Foodstuffs and organic products in ancient SE Arabia: preliminary results of ceramic lipid residue analysis of vessels from Hili 8 and Hili North Tomb A, al Ain, United Arab Emirates

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Summary
Exchange networks in the Bronze Age between SE Arabia, Mesopotamia, SE Iran, SW Pakistan and the Indus Valley moved a variety of raw and finished products, especially pottery. However, we have little understanding of what organic products were a part of these exchange networks, as well as what foodstuffs were prepared in ceramic vessels as part of everyday activities. This paper presents the preliminary results of lipid residue analysis of local and imported vessels from Hili 8 and Hili North Tomb A in al-Ain, United Arab Emirates (UAE). Absorbed lipids were extracted and analysed via Gas Chromatography-Mass Spectrometry (GC-MS) from a range of vessels, including local, regional, Indus, Mesopotamian and Makran wares. A majority of the lipid profiles were indicative of degraded animal fats, however some vessels, including Fine Red Omani Ware and imported Black-Slipped Jars from the Indus Valley, had evidence for plant oils. Further analyses that will shed light on the possible origin of the animal fats and plant oils are ongoing. The preliminary results provide new insights into the use of pottery at Hili, with broader implications for our understanding of subsistence and exchange networks in the region.

Keywords: Ceramics, Lipid residues, Vessel use, Subsistence, Hili 8, Hili North Tomb A

1. Introduction

From the Late Pleistocene, the Arabian Peninsula has served as an important corridor for the movement of people, goods and ideas (see Armitage et al. 2011; Bretzke et al. 2013 for discoveries in the United Arab Emirates). Archaeological research has revealed evidence of exchange networks between eastern Arabia, Iraq and Iran beginning in the Neolithic, then transforming and expanding up to South Asia during the Bronze Age (Méry 2000, Cleuziou and Tosi 2007, Carter 2010, Magee 2014). By *c*. 2600 B.C, settlements and tombs across south-eastern (SE) Arabia provide material that suggest contacts with Mesopotamia, eastern Arabia, Iran, and the Indus Valley (Vogt 1996, Méry 2010, Thornton 2013, Frenez et al. 2016). These include imported pottery, beads and objects from precious and semi-precious stones, gold and ivory (Potts 1993, Cleuziou and Tosi 2007), as well as organic products such as imported wood (Tengberg 2002). While people no doubt moved with these goods, the settling of immigrant communities in SE Arabia has also been hypothesised (Frenez et al., 2016; Méry et al., 2017), and strontium isotopic analyses have detected the presence of a small number of non-locals in communal tombs located on the coast (Gregoricka 2013). Knowledge about the movement of products and people during this period also comes from Mesopotamian textual sources, with references to regions with toponyms such as 'Dilmun', 'Magan' and 'Meluhha' that have been identified as Bahrain/eastern Arabia or SE Iran/SW Pakistan and the Indus Valley respectively (Ratnagar 1981, Potts 1991, Potts 2012). These texts refer to the movement of copper, tin, wood, textiles, perfumes or cosmetics, animals, food and fodder (Ratnagar 1981, Potts 1990, Potts 1993, Weeks 2003, Laursen & Steinkeller 2017). The movement of specific organic products in ceramic vessels may have also constituted an important part of the cross-cultural exchange economy (Gouin 1990, Potts 1993, Méry 2000). Although a number of studies have been conducted to determine the geological/geographical source of different types of pottery and their technology, our knowledge of what vessels were used for, or what they contained, is very limited. Studies have characterised the use and transport of bitumen in ceramic vessels (Connan 1999, van de Velde 2015, van de Velde et al. 2017), but absorbed residues in locally-produced and imported pottery have not been investigated in detail to achieve a broader understanding of subsistence practices or exchange networks.

The introduction and uptake of pottery in SE Arabia also presents an interesting case-study to understand reasons behind the spread and use of ceramic technology. Pottery only seems to have begun to be incorporated in the material culture of the Oman Peninsula around 3000 BC (Cleuziou 1989a: 52). The earliest locally-produced pottery known in SE Arabia draws on the stylistic traditions of south-east Iran (Sistan, Makran) and south-west Pakistan (Makran), strongly suggesting an exchange of both ideas as well as craftspeople (Méry 1991, 2011, Potts 2005). By *c.* 2600 BC, together with southern Mesopotamian pottery, imported vessels from Sistan, Makran and the Indus Valley are found in tombs as well as from settlements (Méry 1991, 2000, Cleuziou and Tosi 2007). Did early pottery in the region have a specialized purpose (Cleuziou 1989a: 52)? What factors may have influenced the local production of pottery in the region? Did specific forms have specific roles in everyday or funerary life in later periods? While pottery has been widely studied and used as both a cultural and chronological marker in Arabian archaeology, its cultural *use* has not been investigated in detail through the analysis of absorbed organic residues.

Chemical and spectrometric methods, such as gas-chromatography and mass-spectrometry, provide a means to extract, identify and reconstruct the contents of ancient vessels, enabling an understanding of foodstuffs that were cooked or stored in ceramic vessels, as well as other organic products (Evershed 2008). This paper presents the preliminary results of lipid residue analysis of pottery from Hili 8 and Hili North Tomb to characterise the extent of lipid preservation in the pottery and the types of organic products that can be detected in both locally-produced and imported pottery. These preliminary results will be incorporated into a broader study of organic residues in pottery from different Bronze Age sites in SE Arabia to understand the movement and everyday use of organic products and foodstuffs across the region.

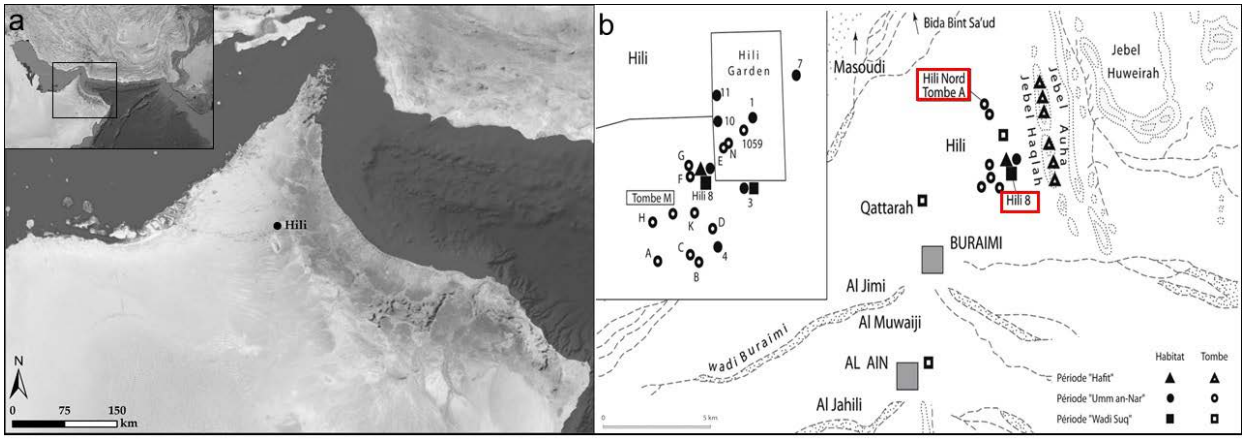

*Figure 1: The location of Hili in south-eastern Arabia; **b.** a map of Hili Garden City and the location of Hili 8 and Hili North Tomb A (in red squares) (**a.** Map by Sophie Costa; **b.** modified from Méry 2013).*

2. Sites and context

2.1. Hili 8

Located in the al-Ain oases on the western foothills of the Hajar mountains, Hili is a large complex of sites including both settlements and tombs spread across at least 25 ha, located about 150 km east of Abu Dhabi, in the eastern province of the Abu Dhabi Emirate, United Arab Emirates (Figure 1). Within the Hili complex, the settlement of Hili 8 provides the longest stratified sequence for the Early Bronze Age (EBA) in south-eastern Arabia (known as the Hafit and Umm an-Nar periods in Arabian archaeology), to the Middle Bronze Age (Wadi Suq period), and possibly the Late Bronze Age (LBA) (Figure 2). The principal architectural remains at Hili 8 consisted of the base of a solid, compartmented mud-brick tower, built at the beginning of the Hafit period (Phase la), with further constructions in the beginning of the Umm an-Nar period (Phase IIa), and at the end of the Umm an-Nar period (Phase IIf), as well as ditches, other mud-brick constructions, and wells (Cleuziou 1989a, 1989b, Cleuziou and Tosi 2007: Figure 147)(Figure 2). Although not published in full detail (Cleuziou 1989a, 1989b), the site presents one of the more reliable chronologies for the EBA, and a reliable pottery sequence for the Bronze Age in the region (Méry 2000).

The site of Hili 8 provides an ideal context to investigate lipid residues in pottery in SE Arabia as it enables the study of vessel use across multiple time periods. Additionally, it provides evidence of early locally-produced pottery in SE Arabia, as well as a diverse range of imported vessels from Mesopotamia, Sistan, Makran and the Indus Valley, allowing for investigations into the earliest uses of pottery in the region and whether there were specialised uses for different types of pottery.

	Hili 8			**Hili North Tomb A**
Cultural period	**Site Period**	**Architectural Phase**	**Date range (cal BC)**	
Hafit	Period I	Ia-b	c.3200-2900	
		Ic	c. 2900-2700/2600	
Umm an-Nar	Period II	IIa-IIc1	c. 2700/2600-2500	
		IIc2-IIe	c. 2500-2200	
		IIf-IIg	c. 2200-2000	Hili IIf; c. 2200-2000
Wadi Suq	Period III	NA	c. 2000-1600	

Figure 2: The chronology of Hili 8 and Hili North Tomb A (revised by S. Méry in 2021).

2.1.1. Imported and locally-produced ceramics across time

During Period I at Hili 8, only a small number of ceramics were discovered, with fragments corresponding to about a dozen pottery vessels (Cleuziou 1989a: 74). Of this small assemblage, three small jars and two bowls were classified as being Mesopotamian in origin (Cleuziou 1989b: 50), while 10 flat bases and 3 necks were assigned to locally/regionally-produced Omani fine black-on-red pottery (Cleuziou 1989b: 51, Méry 2000: 80-85). Black-on-red ware represents a quarter of the pottery assemblage at Hili 8 during this period, and has no clear predecessors in the UAE or Oman. On the other hand, it is closely related to identical wares in SE Iran and SW Pakistan, and petrographic analysis shows that some are certainly imports from this area (Cleuziou & Méry 2002, Méry 2000: 99-103). The rapid appearance of locally-produced fine ceramics that required relatively high levels of skill and ability to manufacture, along with pyrotechnical knowledge of elaborate kilns, is taken as evidence that this "knowledge was borrowed from elsewhere" (Cleuziou & Méry 2002: 282). It is highly probable that potters from Makran travelled to SE Arabia and settled there (Potts 2005, Méry 2013, Thornton and Ghazal 2016). A study of the contents of these early vessels may provide insight into their use.

By Phases IIa and IIb, Mesopotamian jars (sometimes with a ring base) account for most of the pottery assemblage, but are still only represented by a handful of sherds (Cleuziou 1989b: 75). By Phase IIc, Mesopotamian sherds are still found at Hili 8, but there is an increase in evidence of Emir-type ceramics and Incised Grey wares from south-west Pakistan and south-eastern Iran (Cleuziou 1989b: 76). During phase IIc2, the Omani fine black-on-red pottery represents 13% of the assemblage, then it drastically decreases, associated mostly in funerary contexts (Méry 2000: 79). At this time, a locally produced utilitarian pottery known as Sandy Red Ware (SR-HI) is found predominately in domestic contexts at Hili (Méry 2000:126). This ware is present in Period I, where it represents 5% of the pottery, however the stratigraphic context of the sherds is not very reliable (see Méry 2000: 125 for discussion) thus this type of production is likely related to the Umm an-Nar period only, at least at Hili 8. From Phase IIc2, SR-HI ware represents 85% of the assemblage and the percentage of this ware relative to others increases until the end of the Umm an-Nar period at Hili 8 (Méry 2000: 125-126).

Associated for the first time with phase IId at Hili 8 and present until period III, ceramics produced in the Indus Valley are also found, of which Black-Slipped Jars (BSJs) are most common (Cleuziou 1989b: 78, Cleuziou 2002: 209). Petrographic and chemical analysis suggests these large amphora-like jars were produced in the southern part of the Indus Valley (Blackman et al. 1989, Méry 2000: 219-228, Méry & Blackman 1999, 2004) and are presumed to be associated with the import of products from there (Dales and Kenoyer 1986: 83-84, Méry 2000: 226).

2.2. Hili North Tomb A

Tomb A at Hili North is 2 km NNW of Hili 8 and consists of a multi-chambered collective burial tomb. Most probably used for a period of no more than 200 years, it is contemporaneous with phases IIf-g at Hili 8 (Cleuziou & Vogt 1985, Cleuziou et al. 2011; Figure 2). Tomb A is one of the largest known examples of 'Umm an-Nar type' monumental circular-type tombs, which are widespread throughout the Oman Peninsula from the second half of the 3rd millennium B.C. (Frifelt 1975a, 1975b, Cleuziou & Tosi 2007, Méry and Charpentier 2009, Munoz 2014). Built of stones and faced with large ashlars and a single doorstone, Tomb A contained four parallel chambers in which more than 80 individuals were buried (Cleuziou et al. 2011).

Around 900 pottery vessels were recovered from the tomb, of which about 10% of the vessels were imported, and included a large proportion of Indus and Makran/Sistan pottery (4% and 5%, respectively) (Méry 2007: 172). Only a single Mesopotamian bottle has been found in the tomb, which is consistent with other graves of the end of the Umm an-Nar Period in inland Oman which reflect a paucity of Mesopotamian vessels (Méry & Schneider 1996: 83).

The diversity of Makran/Sistan wares is large and several types of pottery are represented: grey, (or less often) red pasteware, fine (or less often semi-fine) granulometry, painted or incised decoration, or not decorated. However, petrographic and/or chemical analysis of a large fraction of these vessels showed that most of them were probably *not* imported, but produced locally (Blackman et al. 1989: 71-72, Méry 2000: 85-217). Of the locally- or regionally-produced vessels found in the tomb, 65% are Hili Sandy Red Ware and 19% are Omani Fine Red Ware (Méry 2007: 172). This suggests that while imported vessels were indeed interred as part of the burial process, most of the vessels used were local in origin. The analysis of lipid residues from both imported and locally-produced vessels from the tomb may provide a glimpse into contents of funerary offerings.

2.3. Subsistence economy at Hili 8

2.3.1. Animal economy

From the earliest occupation of Hili 8 through the Umm an-Nar period, the animal economy was made of more than 90% of domestic fauna (Uerpmann & Uerpmann 2008: 468). Cattle contributed around 60% of the domestic remains, while small ruminants such sheep and goats constituted the rest (Uerpmann & Uerpmann 2008: 468-469). During the Period I, sheep seemed to slightly outnumber the goats. A small sample studied from the Umm an-Nar period indicated a reversal of sheep/goat proportions at Hili 8.

During the entire sequence of Hili 8 animal husbandry appears to have been "stable and sufficient for the meat supply", with hunting being of little importance, except for a few gazelle bones found in all phases (Uerpmann & Uerpmann 2008: 468). Four wild camel bones were found in Phase Ib, but no camel remains were found from the Umm an-Nar period (Period II). The remains of ass are found through Phase I to Phase II at Hili 8, with an increase in bone weight from Phase I to Phase II, which is interpreted as an indication of domestication (Uerpmann 1991, Uerpmann & Uerpmann 2008: 470). Importantly, Hili 8 did not provide any evidence of fish bones or other marine animal remains (Uerpmann & Uerpmann 2008: 470).

Period I of Hili 8 provides insight into the age structure of the slaughtered animals, suggesting that the majority of animals (80%) were slaughtered before they reached the age of 4 years, which points to their main use as a source of meat (Uerpmann & Uerpman 2008: 480). Some of the older cattle bones have pathological alterations of the foot bones, which suggest their use as draught and transport animals (Uerpmann & Uerpmann 2008: 479).

The slaughtering curves of ovicaprine species from Hili 8 suggest a difference between the two species: the curve for sheep resembles that of cattle and indicates that sheep were mainly kept for meat production. The kill-off pattern for goats, however, suggests that about half of them lived beyond 36 months and nearly 20% lived longer than 4 years, a pattern which suggests their use for secondary products such as dairying (Uerpmann & Uerpmann 2008: 480). The use of lipid residue analysis combined with compound-specific isotopic analysis can distinguish between the ruminant carcass and dairy fats (Dudd & Evershed 1998). Thus, future analyses will be able to indicate whether dairy products were present in early ceramic vessels at Hili 8.

2.3.2. Archaeobotanical remains

The settlement of Hili 8 provides some of the earliest evidence of agriculture in SE Arabia, with early excavations reporting the remains of barley (Hordeum, sp.), wheat (Tricitum sp.) (oat (*Avena* sp.), peas (*Pisum sativum*), jujube (*Ziziphus* sp.) date stones and stem fragments of date palm (*Phœnix dactylifera*)(Tengberg 2003: 232) at the site. Although impressions of sorghum (*Sorghum bicolor)* were reportedly found at Hili 8 (Cleuziou & Costantini 1980), this has been called into question on morphological grounds (e.g. Tengberg 2003: 235, Charbonnier 2008, Tengberg 2012). It is well-established that sorghum was domesticated in northeast Africa and had different pathways of dispersion (Venkateswaran et al., 2018). The earliest identification of sorghum in India is after 2000 B.C, suggesting the eastern movement of sorghum through the Arabian Peninsula to India (Venkateswaran et al., 2018). . However, the

movement of millets in the Arabian Peninsula is still a topic that needs further exploration (Fuller & Boivin 2009, Boivin & Fuller 2009).

The remains of date palm found with cereals and pulses has led researchers to suggest the existence of oasis agrosystems at Hili by the third millennium BC, i.e., intensively cultivated date palm gardens in which perennial and annual crops were grown together (Tengberg 2012). While every part of the date palm has some use in daily life, for example, for construction, fuel, basketry, cordage, packing and padding (Tengberg 2012), it was also probably an important element of human diet in this period, as the fruit can be consumed fresh or dried, and can be prepared as beverages and in dishes with other foodstuffs (Méry & Tengberg 2009). A loaf-shaped food preparation, identified as made of date fruits, was found in a pit in Hili North Tomb A, and interpreted as a unique example of a plant food offering in SE Arabia, also suggesting the symbolic importance of dates in the Umm an-Nar period (Méry & Tengberg 2009).

Taken together, the bioarchaeological evidence from Hili 8 suggests the predominance of terrestrial ruminant animals and the presence of winter cereals, pulses and fruits. Such bioarchaeological evidence provides important contextual information for organic residue analysis as it helps to narrow down the options of the most likely sources of organic products that would have been processed in vessels (Evershed 2008: 899).

2.4. Potential imported organic products

Apart from the locally available sources of organic products, it is also important to note the potential organic products that may have been moving through exchange networks. As previously mentioned, cuneiform texts from Mesopotamia attest the movement of different products via ships from 'Dilmun', 'Magan' and 'Meluhha'. Some of the organic products listed as going to 'Magan' during the Ur III period (2100-2000 B.C) include barley, beer, flour, wool, edible plants or plant products, small fish, textiles, garments, oils and hides, in exchange for copper, ivory, gold, semi-precious stones, ochre, 'Magan-goats' (oryxes?) and 'Magan-onions' (Potts 1990: 145; Laursen & Steinkeller 2017: 55). Thus one could expect different types of plant and animal products being moved in Mesopotamian ceramic vessels to 'Magan', including barley or barley products, seed oils, other edible plants, fish, and secondary-products from animals such as hides, wool, and possibly dairy products. We do not know what organic products could have been transported in vessels from SE Iran/SW Pakistan and the Indus Valley, however, the movement of dairy products (Gouin 1990), and plant oils, such as sesame oil (Postgate 1992: 171, Laursen & Steinkeller 2017: 88) has been suggested.

Provenance	Cultural Period	H8 phasing	Sample ID	Sherd or vessel ID	Shape	Macro	Petro	INAA
HILI 8								
Mesopotamian	Hafit	Ia-b	A0002	2898 UF1348	body pot	bs1	H	no
		Ib	A0007	2878 UF1320	body pot	bs1	A	no
		I	A0012	2571 UF829b	body pot	bs1	A	no
		I	A0014	2885 UF1320	body pot	bs1	A	no
		I	A0015	2567 UF820	body pot	bs1	H	no
		Ic	A0801	2684 UF814	base jar	bs1	H	no
SR-HI		Ib	A0010	2679 UF895	body glob jar	rs1	B	no
		Ib	A0011	2678 UF893	body glob jar	rs1	B	3
Indus	Umm an-Nar	IIf	A0047	600 UF23	body BSJ	rm1	G	Indus 1
		IIf	A0074	2499 UF742	body BSJ	rm1	no	Indus 1
		IIf	A1064	119	body BSJ	rm1	no	Indus 1
SR-HI		IIa-b	A0026	2867 UF1345	body	rs1	B	3
		IIf	A0043	3097 UF23	body	rs1	B	no
		IIf	A0121	3098 UF23	body	rs1	B	no
		IIf	A0122	3099 UF23	body	rs1	B	no
		IIf	A0123	3100 UF23	body	rs1	B	3
		IIf	A0126	no n. UF23	body glob jar	rs1	no	no
		IIf	A0127	3102 UF23	body	rs1	B	no
		IIf	A0129	571 UF23	body	rs1	B	no
		IIf	A0136	3107 UF23	body	rs1	B	no
		IIf	A0145	3108 UF23	base	rs1	B	no
FR-OM		IIc2-d	A0042	1764 UF177	body pot	ra1	C	no
		IIf	A0148	67 UF1704	body pot	ra1	C	1
		IIc2-d	A0559	1153 UF100	body pot	ra1	C	no
		IIe	A0574	2139 UF555	base pot	ra1	no	no
Indus	Wadi Suq	III	A0802	750 UF59	body BSJ	rm1	G	Indus 1
Fine		III	A0008	2664 UF690	base	rs3	L	no
		III	A0050	780 UF59	body	rs3	L	no
Coarse	LBA?	III	A0049	2340 UF728	body	rg2	J	no
		III	A1110	396 UF12	body	rg1	K	no
		III	A1112	729 UF66	body	rg1	K	no
		III	A0051	756 UF59	body	rs3	L	no
HILI NORTH Tomb A								
Mesopotamian	Umm an-Nar	IIf	A0076	V77	body bottle	bs1	H	no
FG-IR Makran		IIf	A0533	V26	body pot	ga1	no	no
SG-IR Hili		IIf	A0439	V276	body pot	gs1	F	5
		IIf	A0532	no Vessel n.	body pot	gs1	no	no
FR-OM		IIf	A0410	V0366	necked pot	ra1	C	no
		IIf	A0377	V148	necked pot	ra1	no	no
		IIf	A0389	V89	necked pot	ra1	no	no
		IIf	A0413	V365	necked pot	ra1	no	no

Figure 3: Details of pottery analysed from Hili 8 and Hili North Tomb A. Key: SR-HI: Sandy Red Hili Ware; FR-OM: Fine Red Omani Ware; BSJ: Black-Slipped Jar; FG-IR: Fine Grey Painted Ware (imported); SG-IR: Sandy Grey Incised 'Iranian' Ware (probably produced locally by Makran potters); for further details of macroscopic, petrographic, and INAA analysis, see Méry 2000.

3. Method

3.1. Sample details

To begin assessing the degree of lipid preservation in the pottery, an initial sampling of 40 sherds was conducted. The sherds had been previously studied via previous macroscopic, petrographic and/or instrumental neutron activation analysis (INAA), and thus had established compositional information that could indicate their provenance (Méry 2000). The sherds were chosen depending on their provenance and chronological time period, and included 32 sherds from Hili 8, spanning across the Hafit (n=8), Umm an-Nar (n=17) and Wadi Suq and LBA (n=7) periods, and 8 sherds from Hili North Tomb A (Umm an-Nar period).

From Hili 8, imports from Mesopotamia (n=6) dating to the Hafit period, and Black-Slipped Jars from the Indus Civilisation (n=4), dating to the Umm an-Nar and Wadi Suq periods (n=3 and n=1, respectively) were analysed, along with Hili Sandy Red Ware (n=12) produced locally at the settlement (n=2 from the Hafit period and n=10 from the Umm an-Nar period) and Fine Red Omani Ware (n=4, Umm an-Nar period), produced regionally. Both Fine (n=3) Wadi Suq potsherds and samples of Coarse ware (n=3) were also analysed. Coarse Ware was considered before as dated to the Wadi Suq period, but a recent re-evaluation of Early, Middle and Late Bronze Age pottery in a wider geographical context (the UAE) indicates a more recent date, i.e., the Late Bronze Age, for these sherds. From Hili 8, the potsherds analysed were body (n=20) and base (n=2) fragments of pots, globular pots or jars, previously characterised (Méry 2000) (Figure 3).

The eight samples analysed from Hili North Tomb A included 2 imports (fragments of a Mesopotamian bottle and a Fine Grey Emir Painted pot) and 6 locally-produced pottery (2 fragments of Sandy Grey Incised wares produced at or near Hili, but most probably by potters from Makran, and 4 fragments of Fine Red Omani necked pots).

3.2. Sample extraction and analysis

Lipids were extracted from all 40 potsherds via a one-step acidified methanol protocol (Craig et al., 2013), which was slightly modified. The external surfaces of the potsherds were cleaned with a modelling drill to remove any exogenous lipids. For each sample, ~1 g of the potsherd was ground to a powder in a solvent-cleaned mortar and pestle, and 10 µl of an internal standard, *n*-triacontane (*n*-C_{30}, triacontane, 1 mg·ml^{-1} in *n*-hexane) was added. Four ml of methanol (HPLC grade) was added to the powdered samples, sonicated for 15 min and acidified with concentrated sulphuric acid (800 µl). The samples were heated (4 hours at 70 °C), and after cooling, lipids were extracted with cyclohexane (3×2 ml) and centrifuged (3000 rpm, 5 min), and finally concentrated under a flow of nitrogen. Due to the co-elution of minor contaminants (silanes) present in the cyclohexane, the internal standard used was different from what is used in established protocols (tetratriacontane, *n*-C_{34}). All the lipid extracts were analysed directly by Gas Chromatography-Mass Spectrometry (GC-MS) to quantify and identify the different organic compounds in the extracts.

A selection of fragments (n=22) were also analysed via conventional solvent extraction to test for the preservation of triacylglycerols and esters. Remaining available potsherd fragments (1-2 g) were cleaned with a modelling drill and ground to a powder in a solvent-cleaned mortar and pestle. Then, an internal standard (20 µg of *n*-C_{34}, 1 mg·ml^{-1} in *n*-hexane) was added, and 10 ml of solvent (dichloromethane/ methanol 2:1 v/v; HPLC grade) was added to the powder and sonicated. After centrifugation, the supernatant was evaporated to dryness and dissolved in 500 µl of DCM/MeOH to obtain the total lipid extract (TLE). Aliquots of the TLE were trimethylsilylated (N,O-bis(trimethylsilyl)trifluoroacetamide, 50 µl, 70 °C, 60 min), and submitted to analysis by GC and GC–MS.

GC analyses were performed for the solvent extracts on an Agilent Technologies 7890A device. 1 µl of sample was introduced via an on-column injector into a 15 m × 0.32 mm i.d. fused silica capillary (DB5-MS, 0.1 µL film thickness, Agilent J&W), with helium used as

carrier gas. The GC temperature programme was as follows: increased from 50 °C to 100 °C at 15 °C min^{-1}, then from 100 °C to 375 °C at 10 °C min^{-1}.
GC-MS analysis of both solvent and acidified methanol extracts were performed on a Shimadzu GC 2010 PLUS chromatograph coupled to a Shimadzu QP 2010 ULTRA mass spectrometer, fitted with a high temperature non-polar column (DB5-HT, 15 m × 0.322 mm i.d., 0.1 μm film thickness, Agilent J&W). The injection was performed using a splitless injector. The temperature programme consisted of a 1 min isothermal hold at 50 °C followed by an increase to 150°C at 20 °C·min^{-1}, then to 250 °C at 10 °C·min^{-1} and to 350 °C·min^{-1} and a final isothermal hold for 10 min. The GC–MS interface was maintained at a temperature of 300 °C and the mass spectrometer run in electron ionization mode (EI, 70 eV). Mass spectra were acquired over the range *m/z* 50–950.

4. Results

4.1. Survival of lipids and contamination

All the potsherds had interpretable lipid concentrations (above 5 μg g^{-1})(Heron et al., 1991) via extraction with the acidified methanol protocol, with some fragments demonstrating excellent lipid yields (above 1 mg g^{-1}). The mean lipid yields for sherds from Hili 8 was 697 μg g^{-1} and median was 177 μg g^{-1}. In particular, the Mesopotamian sherds from Hili 8 demonstrated very high lipid concentrations (mean: 2.8 mg g^{-1}) (Figure 4). From Hili North Tomb A, the mean lipid yield of all analysed fragments was 377 μg g^{-1} while the median was 260 μg g^{-1}. Figure 4 provides the average and median lipid concentrations for each potsherd type by time period for both Hili 8 and Hili North Tomb A. Lipid concentrations were relatively higher to those obtained from other arid regions, particularly South Asia, also using acidified methanol extraction) (Chakraborty et al., 2020; Suryanarayan et al., 2021).
All the samples had high levels of contaminating compounds such as plasticisers, which are commonly detected when analysing archaeological pottery. This is probably a result of the sherds being stored in plastic bags after excavation for at least 30 years. Many sherds had also been labelled with a combination of varnish, correction-fluid and ink, which could lead to the addition of contaminating compounds to the lipid extract. Although plastic contaminants are easily distinguished in the chromatograms, their presence can be problematic as they could mask other informative compounds that are of archaeological origin. Other contaminating compounds introduced via the storage environment may also affect the interpretation of results.

Provenance	**Cultural Period**	**H8 phasing**	**Sample ID**	**Shape**	**Acidified methanol extraction**			**Solvent extraction**
					Lipid yield (µg.g-1)	Average lipid yield (µg.g-1)	Median lipid yield (µg.g-1)	TLE (µg.g-1)
Mesopotamian	Hafit	Ia-b	A0002	body pot	4029			16
		Ib	A0007	body pot	5265			9
		I	A0012	body pot	96			7
		I	A0014	body pot	4324			6
		I	A0015	body pot	2256			
		Ic	A0801	base jar	777	2791	3142	2
SR-HI		Ib	A0010	body glob jar	391			4
		Ib	A0011	body glob jar	249	320		4
Indus	Umm an-Nar	IIf	A0047	body BSJ	146			trace
		IIf	A0074	body BSJ	82			3
		IIf	A1064	body BSJ	149	126	146	14
SR-HI		IIa-b	A0026	body	171			
		IIf	A0043	body	35			
		IIf	A0121	body	38			
		IIf	A0122	body	99			
		IIf	A0123	body	63			
		IIf	A0126	body glob jar	74			trace
		IIf	A0127	body	79			trace
		IIf	A0129	body	50			
		IIf	A0136	body	81			
		IIf	A0145	base	59	87	68	
FR-OM		IIc2-d	A0042	body pot	182			
		IIf	A0148	body pot	184			
		IIc2-d	A0559	body pot	37			
		IIe	A0574	base pot	411	204	183	
Indus	Wadi Suq	III	A0802	body BSJ	223			32
Fine		III	A0008	base	209			8
		III	A0050	body	322	265		
Coarse	LBA?	III	A0049	body	191			
		III	A1110	body	1521			2
		III	A1112	body	104			
		III	A0051	body	395	673	395	5
				TOTAL		697	177	

HILI NORTH TOMB A								
Provenance	**Cultural Period**	**H8 phasing**	**Sample ID**	**Shape**	**Acidified methanol extraction**			**Solvent extraction**
					Lipid yield (µg.g-1)	Average lipid yield (µg.g-1)	Median lipid yield (µg.g-1)	TLE (µg.g-1)
Mesopotamian	Umm an-Nar	IIf	A0076	body bottle	61			trace
FG-IR Makran		IIf	A0533	body pot	211			
SG-IR Hili		IIf	A0439	body pot	976			4
		IIf	A0532	body pot	663	819		2
FR-OM		IIf	A0410	necked pot	459			2
		IIf	A0377	necked pot	128			4
		IIf	A0389	necked pot	249			3
		IIf	A0413	necked pot	271	277	260	
				TOTAL		377	260	

Figure 4: Lipid yields of acidified methanol extracts and solvent extracts of pottery analysed from Hili 8 and Hili North Tomb A. Key: TLE: Total Lipid Extract; SR-HI: Sandy Red Hili Ware; FR-OM: Fine Red Omani Ware; BSJ: Black-Slipped Jar; FG-IR: Fine Grey Painted Ware; SG-IR: Sandy Grey Incised 'Iranian' Ware.

4.2. Molecular characterisation

4.2.1. Acidified methanol extracts

A majority of the lipid profiles of the acidified methanol extracts showed the presence of saturated fatty acids ranging from $C_{12:0}$ to $C_{28:0}$, mainly dominated by palmitic acid ($C_{16:0}$), myristic acid ($C_{14:0}$) and stearic acid ($C_{18:0}$). They also contained odd-chain fatty acids such as $C_{15:0}$ and $C_{17:0}$, as well as branched-chain fatty acids such as C_{14}, C_{15} and C_{17}. Such profiles are characteristic of degraded animal fats (Dudd & Evershed 1998, Dudd et al., 1999) (Figure 5a). Almost all the vessels also contained peaks of unsaturated fatty acids such as $C_{16:1}$, $C_{18:1}$ and $C_{22:1}$. The presence of more than one positional $C_{18:1}$ isomer in every sample, as well as those of branched-chain fatty acids, may be indicative of ruminant fats (Evershed et al. 1997, Dudd et al. 1999).

All vessels also had evidence of a single α,ω-dicarboxylic acid, specifically C_9, while two vessels exhibited the presence of α,ω-dicarboxylic acids C_9 to C_{12}. Short-chain α,ω-dicarboxylic acids are formed through a variety of oxidative degradation mechanisms of the double bond(s) in unsaturated fatty acids (Regert et al. 1998).The presence of the C_9 diacid likely implies the presence of moieties with unsaturation mainly at position 9 (most probably a Δ9 fatty acid) (Regert et al. 1998; Copley et al. 2005). Dicarboxylic acids may indicate the cooking of plant and animal resources as they are typical oxidation products of unsaturated fatty acids, although they can also result from different degradation processes occurring during burial (Regert et al. 1998; Copley et al. 2005).

Twelve extracts had very small peaks for odd and even long-chain *n*-alkanes. Wax covering the leaf and stem surfaces of plants is one of the main sources of *n*-alkanes (Kolattukudy 1970), however, the *n*-alkane distributions did not show a very typical profile associated with the processing of leafy plants, and none of the tri-methylsilylated samples (n=23) indicated the presence of *n*-alkanols, also indicative of plants or beeswax products. As *n*-alkanes are also common lipid components of soils (van Bergen et al. 1998), their presence in the pottery could be related to the burial context, and is not particularly diagnostic.

Three vessels (A802 and A1064 from Hili 8, A410 from Hili North Tomb A) had higher abundances of unsaturated $C_{18:1}$ than $C_{18:0}$, and a small peak of $C_{18:2}$, while other vessels (A148 from Hili 8, A377 from Hili North Tomb A) had relatively high abundances of $C_{18:1}$ (*trans* and

cis moieties). As high concentrations of unsaturated fatty acids are present in plant oils (Copley et al. 2005), it is likely these vessels contained plant oils (Figure 5b and 5c).

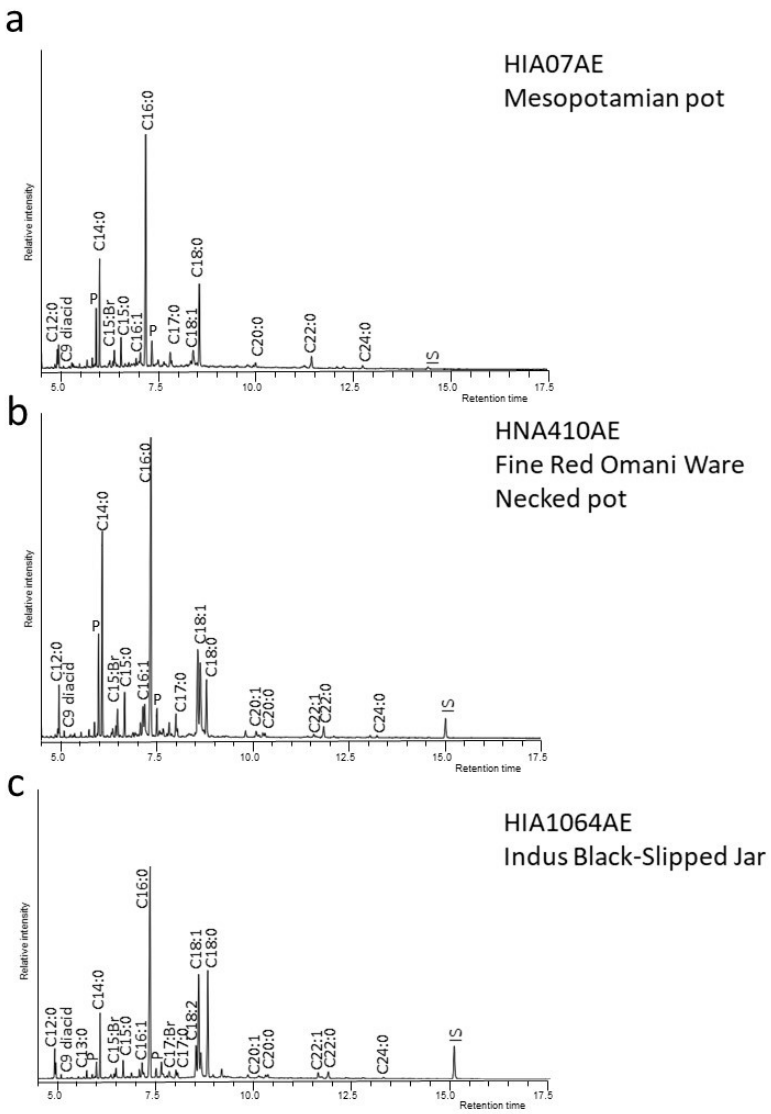

*Figure 5: Chromatograms of acidified methanol extracts from vessels from Hili 8 (A007 and A1064) and Hili North Tomb A (HNA410AE); **a** has a profile typical of degraded animal fats, whereas **b** and **c** have profiles suggestive of the presence of plant oils (high abundances of C18:1); Cn:x indicates fatty acid with n carbon atoms and x double bonds. Key: IS: Internal Standard; P: plasticiser; diacid: dicarboxylic acid; Br: branched-chain fatty acid.*

4.2.2. Solvent extracts

Solvent extraction of selected potsherds (*n*=22) exhibited poor lipid yields, with only 6 containing more than 5 μg g^{-1} of lipid; and plasticisers and contaminants dominating the extracts. Despite very low lipid concentrations, free fatty acids ($C_{16:0}$, $C_{18:0}$), trace quantities of monoacylglycerols, diacylglycerols and cholesterol derivatives were detected, and 4 extracts demonstrated the survival of triacylglycerols (TAGs).

One Mesopotamian vessel (A002) from Hili 8 had a total lipid extract (TLE) of 16 μg g^{-1}, and exhibited free fatty acids $C_{12:0}$-$C_{18:0}$, monoacylglycerols, diacylglycerols, cholesterol derivatives and trace concentrations of TAGs. Two vessels, A802 and A1064, both Black-Slipped Indus Jars found at Hili 8 in levels dating to the Wadi Suq and Umm an-Nar periods, respectively, had TLEs above 5 μg g^{-1} (32 μg g^{-1} and 14 μg g^{-1}, respectively) and demonstrated very high survival of unsaturated TAGs (over 50% of the TLE), with T_{54} (triolein) dominating,

and the T_{52} and T_{50} peaks comprising oleic and palmitic acid moieties (Copley et al., 2005; Garnier et al., 2009; Drieu et al. 2018), which were identified based on their specific mass spectra and retention time (Figure 6). This particular profile matches TAG profiles of different types of plant oils, which include olive oil, linseed and sesame oil (Copley et al., 2005). These extracts also contained high quantities of unsaturated $C_{18:1}$, trace quantities of $C_{18:2}$, and stigmasterol, further confirming the presence of degraded plant oils.

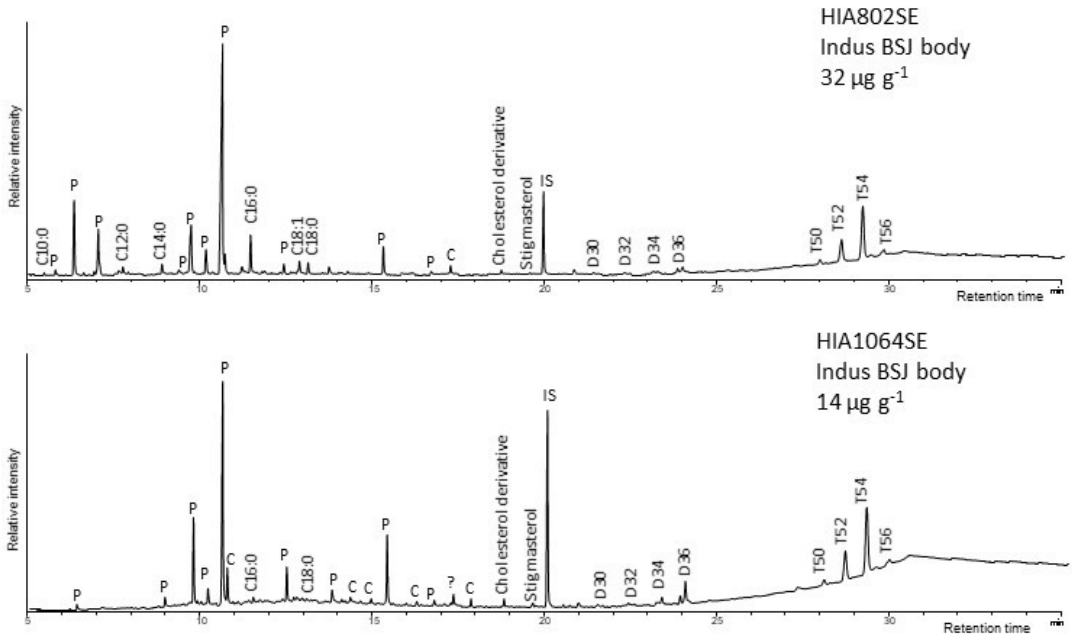

Figure 6. Chromatograms showing the total lipid extract (TLE) and relative distributions of triacylglycerols of HIA802 and HIA1064, two Indus Black-Slipped Jars from Hili 8 that are indicative of plant oils; Cn:x indicates fatty acid with n carbon atoms and x double bonds.Key: IS: Internal Standard; C: contaminant; P: plasticiser; ?: unknown compound; D: diacyglycerol; T: triacylglycerol.

Provenance	Cultural Period	H8 phasing	Sample ID	Shape	Acidified methanol extraction				Solvent extraction	
					Lipid yield (µg.g-1)	P/S ratio	Major compounds detected	Interpretation	TLE (µg.g-1)	Compounds detected
Mesopotamian	Hafit	Ia-b	A0002	body pot	4029	1.8	FAs(C12:0-C26:0), BrFAs (C14, C15, C17), UFAs (C16:1, C18:1, C22:1), Diacids (C9)	Degraded animal fats	16	FAs(C12:0, C14:0, C16:0, C18:0) UFAs (C16:1, C18:1), cholesterol deriv., MAGs (M16, M18), DAGs(D32,D34, D36), TAGS(trace)
Mesopotamian	Hafit	Ib	A0007	body pot	5265	1.1	FAs(C12:0-C24:0), BrFAs (C14, C15, C17), UFAs (C16:1, C18:1, C22:1), Diacids (C9)	Degraded animal fats	9	FAs(C16:0, C18:0), UFAs (C16:1, C18:1), cholesterol deriv., MAGs (C16, 18), DAGs(D32, D34, D36), TAGS(trace)
Fine	Wadi Suq	III	A0008	base	209	2.5	FAs(C12:0-C26:0), BrFAs (C14, C15, C17), UFAs (C16:1, C18:1, C22:1), Diacids (C9)	Degraded animal fats	8	FAs(C16:0, C18:0), MAGs (M16, M18), cholesterol deriv., DAGS(trace), TAGs (trace)
SR-HI	Hafit	Ib	A0010	body glob jar	391	2.6	FAs(C12:0-C24:0), BrFAs (C14, C15, C17), UFAs (C16:1, C18:1), Diacids (C9)	Degraded animal fats	4	NA
SR-HI	Hafit	Ib	A0011	body glob jar	249	3.9	FAs(C12:0-C24:0), BrFAs (C14, C15, C17), UFAs (C16:1, C18:1), Diacids (C9)	Degraded animal fats	4	NA
Mesopotamian	Hafit	I	A0012	body pot	96	2.9	FAs(C12:0-C26:0), BrFAs (C14, C15, C17), UFAs (C16:1, C18:1), Diacids (C9)	Degraded animal fats		
Mesopotamian	Hafit	I	A0014	body pot	4324	1.7	FAs(C12:0-C28:0), BrFAs (C14, C15, C17), UFAs (C16:1, C18:1), Diacids (C9)	Degraded animal fats	6	FAs(C16:0, C18:0), MAGs (M16, M18), cholesterol deriv., DAGS(trace), TAGs (trace)
Mesopotamian	Hafit	I	A0015	body pot	2256	1.5	FAs(C12:0-C26:0), BrFAs (C14, C15, C17),	Degraded animal fats + plant oils?		

							UFAs (C16:1, C18:1), Diacids (C9, C10, C11)			
SR-HI	Umm an-Nar	IIa-b	A0026	body	171	3.1	FAs(C12:0-C24:0), BrFAs (C15, C17), UFAs (C16:1, C18:1), Diacids (C9), ALK(C22-C31)	Degraded animal fats		
FR-OM	Umm an-Nar	IIc2-d	A0042	body pot	182	1.7	FAs(C12:0-C28:0), BrFAs (C14, C15, C17), UFAs (C16:1, C18:1, C22:1), Diacids (C9, C10, C11, C12), ALK(C25-C31)	Degraded animal fats + plant oils?		
SR-HI	Umm an-Nar	IIf	A0043	body	35	2.3	FAs(C12:0-C26:0), BrFAs (C15, C17), UFAs (C16:1, C18:1, C22:1), Diacids (C9), ALK(C23-C31)	Degraded animal fats		
Indus	Umm an-Nar	IIf	A0047	body BSJ	146	3.4	FAs(C12:0-C26:0), BrFAs (C14, C15, C17), UFAs (C16:1, C18:1, C22:1), Diacids (C9)	Degraded animal fats	trace	NA
Coarse	LBA	III	A0049	body	191	1.7	FAs(C12:0-C28:0), BrFAs (C14, C15, C17), UFAs (C16:1, C18:1, C22:1), Diacids (C9)	Degraded animal fats		
Fine	Wadi Suq	III	A0050	body	322	2.2	FAs(C12:0-C26:0), BrFAs (C14, C15, C17), UFAs (C16:1, C18:1, C22:1), Diacids (C9)	Degraded animal fats		
Coarse	LBA	III	A0051	body	395	1.2	FAs(C12:0-C28:0), BrFAs (C14, C15, C17),	Degraded animal fats	5	FAs(C16:0, C18:0), MAGs (M16, M18),

							UFAs (C16:1, C18:1, C22:1), Diacids (C9)			cholesterol deriv.
Indus	Umm an-Nar	IIf	A0074	body BSJ	82	2.6	FAs(C12:0-C26:0), BrFAs (C14, C15, C17), UFAs (C16:1, C18:1, C22:1), Diacids (C9)	Degraded animal fats	3	NA
SR-HI	Umm an-Nar	IIf	A0121	body	38	2.2	FAs(C12:0-C26:0), BrFAs (C14, C15, C17), UFAs (C16:1, C18:1, C22:1), Diacids (C9), ALK(C25-C31)	Degraded animal fats		
SR-HI	Umm an-Nar	IIf	A0122	body	99	1.1	FAs(C12:0-C26:0), BrFAs (C14, C15, C17), UFAs (C16:1, C18:1, C22:1), Diacids (C9), ALK(C23-C31)	Degraded animal fats		
SR-HI	Umm an-Nar	IIf	A0123	body	63	2.7	FAs(C12:0-C26:0), BrFAs (C14, C15, C17), UFAs (C16:1, C18:1, C22:1), Diacids (C9), ALK(C25-C31)	Degraded animal fats		
SR-HI	Umm an-Nar	IIf	A0126	body glob jar	74	3.3	FAs(C12:0-C26:0), BrFAs (C14, C15, C17), UFAs (C16:1, C18:1, C22:1), Diacids (C9), ALK(C25-C31)	Degraded animal fats	trace	NA
SR-HI	Umm an-Nar	IIf	A0127	body	79	3.1	FAs(C12:0-C26:0), BrFAs (C14, C15, C17), UFAs (C16:1, C18:1, C22:1),	Degraded animal fats	trace	NA

							Diacids (C9), ALK(C25-C31)	
SR-HI	Umm an-Nar	IIf	A0129	body	50	2.0	FAs(C12:0-C26:0), BrFAs (C14, C15, C17), UFAs (C16:1, C18:1, C22:1), Diacids (C9), ALK(C25-C31)	Degraded animal fats
SR-HI	Umm an-Nar	IIf	A0136	body	81	2.5	FAs(C12:0-C26:0), BrFAs (C14, C15, C17), UFAs (C16:1, C18:1, C22:1), Diacids (C9), ALK(C25-C31)	Degraded animal fats
SR-HI	Umm an-Nar	IIf	A0145	base	59	2.6	FAs(C12:0-C26:0), BrFAs (C14, C15, C17), UFAs (C16:1, C18:1, C22:1), Diacids (C9), ALK(C25-C31)	Degraded animal fats
FR-OM	Umm an-Nar	IIf	A0148	body pot	184	2.6	FAs(C12:0-C26:0), BrFAs (C14, C15, C17), UFAs (C16:1, **C18:1**, C22:1), Diacids (C9), ALK(C25-C31)	Plant oil?
FR-OM	Umm an-Nar	IIc2-d	A0559	body pot	37	2.0	FAs(C12:0-C26:0), BrFAs (C14, C15, C17), UFAs (C16:1, C18:1, C22:1), Diacids (C9)	Degraded animal fats
FR-OM	Umm an-Nar	IIe	A0574	base pot	411	1.7	FAs(C12:0-C28:0), BrFAs (C14, C15, C17), UFAs (C16:1, C18:1, C22:1), Diacids (C9,	Degraded animal fats

							C10), ALK(C25-C31)			
Mesopotamian	Hafit	Ic	A0801	base jar	777	1.0	FAs(C12:0-C26:0), BrFAs (C14, C15, C17), UFAs (C16:1, C18:1, C22:1), Diacids (C9, C10)	Degraded animal fats	2	NA
Indus	Wadi Suq	III	A0802	body BSJ	223	3.4	FAs(C12:0-C26:0), BrFAs (C14, C15, C17), UFAs (C16:1, **C18:1**, C22:1), Diacids (C9)	Plant oil	32	FAs(C10:0, C12:0, C14:0, C16:0, C18:0) UFAs (**C18:1**), cholesterol deriv., stigmasterol, DAGs(D32, D34, **D36**), TAGS(T50, T52, **T54**, T56)
Indus	Umm an-Nar	IIf	A1064	body BSJ	149	2.0	FAs(C12:0-C26:0), BrFAs (C14, C15, C17), UFAs (C16:1, **C18:1**, C22:1), Diacids (C9)	Plant oil	14	FAs(C16:0, C18:0), cholesterol deriv., stigmasterol, DAGs(D32, D34, **D36**), TAGS(T50, T52, **T54**, T56)
Coarse	LBA	III	A1110	body	1521	0.9	FAs(C12:0-C26:0), BrFAs (C14, C15, C17), UFAs (C16:1, C18:1, C22:1), Diacids (C9)	Degraded animal fats	2	NA
Coarse	LBA	III	A1112	body	104	0.9	FAs(C12:0-C26:0), BrFAs (C14, C15, C17), UFAs (C16:1, C18:1, C22:1), Diacids (C9)	Degraded animal fats	5	FAs(C16:0, C18:0) MAGs (M16, M18)

Figure 7: Lipid yields, Total Lipid Extracts (TLEs), palmitic/stearic (P/S) ratios, major compounds detected and interpretations of acidified methanol and solvent extracted samples from Hili 8. FA: Fatty acid, UFA: Unsaturated Fatty Acid, where Cn:x indicates fatty acid with n carbon atoms and x double bonds, Diacid: dicarboxylic acid, Br: branched-chain fatty acid, ALK: Alkane, cholesterol deriv: cholesterol and its derivatives, MAGs: monoacylglycerol, DAGs: diacylglycerol, TAGs: triacylglycerol. Items in bold are relatively abundant in the extract.

Provenance	**Cultural Period**	**H8 phasing**	**Sample ID**	**Shape**	**Acidified methanol extraction**				**Solvent extraction**	
					Lipid yield (µg.g-1)	**P/S ratio**	**Major compounds detected**	**Interpretation**	**TLE (µg.g-1)**	**Compounds detected**
Mesopotamian	Umm an-Nar	IIf	A0076	body bottle	61	2.4	FAs(C12:0-C26:0), BrFAs (C14, C15, C17), UFAs (C16:1, C18:1, C22:1), Diacids (C9)	Degraded animal fats	trace	NA
FR-OM	Umm an-Nar	IIf	A0377	necked pot	128	3.6	FAs(C12:0-C24:0), BrFAs (C14, C15, C17), UFAs (C16:1, **C18:1**, C22:1), Diacids (C9)	Plant oil?		
FR-OM	Umm an-Nar	IIf	A0389	necked pot	249	1.5	FAs(C12:0-C24:0), BrFAs (C14, C15, C17), UFAs (C16:1, C18:1, C22:1), Diacids (C9), ALK(C25-C31)	Degraded animal fats	3	NA
FR-OM	Umm an-Nar	IIf	A0410	necked pot	459	5.3	FAs(C12:0-C24:0), BrFAs (C14, C15, C17), UFAs (C16:1, **C18:1**, C22:1), Diacids (C9)	Plant oil	2	NA
FR-OM	Umm an-Nar	IIf	A0413	necked pot	271	2.0	FAs(C12:0-C24:0), BrFAs (C14, C15, C17), UFAs (C16:1, C18:1, C22:1), Diacids (C9)	Degraded animal fats		
SG-IR Hili	Umm an-Nar	IIf	A0439	body pot	976	0.9	FAs(C12:0-C30:0), BrFAs (C14, C15, C17), UFAs (C16:1, C18:1, C22:1), Diacids (C9)	Degraded animal fats	4	NA
SG-IR Hili	Umm an-Nar	IIf	A0532	body pot	663	1.0	FAs(C12:0-C28:0), BrFAs (C14, C15, C17), UFAs (C16:1, C18:1,	Degraded animal fats	2	NA

							C22:1), Diacids (C9)		
FG-IR Makran	Umm an-Nar	IIf	A0533	body pot	211	2.2	FAs(C12:0-C30:0), BrFAs (C14, C15, C17), UFAs (C16:1, C18:1, C22:1), Diacids (C9), ALK(C25-C31)	Degraded animal fats	

Figure 8: Lipid yields, Total Lipid Extracts (TLEs), palmitic/stearic (P/S) ratios, major compounds detected and interpretations of acidified methanol and solvent extracted samples from Hili North Tomb A. FA: Fatty acid, UFA: Unsaturated Fatty Acid, where Cn:x indicates fatty acid with n carbon atoms and x double bonds, Diacid: dicarboxylic acid, Br: branched-chain fatty acid, ALK: Alkane, cholesterol deriv: cholesterol and its derivatives, MAGs: monoacylglycerol, DAGs: diacylglycerol, TAGs: triacylglycerol. Items in bold are relatively abundant in the extract.

5. Discussion

5.1. Lipid preservation and uses of pottery

Overall, the preservation of lipids in pottery from Hili 8 appears to be variable. The Mesopotamian vessels from Phase 1 demonstrate the highest lipid yields from the studied assemblage (range: 95-5265 $\mu g.g^{-1}$; mean = 2.7 $mg.g^{-1}$, median = 3.1 $mg.g^{-1}$). High lipid yields were also observed in other types of pottery, including Sandy Red Ware from Hili 8 (SR-HI), some examples of Fine Red Omani Ware from both Hili 8 and Hili North Tomb A, semi-fine Wadi Suq pottery and Coarse LBA pottery from Hili 8.

The preservation of lipids in pottery is affected by a number of factors. These include variations in temperature and precipitation (Pollard et al. 2007), specific conditions of the depositional environment (Eglinton et al. 1991), as well as the manufacturing characteristics of the pottery itself, such as the size and shape of ceramic pores, firing conditions and surface treatments, which will impact vessel porosity and the degree of absorption of lipids (Evershed et al. 1995, Raven et al. 1997, Debono Spiteri 2012, Drieu et al. 2019). For example, observations from studies on lipid yields and vessel porosity have demonstrated that the presence of 'micro pores', generally > 1 μm, limits the degradation of lipids by micro-organisms (Davis et al. 1990, Drieu et al. 2019). Finally, the use-history of a vessel will affect not only the type of lipids obtained, but also lipid recovery. The frequent use of a vessel for fatty or oily products may also lead to a higher chance of good lipid recovery.

In the case of Hili 8, if one assumes that the depositional environment is generally similar across the assemblage, it is likely that either vessel manufacture or vessel use are the primary influencing factors for variable lipid preservation. The Mesopotamian, Sandy Red Ware (SR-HI), as well as the semi-fine Wadi Suq and Coarse LBA pottery fabrics are microporous (Méry 2000: 102, 152) and the semi-fine Wadi Suq ware contains vegetal temper imprints (Ibid: 254). The Fine Red Omani Ware (FR-OM) is not microporous, but contains fine desiccation slots (Ibid:106). Thus, it is possible the high lipid yields of the Mesopotamian, Sandy Red Ware (SR-HI) and Wadi Suq ceramics may be linked to the fabric and porosity of the vessels. It is also possible that very fatty products, such as animal fats, were contained in Mesopotamian, Sandy Red Ware and Wadi Suq pottery, contributing to increased lipid yields.

5.2. Organic products used or processed in vessels

The results suggest that vessels were linked with the use, store and/or transport of products of both animal and plant origin.

5.2.1. Animal products

Most of the studied vessels indicate that they contained animal products (Fig 5a). These include all the Mesopotamian vessels dating to the Hafit period, most of the Sandy Red vessels (SR-HI) from Hili 8 and Hili North Tomb A dating to the Umm an-Nar period, as well as the Fine Wadi Suq and Coarse LBA vessels from Hili 8, which also corresponds to high lipid yields from these vessels. The presence of more than one positional $C_{18:1}$ isomer and branched-chain fatty acids (known to be formed in the gut by bacterial synthesis) in the extracts may be assigned to ruminant animal fats (Christie 1981, Dudd et al. 1999), which corresponds well with the available zooarchaeological evidence that is dominated by ruminant animals such as cattle and ovicaprine species in the faunal assemblage at Hili 8. The presence of degraded animal fats in Mesopotamian vessels may also come from imported meat or milk products. At present, no biomarkers of aquatic products have been detected, however further analyses are ongoing. A selection of these vessels will be analysed via compound-specific isotopic analysis to distinguish between the different types of animal products in the vessels, namely terrestrial and non-terrestrial and ruminant dairy and ruminant carcass fats.

5.2.2. Plant oils

Out of the studied assemblage, two Fine Red Omani necked pots from Hili North Tomb A (A0377 and A0410) and one from Hili 8 (A0148) have high abundances of unsaturated fatty acids (specifically $C_{18:1}$), and two Indus BSJs from Hili 8 (A0802 and A1064) have a high percentage of unsaturated fatty acids and unsaturated TAGs, providing the first clear evidence of the use of plant oils in the region, both in the Umm an-Nar and Wadi Suq periods. The results also provide the first evidence of the use of Indus Black-Slipped Jars for the storage or transportation for plant oils, however, one cannot exclude that they had other uses.

The TAG profiles of the lipid extracts of the two Indus BSJs found at Hili 8 closely match TAG profiles of modern seed oils such as fresh sesame oil, linseed oil and olive oil (Copley et al., 2005). Interestingly, the archaeological TAG profiles do not appear to match those of modern or archaeological date palm fruit lipids (Copley et al. 2001), which was presumably an important culinary resource in SE Arabia in the Bronze Age (Tengberg 2003). Sesame and/or linseed oil could be potential candidates of the archaeological plant oil/s as they fit with available evidence from Mesopotamia, SE Iran/SW Pakistan and the Indus Valley. Sesame (*Sesamum indicum*) was domesticated in India and presumably dispersed westwards from South Asia in the late third millennium BC (Bedigian & Harlan 1989, Tengberg 1999, Fuller 2003, Dossa et al., 2016, Garcia-Granero et al., 2016). Sesame seeds have been reported from Miri Qalat in Baluchistan (SW Pakistan) between 2500-2000 BC (Tengberg 1999), and from Abu Salabikh in lowland Iraq in the Early Dynastic period (third millennium) (Charles 1989). As previously mentioned, cuneiform texts from 2100 BC also mention the transport of sesame oil (Potts 1990:145), however the precise translation is disputed, and this could refer to another oilseed (Postgate 1992: 171). Conversely, flax or linseed (*Linum usitatissimum*) was one of the 'founder crops' in the Neolithic Levant (Zohary 1996, Boivin and Fuller 2009), and its development as a large-scale textile fibre crop in the late fifth and fourth millennium BC is associated with the emergence of intensive Mesopotamian agriculture and urbanism (McCorriston 1997). Flax is present throughout the sequence at Miri Qalat and the neighbouring site of Shahi Tump in Baluchistan (Tengberg 1999). It was also a major fibre crop in the Indus Civilisation (Fuller 2008, Fuller and Madella 2001: 337–338). No *Linum* macroremains have been reported from the Arabian Peninsula (Boivin and Fuller 2009), however, remains of linen textiles are known from the third millennium BC from Tell Abraq (Reade and Potts 1993).

In cases of excellent preservation, TAG profiles can enable the identification of the source product (Dudd et al., 1999; Mirabaud et al., 2007). However, assigning specific products based on TAG profiles is undermined by preferential loss of lower molecular weight components and the mixing of resources (Dudd & Evershed, 1998). Thus in this scenario, the analysis of additional reference oils for comparison, and factoring in the possibility of mixtures of oils is necessary before being able to propose (a) potential source(s) for the archaeological plant oils, and whether it/they were imported or available locally.

5.2.3. Mixtures of plant and animal products

Some other vessels (Hafit-period Mesopotamian vessel (A0015) and two Umm an-Nar Fine Red Omani pots from Hili 8) demonstrate tentative evidence of plant oils, based on the presence of α,ω-dicarboxylic acids and high abundances of unsaturated fatty acids, specifically $C_{18:1}$, but they also contain compounds that are characteristic of degraded animal fats, such as saturated fatty acids and branched-chain fatty acids (Dudd et al. 1999). This perhaps indicates the mixing of animal and plant products in vessels, or multiple uses of vessels.

5.3. Potential of organic residue analysis in SE Arabia

The preservation of intact triacylglycerols in some vessels and the high concentrations of lipids found at vessels from Hili 8 and Hili North Tomb A relative to other arid regions of the world, especially South Asia (Suryanarayan et al., 2021), suggest that the study of lipid residues in pottery has potential in SE Arabia, and deserves further study. It is important to test the preservation of lipids at various sites and to analyse a large number of different types of vessels before being able to make any conclusive remarks about the function or specialised uses of specific imported or local vessels, however, the initial results are promising.

6. Conclusions and future perspectives

Lipid residue analysis of vessels from Hili 8 and Hili North Tomb A suggest the presence of animal products in most of the vessels, and plant oils in a small minority of vessels, including Indus Black-Slipped Jars. Future analyses will include compound-specific isotopic analysis, which will help distinguish between the presence of ruminant carcass and dairy fats in the vessels at Hili. Exploratory lipidomics analysis (in case of good survival of TAGs) may be able to determine the potential source of plant oils. Although aquatic products have not yet been detected in the vessels, isotopic analyses may aid in detecting the presence of marine vs. terrestrial fats. These preliminary results demonstrate that the use of lipid residue analysis can facilitate exciting new ways to study subsistence practices and the use of pottery in ancient SE Arabia.

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