafy plants, suggest that the vessels may have been used to cook meals comprising grains, tubers, and leafy vegetables, sometimes with the addition of meat products.

### **Discussion of the Lipid Residue Results**

Pottery use across the Mesolithic (see Table 2), covering the 1200 years of the Early Mesolithic (c. 7000 cal BC) to the Late Mesolithic A (c. 5800 cal BC), seems to be entirely consistent. However, by the Early Neolithic (4500 cal BC), changes in diet and subsistence can be seen in the clear differences between foods processed in the Mesolithic and Early Neolithic pottery (Fig. 5). In the Mesolithic period (Fig. 5a, c), most vessels (n=18; 82%) plot either within the non-ruminant/plant range or between the ruminant and non-ruminant/plant region. Fifteen of these (68% of total Mesolithic sherds) were used to process wild plants or, possibly, combinations

of plant and low lipid-yielding animal products, evidenced by the combination of even-numbered LCFAs, diacids and very low lipid concentrations (all at  $\leq$  50.0 µg g<sup>-1</sup>, save for ALK545 at 81.1 µg g<sup>-1</sup>). The animal products processed mainly comprised non-ruminants, likely low-lipid yielding wild fauna. In contrast, a greater number of Neolithic vessels were used to process ruminant products (Fig. 5b, d), and although it should be noted that the experimental vessel used to process sorghum also plotted at the upper end of this region, only one, of the seven used to process ruminant products, displayed evidence for plant processing. The ruminant carcass products (discussed further below) display a slightly broader range of  $\delta^{13}C_{16:0}$  values, with some vessels being



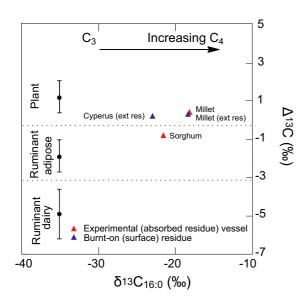
Fig. 6 Ceramic pots being hand-made in Omdurman. Stages of production:  $\mathbf{a}$  the clay is shaped, using a large stone, to make a bowl shape;  $\mathbf{b}$  the lip is thinned using the paddle and anvil technique and raised to close up into a jar shape;  $\mathbf{c}$  the rim and handles are made by adding shaped coils of clay and a

red slip is applied using a cloth soaked in ochre and diluted in water, pots were then polished with a pebble; **d** the pots were then fired in the open-air using wood and dung as fuel. Fragments of broken jars were used to create a cover, thus increasing thermal insulation

Sample number	Vessel	Part of vessel	Plant	Lipid concen- tration ( $\mu g g^{-1}$ )	Total lipid in extract (μg)	$\delta^{13}C_{16:0}$	$\delta^{13}C_{18:0}$	$\Delta^{13}C$
EXP001	1	Base	Cyperus	3.4	8.5	_	_	_
EXP002	1	Shoulder	Cyperus	7.0	15.6	-	-	-
EXP003	2	Base	Millet	55.2	104.9	-18.2	-17.9	0.4
EXP004A	2	Rim	Millet	4.8	12.4	-	-	_
EXP005	3	Base	Sorghum	75.7	177.0	-21.6	-22.4	-0.8
EXP006B	3	Body	Sorghum	15.9	53.0	-	-	-
EXP001ER	1	Surface residue	Cyperus	990.1	168.3	-23.0	-22.9	0.2
EXP002ER	1	Surface residue	Cyperus	99.3	18.9	-23.8	-25.3	-1.5
EXP004ER	2	Surface residue	Millet	5437.5	761.3	-18.4	- 18.1	0.3

**Table 5** Sample number, vessel number, part of the vessel, type of plant processed, lipid concentrations ( $\mu g g^{-1}$ ), total lipid in extract ( $\mu g$ ),  $\delta^{13}C$  and  $\Delta^{13}C$  values from experimental cooking of C<sub>4</sub> plants in ceramics from Sudan

used to process carcass products from animals consuming more  $C_4$  products, in good agreement with the drying environmental conditions. While six vessels plot either in the non-ruminant/plant or between the ruminant and non-ruminant/plant region, only three contain evidence for plant processing, suggesting some vessels may have been used to process mixtures of these animal products and confirming a much greater reliance on meat processing than in the Mesolithic.



**Fig. 7** Graph showing  $\Delta^{13}$ C ( $\delta^{13}$ C<sub>18:0</sub>– $\delta^{13}$ C<sub>16:0</sub>) values from the experimental vessels and surface or burnt-on residues from the same vessels. Ranges represent the mean ± 1 s.d. of the  $\Delta^{13}$ C values for a global database comprising modern reference animal fats from the UK, Africa, and elsewhere (Dudd & Evershed, 1998; Dunne et al., 2012)

# Faunal Remains and Their Lipid Signatures at al-Khiday

The Mesolithic deposits at al-Khiday comprise predominantly fish remains (often at 90% or more), considerably higher than at other Central Sudanese Mesolithic sites (Linseele & Zerboni, 2018). Certainly, al-Khiday is located in an area with easy access to the Nile and other productive aquatic habitats, including the Jebel Baroka lake (Cremaschi et al., 2007) and other (possibly seasonal) small lake formations. At least twenty fish species have been identified (OSM 4, Table 1), indicating the exploitation of diverse aquatic habitats, floodplains, vegetated marshes, and open water, although clariid catfish, a shallow water fish, clearly dominate the assemblage. More details on these fish and a diachronic analysis have been published elsewhere (Linseele, 2020; also see OSM 4, Table 1). Freshwater turtles and Nile monitor lizard (Varanus niloticus) are also found, with the Adanson's mud turtle (Pelusios adansonii) in high abundance. Large numbers of mollusk shells, mainly Pila wernei (OSM 4, Table 1), were also present, particularly at 16-D-3, a shell midden. This freshwater snail is known to be easy to collect from swampy areas and pools during the dry season (Gautier, 1983). Bird remains are not common at the site (possibly due to poor preservation of small and fragile bones), although stork, duck, and geese were identified. The Mesolithic mammal fauna is diverse, indicating a lush savanna environment. Various wild mammal taxa are present (Table 6), particularly antelopes, although these could not often be identified beyond size class. Also found were the remains of baboon, hare, warthog, and (scarce) very

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		EM 16-D-5	EM 16-D-3	MMB 16-D-4	MMB 16-D-4B	MMC 16-D-3	MMC 16-D-4B	Neo 16-D-6
Reptiles	Unidentified reptile	3	1	1	. 1	6	6	I
	Snake	17	I	ŝ	I	11	2	I
	Softshell turtle (Trionychidae)	5	I	8	3	1	3	I
	cf. Adanson's mud turtle (Pelusios adansonii)	240	9	28	57	138	46	I
	Monitor lizard (Varanus niloticus)	55	Ι	3	3	28	85	I
	Crocodile (Crocodylus niloticus)	5	I	I	1	I	I	Ι
	Total	325	6	42	64	184	142	0
Birds	Unidentified bird	10	Ι	2	1	18	6	1
	Stork (Ciconiidae)	1	I	I	I	I	I	I
	Anatidae size Anas crecca	19	I	I	I	1	2	I
	Anatidae size Anas platyrynchus	0	Ι	I	I	1	I	I
	Goose	3	I	I	I	I	4	I
	Total	33	0	2	1	20	15	1
Non ruminants	Unidentified mammal	996	220	76	466	634	228	3082
	Yellow baboon (Papio cynocepha- lus)	I	1	I	I	7	I	I
	Hare (Lepus sp.)	I	I	I	I	1	I	I
	Nile grass rat (Arvicanthis niloti- cus)	I	I	I	I	1	I	ı
	Small rodent	3	I	1	I	8	I	1
	Small carnivore	5	I	I	I	I	I	I
	Medium-sized carnivore	39	3	1	I	14	I	1
	Large carnivore	1	I	I	1	I	I	1
	African elephant ( <i>Loxodonta africana</i> )	0	I	I	I	I	I	I
	Black rhinoceros ( <i>Diceros</i> bicornis)/or white rhinoceros ( <i>Cerathotherium simum</i> )	Ś	I	I	I	I	I	I
	Hippopotamus ( <i>Hippopotamus amphibius</i> )	2	I	I	I	I	I	1
	Warthog ( <i>Phacochoerus</i> sp.)	2	I	I	I	4	I	I

		EM 16-D-5	EM 16-D-3	MMB 16-D-4	MMB 16-D-4B	MMC 16-D-3	MMC 16-D-4B	Neo 16-D-6
Mammals	Very large mammal	I	I	I	I	۸-	I	I
		4	2	1	I	8	24	8
		I	I	Ι	I	Ι	Ι	I
	Giraffe (Giraffa camelopardalis)	63	4	I	I	I	I	33
	Bush duiker (Sylvicapra grimmia) or oribi (Ourebia ourebi)	$\mathfrak{c}$	I	I	I	1	I	ı
	Small antelope	16	I	I	2	14	8	7
	Bushbuck (Tragelaphus scriptus)	I	I	1	I	2	I	I
	Sitatunga (Tragelaphus spekii)	I	I	I	I	1	I	I
	Bohor reedbuck (Redunca redunca)	11	I	I	I	2	I	I
	Kob (Kobus kob)	3	I	I	I	9	5	I
	Medium-sized antelope	87	7	4	4	66	35	б
Ruminants	Small/medium-sized antelope	Ι	I	I	I	2	б	I
	Sheep (Ovis aries)	I	I	I	I	I	I	1
	Sheep (Ovis aries) or goat (Capra hircus)	I	I	5	I	I	I	б
	Small bovid	22	1	ı	1	ı	11	27
	Hartebeest (Alcelaphus buse- laphus) or tiang (Damaliscus korrigum)	4	4	1	I	S.	I	I
	Large antelope	56	4	4	4	28	2	I
	Mediumsized or large antelope	24	26	2	15	9	I	I
	Savannah buffalo (Syncerus caffer)	4	Ι	I	I	Ι	Ι	I
	Cattle ( <i>Bos taurus</i> ) or buffalo ( <i>Syncerus caffer</i> )	I	I	I	I	I	1	Ś
	Large bovid	38	3	I	3	9	I	38
	Total	1360	275	93	496	811	317	3176

large mammal taxa, including elephant, rhinoceros, and hippopotamus.

The faunal assemblage excavated from the Neolithic site 16-D-6 is very small and much more poorly preserved than those from the Mesolithic sites, likely due to the shallow nature of the deposits. The remains of wild ruminants, including three giraffe, two small antelope, and three medium-sized antelope, are more common than those of domesticates. Scant evidence for domesticates comprises three bones attributed to sheep/goat, and one of sheep. Although domesticated cattle bones were not identified, they may be present in those identified as large bovid, cattle, or buffalo. Compared to the Mesolithic, fish remains are much less common, consisting mainly of Nile perch (*Lates niloticus*) and lacking the diverse range of species seen in the Mesolithic.

### Animal Product Processing

During the Mesolithic, eight of the 22 lipid-yielding vessels (36%) were used to process animal carcass products. Of these, only three (14%) appear to have been used solely for animal carcass processing, with the remaining five (23%) comprising lipid profiles suggesting the processing of meat and plants (Fig. 5a, c). Most animal products processed across the 1200 years of the Mesolithic period seem to be either of non-ruminant origin or comprise mixtures of ruminant and non-ruminant carcass products (Table 3). This is surprising considering that wild ruminants contribute to the majority of identified mammalian remains, ranging between 84% and 100% of the identified mammalian taxa in the different Mesolithic assemblages (Table 6). Non-ruminant products may have included reptiles such as Adanson's mud turtle and Nile monitor lizard, found in significant numbers at al-Khiday, or warthog (Phacochoerus aethiopicus). However, these are not common at the site (Linseele, 2020). Certainly, the processing of low lipid-yielding meats from small fauna such as turtles may explain the low concentrations of lipids found. However, a cautionary note is needed as the  $\Delta^{13}C$ values from experimental processing of plants show them to plot either at the extent of the non-ruminant/ plant range or between the ruminant and non-ruminant/plant regions. This suggests that vessels attributed to a carcass product origin may also have been used to process plants.

In contrast, the processing of ruminant products predominates in Neolithic vessels (47%, Fig. 5b, d). These could originate from domesticates, such as cattle and sheep/goat, although the Neolithic faunal assemblage is very small and poorly preserved, making comparisons difficult. Interestingly, wild fauna was present in much higher abundance than domesticates. Africa presents a unique situation where, unlike other continents, there are many wild ruminants, including about 50–60 different species of bovids. Thus, the presence of ruminant adipose products is not a clear indication of the presence of domesticates and, as wild ruminant fauna are present in greater abundance in the animal bone assemblage, the level of exploitation of domesticates at al-Khiday remains unclear.

### Dairy Product Exploitation

Evidence for dairying is, however, a much clearer indication of domesticate exploitation. There is minor evidence for dairying in the Mesolithic, with one sherd plotting at the extent of the dairy range (Table 3 and Fig. 5a). This sherd is of the Laqiya type, from the Late Mesolithic A phase (ALK542, 6000-5800 cal BC), which would suggest a very early presence of domesticates and, indeed, dairying practices, at the site. However, it should be noted that fatty acids deriving from plant product processing can contribute more depleted  $\delta^{13}C$  values to the overall fatty acid signature of the C<sub>16:0</sub> and C<sub>18:0</sub> fatty acids, possibly giving a false positive for the presence of dairy products. Nonetheless, the presence of Laqiya pottery at al-Khiday is intriguing as it is thought to be possibly related to a new group that may originate from the Wadi Howar region in northwestern Sudan (Jesse, 2000, 2003; Usai & Salvatori, 2019). This period at al-Khiday corresponds to the phase when domestic animals are first recorded in the Nubian region, north of al-Khiday. Domestic cattle were identified in a burial at El Barga, dated to ca. 5700 cal BC (Honegger, 2004; Honegger & Williams, 2015), and domestic sheep/goat bones have been directly dated to 5700 cal BC (KIA34817,  $6820 \pm 30$  BP) at the site of Boni S05/140 in the Fourth Cataract region (Petrick, 2012; Pöllath, 2012; Wotzka et al., 2012). If we consider the possibility of an allochthonous origin, then it may be that dairy products become available to Late Mesolithic people at al-Khiday through a system of exchanges with groups from more northern sites known to be exploiting domesticates. Trading between some Mesolithic groups is supported by the identification of clay/temper with a northern origin, found in some al-Khiday pottery, suggesting either the circulation of raw materials used in pottery production or the movement of the pottery itself (Dal Sasso et al., 2014). Alternatively, it may be that small numbers of domesticated animals were present in central Sudan during this phase, possibly as a minor component. However, they are not (currently) present in the al-Khiday faunal assemblage (Salvatori & Usai, 2019), and this remains an open question.

Nevertheless, dairying, albeit on a minor scale, can be confirmed in the Neolithic (Fig. 5b), from at least 4500 cal BC, with two sherds (13%) yielding a ruminant dairy signal, very similar to results from the Neolithic phase (4600–4000 BC) at Khor Shambat 1 where one vessel returned evidence for dairy processing (Dunne et al., 2021). However, these data are in contrast to the Early Neolithic site of Kadero (4600–3800 BC; Krzyzaniak, 1991), where 47% of sherds yielded a ruminant dairy signal, suggesting that dairying was an important economic activity at that site (Dunne et al., 2017). This may be due to the location of Kadero, which, although further from the Nile, had access to good grazing land found on the alluvial plain on the east bank.

### Were Freshwater Fish Cooked in Pots at al-Khiday?

The absence of aquatic biomarkers in the al-Khiday ceramic assemblage was somewhat surprising, bearing in mind the extensive availability of nearby surface waters and the preponderance of fish fauna in the Mesolithic deposits (Linseele, 2020). Certainly, central Sudan is renowned for its Mesolithic hunter-gatherer-fisherfolk, giving rise to the aptly named Aqualithic Culture (Sutton, 1977), where sites are known for the presence of bone harpoons and substantial fish and other aquatic faunal remains (Linseele, 2020; Linseele & Zerboni, 2018). At al-Khiday, harpoons, gorge hooks, and large catfish spines used as tools, possibly perforators, were found (Usai & Salvatori, 2019). Fish remains averaged 90.5% of the 74,000 Mesolithic bones studied, comprising as much as 99% at one site (16-D4-B). In fact, of all central Sudanese Mesolithic faunal assemblages, those at al-Khiday have the greatest number of fish remains relative to the total number of animal remains collected (Linseele & Zerboni, 2018).

Our data suggest that fish were not boiled in pots at al-Khiday, a trend that increasingly appears to be trans-Saharan (Dunne et al., 2020, 2021). Fish could have been wrapped in leaves and cooked on hot stones or coals in the fire or by other methods. The presence of articulated fish skeletons at al-Khiday suggests sun-drying practices, thought to have been practiced by Iron Age people inhabiting the Sahel (Van Neer, 1995) and traditional fishermen at Lake Chad (Blache et al., 1962). Fish could also have been smoked or salted. Indeed, the mineralogical analysis revealed the presence of salt (halite, NaCl) on fish bones and the inner surfaces of several potsherds at site 16-D-4B (Maritan et al., 2018). Large storage containers, with a rim diameter of between 44 and 50 cm, found at 16-D-4 and 16-D-4B (Salvatori, 2012) and similar to those found at Kobadi (Jousse et al., 2008) and in the Atbara region of the Middle Nile (Haaland, 1995), could also have been used to store cured or salted fish.

### Plant Processing: al-Khiday in Regional Context

The survival of archaeobotanical remains at early northern African archaeological sites is patchy due to a lack of systematic sampling and poor preservation of botanical remains, often caused by natural and anthropic post-depositional disturbances (Salvatori, 2012). Lipid analysis of vessels can thus provide direct evidence for the processing of plants within vessels, as demonstrated above. Notably, at al-Khiday, most vessels (82%) were used to process plants (Fig. 5a, c), whether solely or with animal products. Based on the presence of LCFAs and diacids, these plants likely comprised wild grasses, leafy plants, and, possibly, underground storage organs such as sedges. While evidence for plants is rare at al-Khiday, wild grasses of  $C_4$  origin were found at nearby sites, such as Kadero, Umm Direiwa, Zakiab, Shaheinab, Saggai, and Shaqadud cave, in Central Sudan (Ryan et al., 2016). These remains mostly comprise wild panicoid grasses such as Panicum sp., Setaria sp., Sorghum sp., Echinochloa sp., and Pennisetum sp., often identified as impressions of plants on pottery rather than found as macro-remains. A recent analysis of phytoliths and dental calculus collected from two Early Neolithic graves at Ghaba, Central Sudan-Grave 233 (6620±40 BP, 5620-5480 cal BC (Beta-59170)) and Grave 295 (5800  $\pm$  40 BP, 4729–4544 cal BC (Beta-371517)) revealed a dominance of  $C_4$  grasses, including grasses of the large Paniceae tribe, suggesting the exploitation of mixed stands of probably non-domesticated savanna grasses (Out et al., 2016). Macrobotanical remains are absent at al-Khiday, but microbotanical screening of the deposits at 16-D-3 yielded phytoliths, including wild grasses (both Panicoid and Chloridoid) and sedges, with no changes evident through the sequence. Starch from the sedge plant, *Cyperus rotundus*, and grasses of the family Triticeae was also recovered from the calculus of Pre-Mesolithic, as well as Neolithic individuals (Buckley et al., 2014). The extensive use of grinding stones at al-Khiday also highlights the importance of plant processing (Usai & Salvatori, 2019).

## The Possible Exploitation of Cyperaceae (Sedges)

The presence of LCFAS, together with diacids, in six of the al-Khiday Mesolithic vessels (27%) is suggestive of the presence of suberin, the outer covering of underground plants-corms, roots, and tubers from underground storage organs. The likely sources of tubers and rhizomes at Mesolithic al-Khiday are the Cyperaceae (sedges), including nut-grass (Cyperus rotundus), club-rush (Scirpus maritimus or S. tuberosus), catstail or reedmace (Typha spp.), bulrushes (Schoenoplectus spp.), papyrus (Cyperus papyrus), and the common reed (Phragmites australis), together with other wild tubers grown in Sudan such as Xanthosoma sagittifolium and Colocasta antiquorum (Ferguson, 1954). One of the main candidates is Cyperus rotundus, a C<sub>4</sub> plant known as the purple nutsedge or nutgrass, abundant throughout much of the Nile Valley today, growing in dense swards at the water's edge (Wetterstrom, 1993).

Today, their rhizomes are widely used as food by humans (Ertuğ, 2000; Gragson, 1997; Marlowe & Berbesque, 2009; Mattalia et al., 2013; Ochoa & Ladio, 2015; Vickers & Plowman, 1984; Vincent, 1985) and as animal feed/forage (Abbiw, 1990; Kern, 1974; Kulhari & Joshi, 1992; Parsons & Cuthbertson, 1992). There is also a long history of use of *Cyperus rotundus* for non-culinary purposes; for example, in perfume manufacturing by the Mycenaeans (Negbi, 1992) and for aromatic purposes in Predynastic Egypt (Fahmy, 2005). Various ancient writers, including Herodotus and the Hippocratic physicians (fifth century BC) and Theophrastus, Pliny, and Dioscorides (first century AD), also discuss its use as both perfume and in medicine (Manniche, 1989; Negbi, 1992).

# Evidence for Cyperus Exploitation in Holocene North Africa

In the Pleistocene (ca. 17,000 to 15,000 BC), a diverse assemblage of wild plants, dominated by carbonized rhizomes of several sedges (Cyperus rotundus, Scirpus maritimus, and Scirpus tuberosus), was found at the site of Wadi Kubbaniya, Egypt (Hillman, 1989) and at the early Neolithic Nabta Playa site, E-75-6, in the Western Desert of Egypt (ca. 6900 BC). Over 120 wild plant taxa were identified, including wild grasses, small-seeded legumes, and fruit and tuber plants (Wasylikowa et al., 2001; Wendorf et al., 1992). There were also parenchymatous tissues derived from root and tuber organs, with the majority comprising tubers of Cyperus rotundus (Hather, 1995). Tubers and nutlets of Scirpus cf. tuberosus/maritimus in charred human feces at Wadi Kubbaniya provide conclusive evidence of their dietary use (Hillman et al., 1989). In the Libyan Sahara, Cyperaceae (undiff and Carex sp.) were found at Uan Tabu, and Cyperus rotundus was identified at Uan Afuda (Castelletti et al., 1999), both dating to the Late Acacus period ca. 7000-5500 cal BC.

Notably, a combined analytical and morphological analysis of material extracted from dental calculus of seven burials et al-Khiday demonstrates the ingestion of *Cyperus rotundus*. The compound calarene ( $\beta$ -gurjunene), known to be present in the essential oil component of *C. rotundus* (Mekem Sonwa & König, 2001), together with a suite of monoterpenoid and sesquiterpenoids previously identified in the rhizomes and tubers of *C. rotundus* (Meena et al., 2010; Mekem Sonwa, 2000; Mekem Sonwa & König, 2001), is indicative of the plant species. It is identified in dental calculus samples from all periods, from the pre-Mesolithic, Neolithic, and Meroitic, covering more than 9000 years (Buckley et al., 2014).

## Conclusion

Detailed and comprehensive excavations of welldefined stratigraphic sequences at al-Khiday yielded a unique opportunity to use organic residue analysis to investigate dietary and subsistence behavior over the long duration of the Khartoum Mesolithic and into the Neolithic transition in central Sudan. Notably, while zooarchaeological data from the Mesolithic deposits suggests both an intensive and extensive strategy of aquatic exploitation, there is no evidence that these aquatic products were cooked in pots. Instead, the vessels provide direct chemical evidence of plant gathering and processing, which has long been postulated to be a major part of Holocene subsistence in Sudan. The lipid residues, backed up by experimental analysis, confirm the direct processing of abundant wild grasses found in nearby savanna drylands as well as sedges and leafy vegetables, likely located in humid areas along the Nile. Non-ruminant fauna, possibly wild pig and low lipid-yielding reptiles such as Adanson's mud turtle and Nile monitor lizard, may also have been processed alongside the plant resources. Surprisingly, there is little evidence for the processing of ruminants in the pots, despite wild ruminants contributing the majority of identified mammalian taxa (although it is important to note that the total NISP by phase for identified taxa is still very low). This suggests either that hunting was a minor aspect or that large wild fauna may have been cooked in different ways, similarly to the aquatic resources, possibly roasted over fires. Intriguingly, links to other, more northern groups exploiting domesticates may have resulted in the acquisition of a vessel containing dairy products.

Pottery use changed considerably in the Neolithic, as vessels were used to process mainly carcass products from ruminant animals. The question of whether these were domesticated ruminants, however, remains open. Although the Neolithic faunal assemblage is small and rather poorly preserved, it is dominated by wild ruminants, such as antelopes, with scarce evidence for domesticates, suggesting that hunting was an important activity. Nonetheless, minor evidence for dairying confirms either some management of cattle, sheep, or goat for milk or trade with surrounding pastoralists. Certainly, comparison to lipid residue analysis from other Neolithic pottery assemblages (Kadero and Khor Shambat 1: Dunne et al., 2017, 2021) suggests varied and sophisticated economic strategies by middle Holocene groups in this area, possibly related to local ecosystem variation.

**Dedication** We would like to dedicate this paper to our dear friend and much-loved colleague Sandro Salvatori, who was the co-director of the Centro Studi Sudanesi e Sub-Sahariani-ONLUS, working at the site of al-Khiday for 20 years. In following his passion for archaeology, Sandro's remarkable career in the Near East, Italy, Central America and, of course, Egypt and Sudan, spanned over 40 years. His contribution to Northeast African archaeology leaves a vast and enduring legacy, which we know will be called upon for many years to come. We would like to express our deep gratitude, respect and appreciation for both his scholarship and constant generosity. He continues to inspire.

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Data Availability All data are available in the paper.

#### Declarations

**Conflict of Interest** The authors declare no conflict of interest.

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