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ABSTRACT

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This study applies chemical analyses by GC-MS (gas chromatography-mass spectrometry) of absorbed residue in vessels and of vessel fill contents in order to investigate the use and function of pre-Aksumite pottery from the excavations of the German Archaeological Institute in Yeha and Melazo, and to draw conclusions about processing of raw materials, consumption, eating habits and culinary practises, in the ancient Ethiopian highlands. The results of the analyses show fermentation markers of fruits and markers for plant and animal fats. A notable feature is the potential identification of seed oils as content of vessels from the palace in Yeha with probably varied usage. In order to identify the contents more precisely, targeted research is necessary, which requires better knowledge of ancient resources available in the region. The current results provide initial clues for further investigations.

KEYWORDS

Horn of Africa, Yeha, pottery, organic residue analysis, food consumption

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Introduction

1 The usage of resources, as well as the eating habits and culinary practises of the people of northern Ethiopia during the 1st millennium BCE, the so-called pre- and proto-Aksumite periods, are still understudied. This includes the utilization of wild and cultivated plants, of wild and farmed animals, and their various applications, such as in cooking, combusting and medicinal practices. Alongside animal bones and botanical remains, the vessels and utensils necessary for the storage of raw materials (storage containers), for food and other preparations (vessels and utensils for grinding, mixing, cooking, baking, frying, etc.) and for the actual utilisation (table ware and drinking vessels, incense burners) are closely associated with these aspects. One facet of this line of research is presented here, namely the organic residue analysis on ceramic vessels and vessel contents from current excavations by the German Archaeological Institute in the Tigray region in northern Ethiopia (Fig. 1). With the help of such analyses, ancient dietary habits, foodways and subsistence strategies could be explored providing understanding of past human diets and their cultural and economic contexts (e.g., Dunne – Grillo – Casanova et al. 2019). This multidisciplinary approach highlights the importance of combining archaeological research and scientific content analysis to answer complex questions about ancient pastoral lifestyles in the Ethiopian highlands.

Geographical and historical background

2 Since 2009 the Sanaa Branch of the Orient Department of the German Archaeological Institute in cooperation with the Friedrich Schiller University Jena,

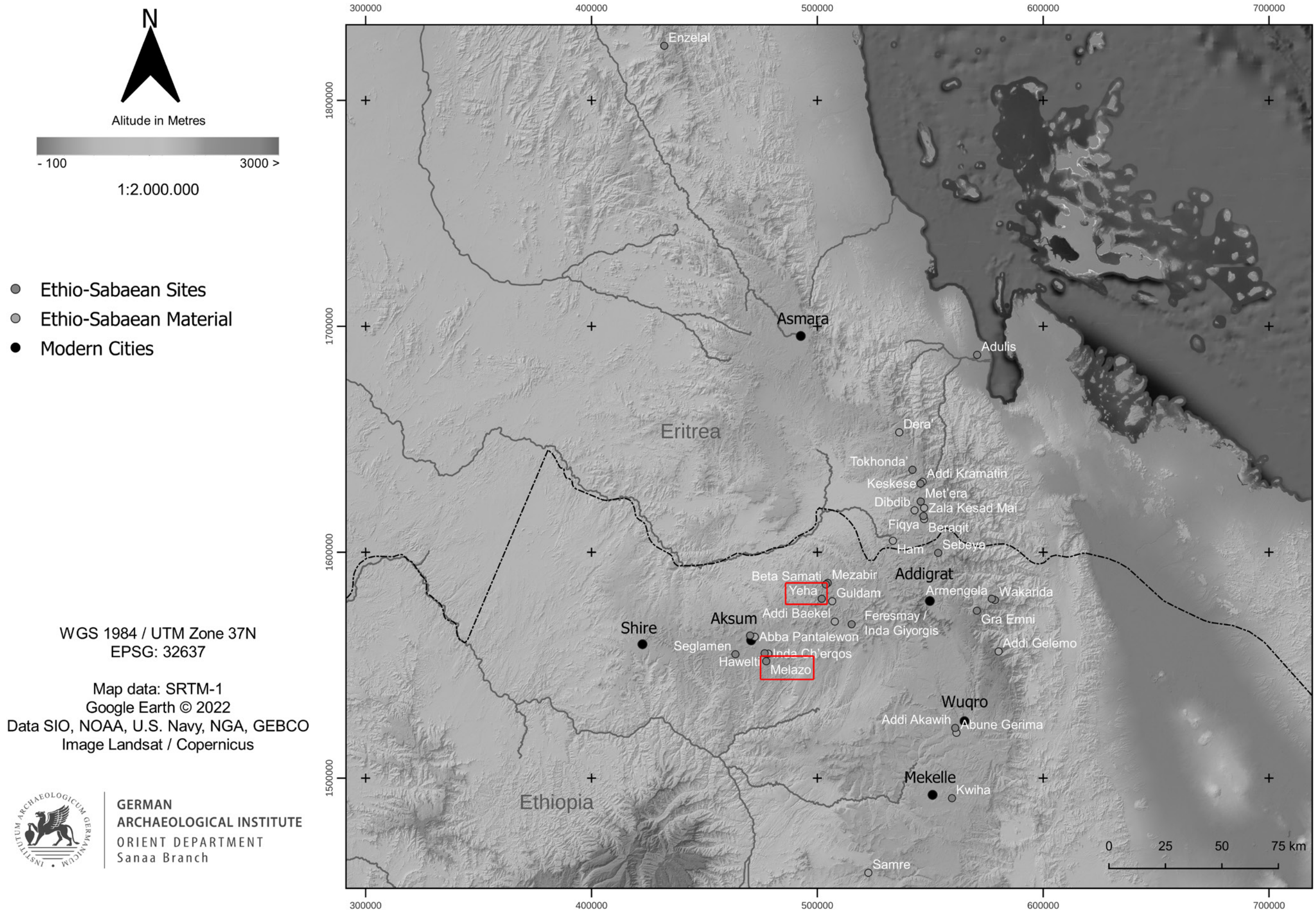

Fig. 1: Map of the area with the location of the sample sites.

the Ethiopian Heritage Authority (EHA) and the Tigrai Culture and Tourism Bureau (TCTB) has conducted a research project in Yeha, Hawelti and surroundings[1]. The archaeological, architectural, historical, epigraphic and ethno-historical investigations focus on the rise and development of the Ethio-Sabaean culture during the first half of the 1st millennium BCE, which came into being through the migration of South Arabian people to the Ethiopian Highlands and a subsequent acculturation process with the indigenous population. This culture ceased around the mid-1st millennium BCE followed by a gap in knowledge until the turn of millennium, when the Aksumite realm emerged. The previous local culture(s) especially of the 2nd millennium BCE is examined also within the framework of the project to note the changes and to view foreign influences. Additionally, lines of tradition between the Ethio-Sabaean and the successive proto-Aksumite and Aksumite cultures are also discussed.

3 Yeha and the Melazo plateau sites are situated in the highlands of Tigray in northern Ethiopia[2] (Fig. 1). In Yeha, the centre of the Ethio-Sabaean entity in the first half of the 1st millennium BCE, monumental edifices, such as a temple made of limestone ashlars, a palatial timber framework building and a necropolis of rock-cut tombs are under examination. In addition, trenches within the ancient settlement area have revealed further representative buildings from this period as well as architectural structures and domestic dwellings from Aksumite times. In Melazo, a site close to Aksum,

1 Cf. as introduction into the project Japp – Gerlach – Hitgen et al. 2011; Gerlach 2013; Gerlach 2017; Gerlach – Japp – Schnelle et al. 2022; Nebes 2014; Schnelle 2021. We would like to thank the EHA and TCTB for permission to extract samples and to export them to Germany.

2 E.g., footnote 1. Excavations and surveys started in 2009 with two campaigns a year. These had to be stopped in the autumn of 2020 due to the pandemic, but above all due to the war in the Tigray and were only recently resumed. Archaeo-zoological and archaeo-botanical investigations were also interrupted particularly in the area of the residential settlement of Yeha.

the capital of the Aksumite realm, a large representative timber framework building of the Ethio-Sabaean culture has been uncovered.

4 The pottery repertoire of the pre-Aksumite period, specifically the Ethio-Sabaean repertoire, is still the subject of debate[3], while that of the 2nd millennium BCE is scarcely established. Better knowledge exists for the Aksumite period (cf. Wilding 1989; Phillipson 2000). The function of the vessels can often only be roughly determined on the basis of size, technical details or surface treatment. However, the specific use of the different pottery forms is not conclusively defined and neither are the commodities stored or prepared in them.

Organic residue analysis

5 The analysis of the organic residues preserved in archaeological ceramic material aims at relating the vessels to their use and function in the past and, in doing so, shedding new light on direct consumption and diets (Mottram – Dudd – Lawrence et al. 1999; Roffet-Salque – Dunne – Altoft et al. 2017). This is done by means of the distribution of molecular components (biomarkers) that can be correlated with the ancient organic mixtures absorbed into the pores of the ceramic container while being used (Evershed 2008). Most organic materials are subject to leaching as well as microbial and chemical degradation. Thus, useful biomarkers are only those that are sufficiently robust to survive long-term deposition, such as fatty acids, terpenoids, sterols and sometimes the decay products of less stable parent compounds (Nigra – Faull – Barnard 2015). Therefore, the assignment of a specific source or constituent of a residue based on the presence of a biomarker component or mixture of components demands a high degree of caution (Whelton – Hammann – Cramp et al. 2021). The biomarker approach requires analytical techniques that reach molecular level resolution because of the complexity and low abundance in the sample (Evershed 2008). Several techniques, such as NMR (nuclear magnetic resonance), IR (infrared) spectroscopy and HPLC (high performance liquid chromatography) have been used to obtain a chemical fingerprint of organic residues. However, gas chromatography combined with mass spectrometry (GC-MS) is the most common method to provide structural information with high sensibility for organic residues characterisation (Mottram – Dudd – Lawrence et al. 1999). Due to their low water solubility, lipids are more easily found than other water-soluble biomarkers or compounds which are more susceptible to microbial degradation (Regert 2011). The presence of saturated and unsaturated fatty acid can be used as a starting point for the identification of complex mixtures preserved in archaeological materials.

State of the art of food production and consumption at the northern Horn of Africa

6 Only a little is known about the food production and consumption traditions at the northern Horn of Africa in ancient times. Organic residue analyses of ceramic material in that region are in their beginnings[4]. Archaeological evidence for the cultivation of plants is known from the early 3rd millennium BP onwards[5]. Certain innovative techniques in agriculture might have been introduced by Sabaeans during

3 Köster 2021. For the results of the XRF and MGR analyses conducted on pottery material from Yeha and Hawelti/Melazo cf. Japp – Köster 2020.

4 See recently on GC-MS and stable isotopes analyses of prehistoric pottery from eastern Africa Grillo – Dunne – Marshall et al. 2020.

5 Macrobotanical evidence was identified in pre-Aksumite sites in Ethiopia and Eritrea, e.g., Bard – Fattovich – Manzo et al. 1997; D'Andrea – Schmidt – Curtis 2008; Curtis 2013. See also Seegeler 1983: 921 on Ethiopian oil plants.

the early 1st millennium BCE[6]. Domesticated plants that appeared during this time are finger millet, emmer, bread wheat and barley in addition to lentils and linseed, and presumably tef already in the pre-Aksumite period[7]. From linseed it could have been possible to extract oil. A plant-based diet is indicated by stable isotope analysis of an individual burial dating to the Aksumite period[8].

7 Moreover, modern food consumption in Tigray is based on plants with only occasionally meat consumption mostly related to religious feasts and social events. Perhaps a similar behaviour can be hypothesised also for pre-Aksumite times (D'Andrea – Richards – Pavlish et al. 2011). Meat consumption could mainly include cattle, sheep, goat, poultry and wild fauna (Tefera 2004; D'Andrea – Richards – Pavlish et al. 2011: tab. 2).

8 The excavations in the deep trench in Yeha revealed numerous animal bones in its different layers. So far cattle and sheep or goat bones can be identified within the strata of the Ethio-Sabaean phase of Yeha. These species seem to be equally part of the foodways of the inhabitants of Yeha[9].

9 The archaeobotanical investigations on flotation samples of the pre-Aksumite layers in the deep trench in Yeha yielded common barley (*Hordeum vulgare*), lentil (*Lens culinaris*) and non-specific cereals[10]. In addition, it was possible to observe oat (*Avena spec.*), unidentifiable seeds from the grass family (*Poaceae*), bristlegrass (*Setaria spec.*) and darnel (*Lolium spec.*). Of great interest is the occurrence of tef (*Eragrostis tef*) in several of the corresponding layers. Thus, there is reason to believe that tef was already present in the first half of the 1st millennium BCE and was probably harvested and consumed during that time. Even the Near Eastern crop hulled barley is documented for this early time, which becomes the most common cereal in the course of the first half of the 1st millennium BCE in the northern Horn of Africa (Curtis 2013; D'Andrea – Schmidt – Curtis 2008: 212). Some of the species mentioned may even appear at Yeha as early as the end of the 2nd millennium BCE according to the stratigraphy, but this observation has yet to be verified. The results of archaeobotanical investigations confirm previous findings in the region. However, they do not reflect the full range of plants to date identified from sites of the early 1st millennium BCE. This is certainly due to the relatively low yield of botanical samples from these layers at Yeha. In addition, it can be assumed that leafy vegetables, roots, tubers etc. were cultivated and eaten. These plant parts are never or only rarely found charred and are therefore seldom evident in the archaeological record.

6 Gerlach 2012: 218. The introduction of bronze and metal working in particular facilitated agriculture through the use of bronze implements such as sickles.

7 Arthur – Curtis – Arthur et al. 2019 with further references. Archaeobotanical analyses have been conducted on the material excavated in Aksum. For the earliest phases (8th to 5th centuries BCE in D site Kidane Mehret) hulled barley, emmer, linseed, and pea and less certain tef, noog and lentil are attested, cf. Phillipson 2000: 469. Wheat and barley were found in the Aksumite settlement of Ona Nagast, cf. Bard – Fattovich – Manzo et al. 1997. Further evidence for crops such as linseed, lentil, barley and emmer are proven at Mezber. Tef appears at Mezber in the late pre-Aksumite period (400 BCE – 1 BCE/CE), cf. D'Andrea – Welton – Manzo et al. 2023: 49–50. 53. Linseed grows also above 2000 m altitude: Seegeler 1983: fig. 1.

8 D'Andrea – Richards – Pavlish et al. 2011. Mainly Near Eastern plants and in a lesser quantity African plants had been consumed. The same trend of dominating Near Eastern crops seems to be true for the economy of the early 1st millennium BCE in the northern Horn of Africa: D'Andrea – Schmidt – Curtis 2008: 207.

9 The archaeozoological investigations are conducted by J. Peters (Ludwig-Maximilian-University Munich).

10 The archaeobotanical research is conducted by R. Neef and V. Podsiadlowski (DAI, Berlin). We thank them for sharing their information.

Material and Method

Selection of ceramic samples

10 Closed vessels were particularly selected for the start of the investigations with regard to nutrition and diets as they were probably used for the storage of food or resources as well as for the preparation and presentation of prepared food[11]. The majority of the vessels were chosen from two representative buildings, which had been destroyed by a conflagration around the middle of the 1st millennium BCE and had not been reused. The expectation was that despite the long period of time, remains of the former contents would still be present. However, it has to be accepted that the fire possibly led to changes in the chemical composition of organic residues there.

Archaeological context of the selected samples

11 The Grat Beʿal Gibri in Yeha (area B) is a monumental structure of 65 x 65 m erected on a high stone podium with several stories of timber framework on top[12]. The interior is organised in two long corridors and several mostly interconnected rooms of different size. Access is enabled by a staircase with a typical South Arabian six-pillar-propylon at its upper end. From a comparison with similar buildings in South Arabia, as well as the building's internal structure, external appearance and associated finds, a function is indicated as an administrative centre of the Ethio-Sabaean entity, combining a storage area for traded materials, space for administrative tasks and palatial architecture. Based on the C14 analyses, the earliest phases of the building can be dated around 800 BCE, while it was probably destroyed by conflagration in the mid-first millennium BCE (Gerlach 2017: 273). Five of the sampled pieces were uncovered in a room in the rear part of the building aside a walled door. It can be assumed that these vessels had been originally placed in a wooden cupboard that was destroyed during the extensive fire, so the vessels crashed down onto the floor (Gerlach 2021: 151). Therefore, they belong to the latest usage phase of the building before its destruction. The complex comprised two larger vessels with out-curving rims and seven bulbous pots with out-turning rims sometimes decorated with incised lines (sample numbers X2, X3 and its content X11, X9, X10, X15, see attached catalogue of the samples for further information) as well as one bell- and nine convex-shaped lids. Given the thin walls and the two handles of the pots, it seems plausible that these were used for the presentation and serving of prepared food during meals. From the same room came a jar (X17). To the east of this room another chamber was uncovered in which a kind of oven was found, suggesting that this area might have been used as a kitchen at least during the last occupation phase of the Grat Beʿal Gibri. In its destruction layer a typical storage jar came to light, whose content soil was sampled (X12). One vessel, a bag-shaped jug with one handle (X1), was found scattered on the beaten soil floor of the southern corridor of the building buried in the destruction layer. This jug also belongs to the last occupation phase. Whether it was originally stored in this storey or fell from an upper storey cannot be determined. Also located on the floor of the corridor was a small bag-shaped vessel (X14). In a central room of the building the destruction layer of the Ethio-Sabaean phase contained a small beaker-like vessel, whose residues were sampled (X13).

12 A stratigraphic sondage was conducted in the centre of Yeha to obtain information on the chronological sequence of the settlement's history (Japp 2019).

11 See the attached catalogue for the detailed description of the vessels.

12 See regarding architecture and interpretation Gerlach 2013: 264–266; Gerlach 2017: 371–376; Schnelle 2014; Schnelle 2019; Schnelle 2021.

This deep trench extended from the modern surface down to the bedrock. The oldest stratum dates to the late 2nd millennium BCE, followed by layers of the first half of the 1st millennium BCE, the early, late and post-Aksumite periods until recent times. One sample of a flat-bottomed bowl stems from the Aksumite period (X4).

13 In the south of the modern village, excavations uncovered remains of a domestic quarter that had probably been agriculturally oriented (Japp 2024). It consisted of rubble stone walls, which enclose rectangular spaces in which remains of the inventory were still extant. On the basis of C14 analyses as well as the pottery finds, these structures can be dated to the early Aksumite period. A typical jug fragment was chosen from this location for analysis (X16).

14 The four samples from Melazo derive from the excavations in an Ethio-Sabaean timber framework building located not far from the church Enda Cherqos. They were found in the destruction stratum presumably belonging to the last occupation phase of the building, dated to the late first half of the 1st millennium BCE. One pot or brazier (X5) was selected due to the particular white cover of its interior surface. This cover is not an isolated case but occurs frequently within the find material of Melazo. The second sample stems from a chalice (X6), which might have been used as a drinking vessel; however, due to the unusual shape it could also have been a kind of cultic device. The third piece (X7) belongs to a vessel with a typical decoration of a combination of horizontal comb lines and dots which appear often in pre-Aksumite contexts. The fourth sample (X8) displays a footed jar, locally produced and commonly used during that period.

GC-MS analysis

15 A set of seventeen archaeological samples (X1 to X17) were selected for organic residue analysis. Fourteen samples are fragments of vessels and three samples (X11–13) are the loose content found in vessels. The fragments were cleaned with a drill in order to avoid external contamination, and then crushed with a mortar to a fine powder. The loose contents were available as powder and were crushed and refined using a mortar. Finally, the prepared samples were analysed by GC-MS in order to extract lipids and other biomarkers.

16 The above-mentioned contexts should offer optimal conditions for the preservation of organic residues: dry climate and soil and freshly excavated material from closed contexts. However, fire seems to have destroyed wooden furniture where some vessels have been probably stored in (X2, X3, X9, X10, X15), a fact that may in general lead to taphonomic alteration and degradation of organic materials (Garnier 2015). The same is true for other samples that came from the destruction layers of the representative building in Melazo (X5–8), which was also devastated by fire.

17 Two extraction protocols have been applied in order to extract lipids according to Mottram – Dudd – Lawrence et al. 1999 and Pecci – Cau Ontiveros – Garnier 2013 (a), and other small organic acids according to Pecci – Giorgi – Salvini et al. 2013 (b).

- a. Samples, weighing 2 g, are extracted in 10 ml chloroform/methanol (2:1 v/v) for 2×15 min in an ultrasonic bath. The samples are then centrifuged for 10 min and the supernatant is evaporated to dryness under a steady stream of nitrogen. The samples are saponified with 2 ml sodium hydroxide solution (2M in MeOH) for 1 h at 70° C. After cooling, each sample is acidified with 15 drops of concentrated hydrochloric acid and the pH value is checked (pH 1). It is then extracted twice with 3 ml of chloroform. Then the solvent of the organic phase is removed under a constant stream of nitrogen and the sample is transferred to a sample vial with two times 50 µl of chloroform. The sample is then evaporated off and is derivatised with 25 µl of BSTFA at 70 °C for 1 hour. After the addition of 75 µl n-hexane and 5 µl internal standard (dotriacontane in hexane), the samples are analysed by GC-MS.

- b. A sample of 500 mg is extracted in 3 ml potassium hydroxide solution (1M in H2O) for 90 min at 70 °C. The samples are then centrifuged for 10 min and the supernatant is acidified with 15 drops of concentrated hydrochloric acid, and the pH value (pH 1) is checked. Then the samples are shaken intensively with 3 ml ethyl acetate for 2 min and centrifuged for 10 min. This step is repeated 3 times in total. The supernatant (organic phase) is concentrated under a constant stream of nitrogen and transferred to a sample vial with two 50 µl portions of ethyl acetate. The solvent is then evaporated off and the sample is derivatised with 25 µl of BSTFA at 70 °C. for 1 hour. After the addition of 75 µl n-hexane and 5 µl internal standard (dotriacontane in hexane), the samples are analysed by GC-MS.

18 Because of the very limited number of samples, no previous determination of the rates of samples with sufficient amounts of extractable lipids (recovery rates) was performed[13]. Instead, an additional extraction protocol (b) focusing on more polar markers was used to increase the certainty of the analysis. Overall, taking the differing extraction efficiencies of the two protocols for the different compound classes into account, the results coincide very well.

19 All samples were analysed using Agilent 7820A GC system and Agilent 5977 MSD, equipped with HP5-MS capillary column and EI as ion source. Initial oven temperature was set to 50 °C with a temperature ramp of 5 °C/min to 320 °C and hold time of 10 min. For this method a split ratio of 1:1 (for samples X1–11) or 1:10 (samples X12–17) has been used.

Results

20 The results of the GC-MS analysis are summarised in Fig. 2 and by way of example in the chromatograms (Fig. 3, Fig. 4, Fig. 5). They can be discussed as follows according to a preliminary grouping of detected compounds, i.e., the biomarkers profile and fatty acids distribution in the analysed samples (see also Fig. 6).

21 The presence of the major saturated fatty acid, i.e., palmitic (C16:0, hexadecanoic acid) and stearic (C18:0, octadecanoic acid) acids, is ubiquitous in the samples. They are very common compounds of both animal and plant origin (Heron – Evershed 1993; Regert 2011). In ten samples, the presence of lauric acid (C12:0, dodecanoic acid) may suggest a plant origin of the residues (Rosiak – Kałużna-Czaplińska – Gątarek 2020). Valeric acid (C5:0, pentanoic acid), a short chain fatty acid, is present in sample X2 and may also be related to plants (Kałużna-Czaplińska – Rosiak – Kwapińska et al. 2016). The ubiquitous pelargonic acid (C9:0, nonanoic acid) indicates a possible plant (oil) origin of the compounds[14]. In general, low lipid concentrations in the samples may be indicative of plant processing (Grillo – Dunne – Marshall et al. 2020). On the other hand, myristic acid (C14:0, tetradecanoic acid), present in the samples X1, X4, X6, X7, X8 and X11 (=content of X3), mostly suggests an animal source of fats, i.e., degradation of animal products (dairy products), but can be

13 A lipid concentration above 5 µg g^{-1}. is considered the limit of reliable interpretation according to Evershed – Dudd – Charters et al. 1999 and Evershed 2008. Recovery rates and concentrations in Grillo – Dunne – Marshall et al. 2020 are based on 125 ceramic sherds and in Dunne – Höhn – Neumann et al. 2022 on 458 samples. See also the results section in Lundy – Drieu – Meo et al. 2021 and Lundy – Drieu – Orecchioni et al. 2023 respectively based on 134 and 114 samples. A more fundamental study is presented in Correa-Ascencio – Evershed 2014, comparing the amounts of lipids extracted by different approaches and possible reasons ike, e.g., microencapsulation, based on 33 samples.

14 See Irto – Micalizzi – Bretti et al. 2022 for a general overview.

Samples Nr.	X1	X2	X3	X4	X5	X6	X7	X8
compound, confirmed by measurement of standard (after derivatisation with BSTFA, with GC-MS)	α-pinene	α-pinene	α-pinene	α-pinene	α-pinene	α-pinene	α-pinene	α-pinene
	lactic acid	lactic acid	lactic acid	lactic acid	lactic acid	lactic acid (traces)	lactic acid	valeric acid (traces)
	glycolic acid	glycolic acid	caproic acid	caproic acid (traces)	caproic acid	caproic acid	glycolic acid	lactic acid
	levulinic acid	levulinic acid	levulinic acid	glycolic acid	oxalic acid	glycolic acid	levulinic acid	glycolic acid
	oxalic acid	benzoic acid	benzoic acid	levulinic acid	benzoic acid	benzoic acid	oxalic acid	levulinic acid
	benzoic acid	caprylic acid	caprylic acid	oxalic acid	caprylic acid	malonic acid	benzoic acid	oxalic acid
	malonic acid	succinic acid	succinic acid	benzoic acid	succinic acid	caprylic acid	malonic acid	malonic acid
	caprylic acid	pelargonic acid	pelargonic acid	caprylic acid	pelargonic acid	succinic acid	caprylic acid	benzoic acid
	succinic acid	lauric acid	capric acid	succinic acid	lauric acid	pelargonic acid	succinic acid	caprylic acid
	pelargonic acid	myristic acid (traces)	lauric acid	pelargonic acid	myristic acid (traces)	capric acid	pelargonic acid	succinic acid
	glutaric acid	palmitic acid	myristic acid (traces)	2-Hydroxy-butanoic acid	palmitic acid	lauric acid	capric acid	pelargonic acid
	capric acid	stearic acid	palmitic acid	capric acid	stearic acid	myristic acid (traces)	4-Hydroxy-benzoic acid	capric acid
	adipic acid		margaric acid	4-Hydroxy-benzoic acid		palmitic acid	lauric acid	lauric acid
	4-Hydroxy-benzoic acid		stearic acid	lauric acid		stearic acid	myristic acid	myristic acid (traces)
	azelaic acid		cinnamic acid (traces)	myristic acid			palmitic acid	palmitic acid
	lauric acid			palmitic acid			stearic acid	stearic acid
	myristic acid			stearic acid				
	palmitic acid			cinnamic acid (traces)				
	stearic acid							
Probable source of compounds	**fruits?/ animal fat?**	**plant (oil)?/ animal fat?**	**animal fat/ plant (oil)/ resins?**	**animal fat/ plant (oil)/ resins?**	**animal fat?**	**fruits/ animal fat?**	**fruits/ animal fat?**	**fruits/ animal fat?**

Fig. 2: List of the detected compounds and their probable source.

found also in plant seed oils (Evershed – Mottram – Dudd 1997). Margaric acid (C17:0) is present in X3 and may also indicate an origin related to bacterial, milk or ruminant fats (Irto – Micalizzi – Bretti et al. 2022). No cholesterol has been detected in the samples. However, degradation of cholesterol in archaeological samples has been previously demonstrated (Hammann – Whittle – Cramp et al. 2018) and this may account for the absence in our samples.

22 In X1 small carboxylic acids (levulinic, oxalic, succinic, glutaric acids) and dicarboxylic acids (adipic, azelaic) indicating fermentation/degradation by-products together with major long chain saturated fatty acids (myristic, palmitic, stearic acids) have been detected.

23 In sample X9 the unsaturated fatty acid oleic acid (C18:1) is present, probably implying a plant origin of the compounds. Nevertheless, unsaturated fatty acids like oleic, linolic, linolenic are generally not discernible and might have decomposed (the presence of dicarboxylic acids support this possibility). Unfortunately, other possible markers for linseed oil like, e.g., coumaric acid or ferulic acid were not detected. The presence of small acidic components, i.e., levulinic, oxalic, succinic, malonic, glycolic and benzoic acids, may suggest the processing/storage of fruits. While discrete fermentation markers

X9	X10	X11	X12	X13	X14	X15	X16	X17
α-pinene	α-pinene	α-pinene	α-pinene	α-pinene	α-pinene	α-pinene	α-pinene	α-pinene
lactic acid (traces)	caproic acid	lactic acid	lactic acid	lactic acid	lactic acid	lactic acid	levulinic acid	lactic acid
caproic acid	glycolic acid	caproic acid (traces)	glycolic acid	glycolic acid	levulinic acid	glycolic acid (traces)	oxalic acid (traces)	glycolic acid
glycolic acid	levulinic acid	glycolic acid	levulinic acid	levulinic acid	oxalic acid (traces)	levulinic acid	benzoic acid	levulinic acid
benzoic acid	benzoic acid	levulinic acid	oxalic acid (traces)	oxalic acid (traces)	benzoic acid	oxalic acid (traces)	succinic acid	oxalic acid (traces)
caprylic acid	succinic acid	benzoic acid	benzoic acid	benzoic acid	caprylic acid	benzoic acid	glutaric acid	benzoic acid
succinic acid	pelargonic acid	caprylic acid	caprylic acid	caprylic acid	succinic acid	caprylic acid	adipic acid	succinic acid
pelargonic acid	capric acid	succinic acid	succinic acid	succinic acid	2-Hydroxy-butanoic acid	succinic acid	salicylic acid (traces)	salicylic acid
capric acid	lauric acid	pelargonic acid	2-Hydroxy-butanoic acid	glutaric acid	capric acid	2-Hydroxy-butanoic acid	myristic acid	lauric acid
cinnamic acid (traces)	myristic acid	capric acid	capric acid	2-Hydroxy-butanoic acid	lauric acid	lauric acid	palmitic acid	myristic acid
lauric acid	palmitic acid	adipic acid	adipic acid	capric acid	myristic acid	myristic acid	stearic acid	palmitic acid
palmitic acid	stearic acid	salicylic acid (traces)	salicylic acid	adipic acid	palmitic acid	palmitic acid	arachidic acid	stearic acid
oleic acid		4-Hydroxy-benzoic acid	4-Hydroxy-benzoic acid	salicylic acid	stearic acid	stearic acid		arachidic acid
stearic acid		lauric acid	azelaic acid	4-Hydroxy-benzoic acid				behenic acid
arachidic acid (traces)		myristic acid (traces)	myristic acid	lauric acid				lignoceric acid
		palmitic acid	palmitic acid	azelaic acid				
		stearic acid	dodecanedioic acid	myristic acid				
			stearic acid	palmitic acid				
				dodecanedioic acid				
				stearic acid				
plant/ resins?	animal fat?	animal fat?	plant oil?/ animal fat?	plant oil?/ animal fat?	animal fat?/ plant (oil)?	animal fat?/ plant (oil)?	animal fat?/plant (oil)?	plant seed oil?, i.e., moringa seed oil?

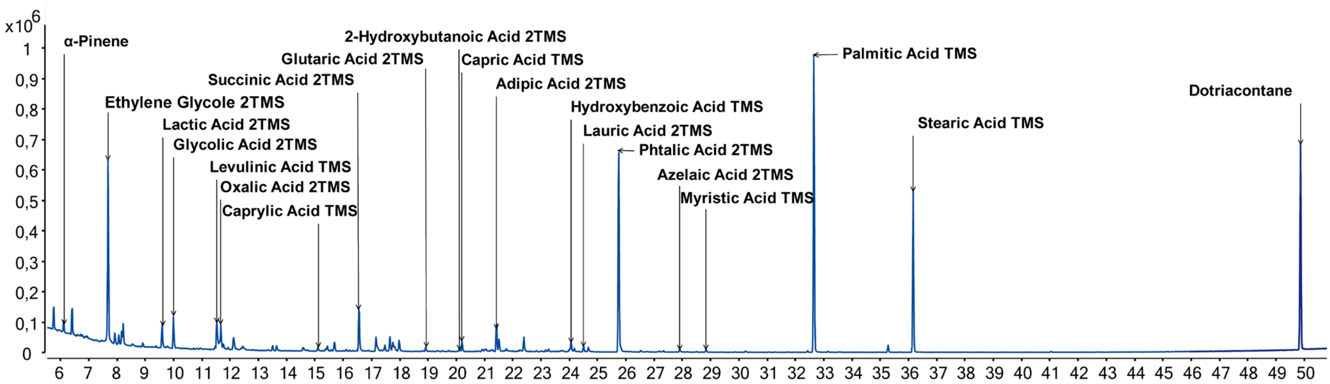

Fig. 3: Chromatogram of sample X13 according to extraction protocol b.

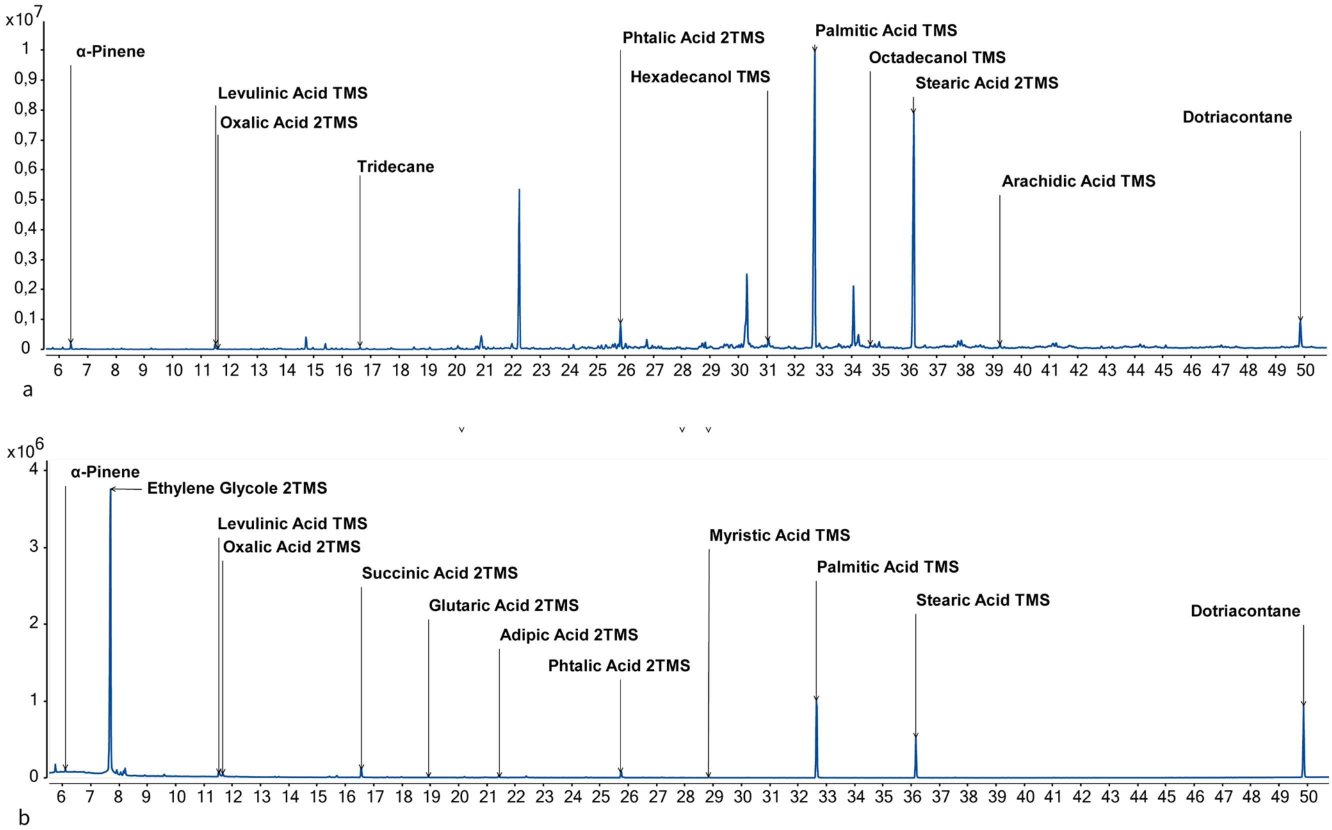

Fig. 4: a: Chromatogram of sample X16 according to extraction protocol a. b: Chromatogram of sample X16 according to extraction protocol b.

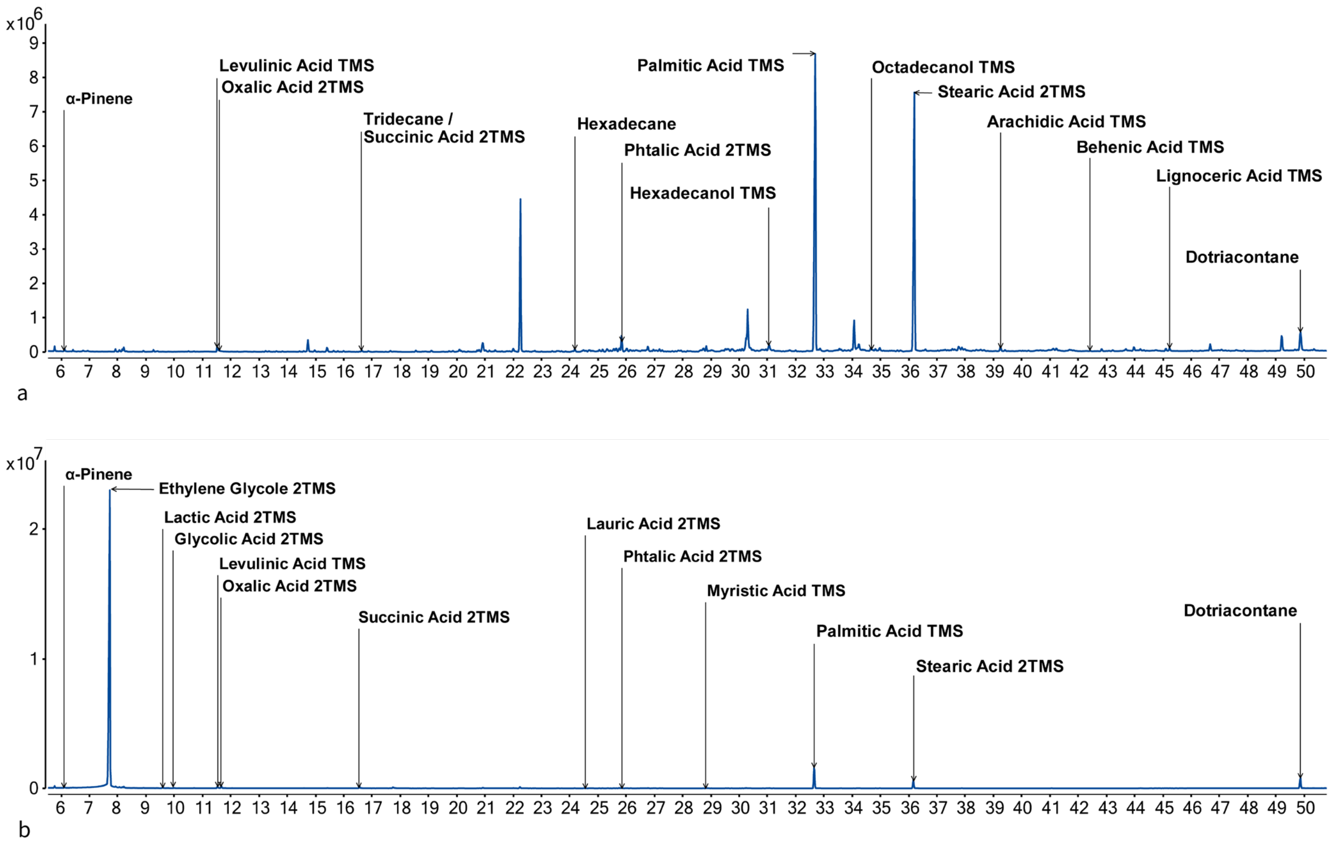

Fig. 5: a: Chromatogram of sample X17 according to extraction protocol a. b: Chromatogram of sample X17 according to extraction protocol b.

No.	Type	Site	Find spot	Context	Date	Content
X1	Vessel (amphora) (2 fragments)	Yeha	Palatial building	Destruction layer in the corridor on the floor	late first half of 1st mill. BCE	fruit/juice (wine?) animal
X2	Vessel (pot)	Yeha	Palatial building	Destruction layer in room (in situ)	late first half of 1st mill. BCE	plant (oil)? animal fat?
X3	Vessel (pot)	Yeha	Palatial building	Destruction layer in room (in situ)	late first half of 1st mill. BCE	animal fat plant (oil)? resins?
X11	Content of X3	Yeha	Palatial building	Destruction layer in room (in situ)	late first half of 1st mill. BCE	animal fat
X9	Vessel (pot)	Yeha	Palatial building	Destruction layer in room (in situ)	late first half of 1st mill. BCE	plant resins?
X10	Vessel (jar)	Yeha	Palatial building	Destruction layer in room (in situ)	late first half of 1st mill. BCE	animal fat
X15	Vessel (pot)	Yeha	Palatial building	Destruction layer in room (in situ)	late first half of 1st mill. BCE	animal fat plant oil
X12	Content of vessel (jar)	Yeha	Palatial building	Destruction layer	late first half of 1st mill. BCE	plant oil, i.e., *Brassicacae* seed oil? animal fat?
X13	Residues of small vessel (beaker)	Yeha	Palatial building	Destruction layer	late first half of 1st mill. BCE	plant oil, i.e., *Brassicacae* seed oil? animal fat?
X14	Vessel (small pot)	Yeha	Palatial building	Destruction layer	late first half of 1st mill. BCE	animal fat plant oil
X17	Vessel (jar)	Yeha	Palatial building	Destruction layer	late first half of 1st mill. BCE	plant oil, i.e., Moringa seed oil?
X4	Vessel (bowl?)	Yeha	Deep trench	Occupation layer	Aksumite period	animal fat plant oil resins?
X16	Vessel (jug/pot?)	Yeha	Domestic quarter	Occupation layer	Early Aksumite period	animal fat plant oil
X5	Vessel (jar/brazier?)	Melazo	Representative building	Destruction layer	late first half of 1st mill. BCE	Animal fat? ruminant milk fat?
X6	Vessel (beaker)	Melazo	Representative building	Destruction layer	late first half of 1st mill. BCE	fruit/juice (wine?) animal fat?
X7	Vessel (juglet)	Melazo	Representative building	Destruction layer	late first half of 1st mill. BCE	fruit/juice (wine?) animal fat? milk fat?
X8	Vessel (jar)	Melazo	Representative building	Destruction layer	late first half of 1st mill. BCE	fruit/juice (wine?) animal fat?

Fig. 6: List of the analysed samples from Yeha and Melazo ordered according to their find spot, with indication of context, date and possible content.

are controversial and not sufficient to identify wine (Drieu – Rageot – Wales et al. 2020), especially in absence of tartaric, fumaric, malic acids as evidence for grapes, other fruits may be a source of the detected compounds (Lundy – Drieu – Orecchioni et al. 2023). However, the detection of tartaric acid strongly depends on the adopted extraction protocol (Drieu – Rageot – Wales et al. 2020). The presence in the samples X1, X6, X7, and X8 of malonic acid, possibly formed by oxidation of malic acid, among the dicarboxylic acids group, may be related to fruits/fruit juice, with X1 and X6 being elongated closed forms likely suitable for the consumption of liquid contents. Nevertheless, the occurrence of dicarboxylic acids (i.e., oxalic and succinic acids) together with lactic acid indicates possible fermentation products and/or microbial degradation in the samples. Oxalic acid is present also in the samples X1, X4, X7, X8, while benzoic acid is nearly ubiquitous and 4-Hydroxybenzoic acid has been detected in the samples X1, X11, X4, X7, X8, X10. Small carboxylic acids also result as breakdown products by heating carbohydrates (Matheson – Vickruck – McEvoy et al. 2022). However, the white inner surface of sample X5 may be also related to dairy products, either consumed or used as coating (Hammann – Whittle – Cramp et al. 2018; Drieu – Lepère – Regert et al. 2020). In samples X3, X4, X9 cinnamic acid in trace may suggest a plant origin of the compounds. Free cinnamic acid is a characteristic aromatic compound of resins together with benzoic acid found for example in Egyptian incense burners (Modugno – Ribechini – Colombini et al. 2006).

24 In the samples X3 and X4 some n-alkanes could be observed using extraction protocol (a), suggesting also waxes (beeswax?) as possible source of the compounds.
The detection of caproic (C6:0, hexanoic acid), caprylic (C8:0, octanoic acid), and capric (C10:0, decanoic acid) together with the predominance of palmitic and stearic acids in all samples may point to ruminant milk fats.

25 A series of dicarboxylic acids, i.e., adipic and azelaic acid, a breakdown product of large (unsaturated) fatty acids, together with major saturated fatty acids, i.e., myristic, palmitic, stearic acids, are present in the samples X12 and X13. Pelargonic, salicylic and lauric acids found in the samples are to be related to plant fats (Irto – Micalizzi – Bretti et al. 2022). In the same samples capric acid (C10:0) may be related to animal and plant fats (Regert 2011), while small carboxylic acids, i.e., levulinic, oxalic, benzoic, succinic and glutaric acids, may be indicative of fermentation of fruits and/or microbial degradation. Some oxidation may has been caused by the conflagration of the building, as α-pinene, a wood monoterpene, is present in all samples. However, its presence might also have resulted from burning of the clay material.

26 The use of *Brassicacae* seed oil could be suggested by the presence of dodecanedioic acid in samples X12 and X13, an oxidation by-product of aged erucic acid, which is a chemical fingerprint of *Brassicacae* seed oil together with gondoic acid (Colombini – Modugno – Ribechini 2005; Irto – Micalizzi – Bretti et al. 2022: Fig. 3.).

27 In the samples X16 and X17 palmitic and stearic acids together with short chain fatty acids, i.e., capric acid, may point to degraded animal ruminants / plant(?) fats. In sample X17 small carboxylic acids are also present together with plant related compounds, i.e., pelargonic, salicylic, lauric acids. In X16 and X17 arachidic acid (C20:0) also points to a possible plant origin. Interestingly, in sample X17 behenic acid and lignoceric acid, two long-chain saturated fatty acids (C22:0 and C24:0) are observable. Together with palmitic acid it may be also considered an indicator for beeswax,[15] but a more complete series of long-chain fatty acids as well as a lack of longer chain alcohols (C26, C28, C30, C32 alcohols) renders this assignment less probable (Charters – Evershed – Blinkhorn et al. 1995; Garnier – Cren-Olivé – Rolando et al. 2002; Roffet-Salque – Regert – Evershed 2015). However, behenic acid is also present in coconut, palm and peanut oils (Irto – Micalizzi – Bretti et al. 2022) and it is considered a specific compound for ben oil, i.e., Moringa (*peregrina*) seed oil (Djaoui – Garnier – Dodinet 2015). Moringa (*peregrina*) is a plant geographically pertinent to Egypt, the Arabian Peninsula and Eastern Africa. Ben oil is an edible oil especially known for its cosmetic use and medical properties since antiquity[16]. In total, the constituents show a plant-derived pattern.

15 Lignoceric acid dominates modern beeswax lipid distribution in Dunne – Höhn – Franke 2021. *Ibidem* on the importance of bee products, honey and larvae both as a food source and in the making of honey-based drinks in West Africa.

16 Pliny: HN XII, 100 and XIII, 2.8. Fadl 2013, on the use of moringa in Ptolemaic Egypt.

28 In the samples X14 and X15, on one hand, small carboxylic acids, i.e., lactic, levulinic, oxalic, glycolic, benzoic, succinic acids, are present, together with some probable plant-related compounds, i.e., pelargonic and lauric acids, but on the other hand, capric acid and the major myristic, palmitic, stearic acids may be related to animal (ruminants, e.g., goats' milk) fats as well as to plant (seed oil?) fat.

29 The presence of hydroxybutanoic acid, an oxidation product of butanoic acid (C4:0), a short chain fatty acid, may corroborate the interpretation of the residues in samples X4, X8, X10, X14, X15 as animal fats.

Conclusion

30 Following from the above analysis, it is possible to note the similarity of components in all archaeological samples, in which unspecific markers and discrete fermentation markers (fruits?) are present (Fig. 6). The ubiquity of palmitic acid and stearic acid in conjunction with short chain fatty acids (i.e., caproic, caprylic, capric acids) may suggest an animal origin of fats, while the presence of unsaturated fatty acids, e.g., oleic acid, together with lauric, arachidic, pelargonic, valeric, salicylic acids may indicate a plant (oils) origin of the compounds. Possible plant oil sources in the region could be *Brassicacae* and linseed (Seegeler 1983). Nevertheless, we lack direct proofs for linseed in terms of specific unsaturated fatty acids, and also of other markers like, e.g., coumaric acid or ferulic acid. In one jar (X17) behenic acid together with other medium- to long-chain saturated fatty acids may suggest moringa seed oil as possible content. Although most of the markers found in the samples are present either in vegetable oil or animal fats, from the lipid extract analysis it was not possible at this stage to state the precise origin of these components.

31 The lack of additional information in the lipid extract analysis could be attributed to the loss of analytes information in the scale-down procedure. The application of further extraction protocols as well as the extension of the number of analysed samples may enhance the interpretative potential of the results in the future allowing identification of more specific compounds characterising the complex organic mixtures and related coherent groups of vessels according to their use/function, for example in the case of samples X2, X3, X9, X15, X11, which belong to similar forms from a coherent context, i.e., find complex.

32 Providing that the vessels where used for food consumption, the results of the residue analyses could also be interpreted from an archaeological point of view in such a way that in the pre-Aksumite period the vessels were not differentiated for the use of animal or vegetable food (Fig. 6). The supposed pots from the closed context in the palatial building (X2, X3, X9, X15) appear to have held predominantly plant-based content. However, the presence of animal fat in one of the pots suggests a diverse use. Due to their thin walls and lack of smoke traces, they probably served as dishes for the presentation of food rather than for cooking. Nonetheless, non-food uses of the pots, such as the preparation or storage of medicine, cosmetics or other fat-based products such as tallow, should also be considered (e.g., Shillito 2019). Fermented fruit and the possible wine prove that certain vessel shapes were intended for the transportation/storage of (semi-)liquid products. At least the two vessels X1 and X6 would fit this theory very well, one as a storage vessel, the other perhaps actually as a drinking vessel. The other two containers may have been used as storage vessels or for serving, but were probably not restricted to fruits.

33 The supposed *Brassicacae* seed oil was found in a jug and a small vessel – both being used as containers. And another jar could have possibly contained Moringa oil. Both oils in question served various purposes. Moringa oil was widely used in

ancient times as carrier for perfumes or as ingredient in ointments (e.g., Kondo 1991). *Brassicacae* seed oil, amongst other uses, was utilised as a lighting appliance in antiquity (e.g., Colombini – Modugno – Ribechini 2005), and vessel X13 might have been used as a small lamp. Therefore, jugs and jars hold plant oil and plant food as well as animal fat. The pots hold plant and/or animal products. The beaker as well as the small vessels also appear to have been widely used.

34 Non-food substances absorbed in the ceramic walls, such as organic matter applied to the ceramic to seal and treat the internal surface, may also contribute to the lipid profile of the vessels analysed. Surface treatment with resins, pitch, tar, beeswax and bitumen, but also dairy products, animal fats, fish or vegetable oils is an ethnographically known are practices. Biomarkers of surface treatment and the actual use of the containers can overlap and be hardly interpreted (Drieu 2020).

35 Overall, the results suggest that in the pre-Aksumite period in Yeha, both plant and animal foods were consumed and/or their oils and fats were used for other purposes, and processed fruits were also eaten/drunk. Unfortunately, the proportion of plant and animal products cannot be clearly determined. Currently, there seems to be a slight preponderance of the plant-based use when transferring the organic residue results to the vessel forms. In addition, several samples come from palatial buildings and therefore do not allow a direct transfer to the foodways of the entire population. However, the ceramic vessel repertoire is similar to that of contemporaneous, non-palatial habitation contexts (see catalogue for comparisons). Thus, the eating habits and culinary practices of the pre-Aksumite people could have differed from that of today, but this still needs to be verified by further investigations.

36 The analysis of organic residue absorbed in pottery provides us with some new glimpses on the use and function of ceramic vessels and on resource/food preparation and consumption. On the one hand, these first results obtained with GC-MS show the potential of the technique for combining ceramic studies with scientific analyses. On the other hand, the occurrence of non-specific and almost ubiquitous compounds also displays the limitations of this technique and the need to integrate further biomolecular analyses, e.g., with the analysis of stable isotopes (Roffet-Salque – Dunne – Altoft et al. 2017). However, the inclusion of other techniques and further work with additional samples promises to refine the level of knowledge in the future.

Catalogue

37 The following catalogue contains the necessary data for the samples.

X1 – Yeh12 Ke 511 plus second sample (62)

Vessel fragment (Fig. 7)

Find Context: Yeha, area B, trench XIV, locus 13, in situ, exposed to fire

Description: Bag-shaped vessel with a bulbous lower section and a rounded base, a vertical handle, an out-flaring rim and a smoothed surface. Medium brown colour.

Production: Local/regional

Dating: Late first half of the 1st millennium BCE

Comparison: De Contenson 1963: Pl. LIV, Fig. d

Sample Area: Base

X2 – Yeh12 B 67

Vessel fragment (Fig. 7)

Find context: Yeha, area B, trench XX, locus 16, in situ, exposed to fire

Description: Thin-walled pot with a globular body, a rounded base, a sloping shoulder with two upright horizontal coil handles, an out-flaring rim and incised oblique lines as decoration on the shoulder area. Medium brown colour.

Production: Local/regional

Dating: Late first half of the 1st millennium BCE

Comparison: Phillips 2000: 306, Fig. 266–267

Sample Area: Base

X3 – Yeh17 B 73

Vessel fragment (Fig. 7)

Find context: Yeha, area B, trench XX, locus 16, in situ, exposed to fire

Description: Thin-walled pot with a globular body, a rounded base, a sloping shoulder with two upright horizontal coil handles, an out-flaring rim and a combed decoration on the upper body part in form of a hasty incised grid design. Medium brown colour.

Production: Local/regional

Dating: Late first half of the 1st millennium BCE

Comparison: Phillips 2000: 306, Fig. 266–267

Sample Area: Base

X4 – Yeh17 Ke 106

Vessel fragment (Fig. 7)

Find context: Yeha, area E, trench VI, locus 17

Description: Fragment of the flat base of a thick-walled medium-sized bowl (dm 17 cm) with oblique ripped outer surface. Red-brown colour.

Production: Regional

Dating: Aksumite (Red Aksumite Ware)

Comparison: Wilding 1989: 256, Fig. 158

Sample Area: Base

X5 – Haw17 Ke107

Vessel fragment (Fig. 8)

Find context: Melazo, area B, trench I, locus 4, destruction layer

Description: Fragment of medium brown to red-brown fabric belonging to a probable pot or brazier with white deposits on the inner surface.

Production: Local/regional

Dating: Late first half of the 1st millennium BCE

Sample Area: Body sherd

X6 – Haw17 B 01

Vessel fragment (Fig. 8)

Find Context: Melazo, area B, trench I, locus 4, destruction layer

Description: Double-conical vessel with a flat base, an out-flaring rim and an applied vertical knob-handle on the upper exterior. Red-brown burnished outer surface.

Production: Local/regional

Dating: Late first half of the 1st millennium BCE

Comparison: Anfray 1967: Pl. XLII, Fig. 3301

Sample Area: Body sherd near the base

X7 – Haw17 Ke 04

Vessel fragment (Fig. 8)

Find Context: Melazo, area B, trench I, locus 1/4

Description: Body wall fragment of a vessel with incised horizontal comb lines and notches as well as two small longish knobs on the exterior. Red-brown burnished outer surface.

Production: Local/regional

Dating: Late first half of the 1st millennium BCE

Comparison: Fattovich – Hiluf Berhe – Phillipson et al. 2010: 70, Fig. 15; Köster 2021: Fig. 17

Sample Area: Body sherd below the shoulder area

X8 – Haw17 Ke 21

Vessel fragment (Fig. 8)

Find Context: Melazo, areal B, trench I, locus 4

Description: Out-turning foot of a larger jar (dm 21 cm). Medium-brown smoothed surface.

Production: Local/regional

Dating: Late first half of the 1st millennium BCE

Comparison: Phillips 2000: 307, Fig. d

Sample Area: Body sherd near the base

X9 – Yeh17 B 66

Vessel fragment (Fig. 8)

Find context: Yeha, area B, trench XX, locus 16, in situ, exposed to fire

Description: Thin-walled pot with a globular body, a rounded base, a sloping shoulder with two upright horizontal coil handles, an out-flaring rim and a combed decoration on the upper body part in form of a hasty incised grid design. Red-brown colour.

Production: Local/regional

Dating: Late first half of the 1st millennium BCE

Comparison: Phillips 2000: 306, Fig. 266–267

Sample Area: Body sherd below the shoulder area

X10 – Yeh17 B 70

Vessel fragment (Fig. 8)

Find context: Yeha, area B, trench XX, locus 16, in situ, exposed to fire

Description: Fragment of a vessel with out-flaring rim. Red-brown colour.

Production: Local/regional

Dating: Late first half of the 1st millennium BCE

Sample Area: Body sherd

X11 – Yeh17 B 73

Soil content from the vessel's interior

Find Context: Yeha, area B, trench XX, locus 16, in situ, exposed to fire

Description: Content of the thin-walled pot X3.

X12 – Yeh19 Ke 04 (8)

Soil content from the vessel's interior

Find Context: Yeha, area B, trench XX, locus 7, exposed to fire

Description: Soil from the interior of a jar of local/regional production, that can be dated to the late first half of the 1st millennium BCE.

X13 – Yeh19 B 95 (10)

Residues from the vessel's interior

Find Context: Yeha, area B, trench XXIX, locus 10, probably exposed to fire

Description: Residues from the vessel's interior. The vessel was a small handmade beaker with an almost flat base and straight body wall with a rounded rim of local or regional production (Fig. 9). The vessel can be dated to the late first half of the 1st millennium BCE (Comparison: Phillips 2000: 304, Fig. 265 d; Curtis 2004: 61, Fig. 4; Wolf – Nowotnick 2010: 184, Fig. 19)

X14 – Yeh18 B 87 (66)

Vessel fragment (Fig. 9)

Find Context: Yeha, area B, trench XIX, locus 1/6 (floor of the palatial structure), exposed to fire

Description: Small bag-shaped vessel with a rounded base, a bulbous body with the largest diameter on the lower part and a short out-turning rim. Four vertical ridges are applied on the exterior.

Production: Local/regional

Dating: Late first half of the 1st millennium BCE

Comparison: Anfray 1963: Pl. CXXXVI b; Awam Cemetery, Marib/Yemen (unpublished)

Sample Area: Body sherd

X15 – Yeh17 B 69 (67)

Vessel fragment (Fig. 9)

Find Context: Yeha, area B, trench XX, locus 16, exposed to fire

Description: Thin-walled pot with a globular body, a rounded base, a sloping shoulder with two upright horizontal coil handles, an out-flaring rim and a combed decoration consisting of a coarse incised oblique incisions. Medium brown colour.

Production: Local/regional

Dating: Late first half of the 1st millennium BCE

Comparison: Phillips 2000: 306, Fig. 266–267

Sample Area: Base

X16 – Yeh19 Ke 161 (61)

Vessel fragment (Fig. 9)

Find Context: Yeha, area D, trench IV, locus 11

Description: Out-curving rim fragment of a jug.

Production: Local/regional

Dating: Early Aksumite (1st–4th c. CE)

Sample Area: Body sherd of upper shoulder area

X17 – Yeh19 Ke 170 (88)

Vessel fragment (Fig. 10)

Find Context: Yeha, area B, trench XXX, locus 48, exposed to fire

Description: Jar.

Dating: Due to find context probably first half of the 1st millennium BCE

Sample Area: Body sherd

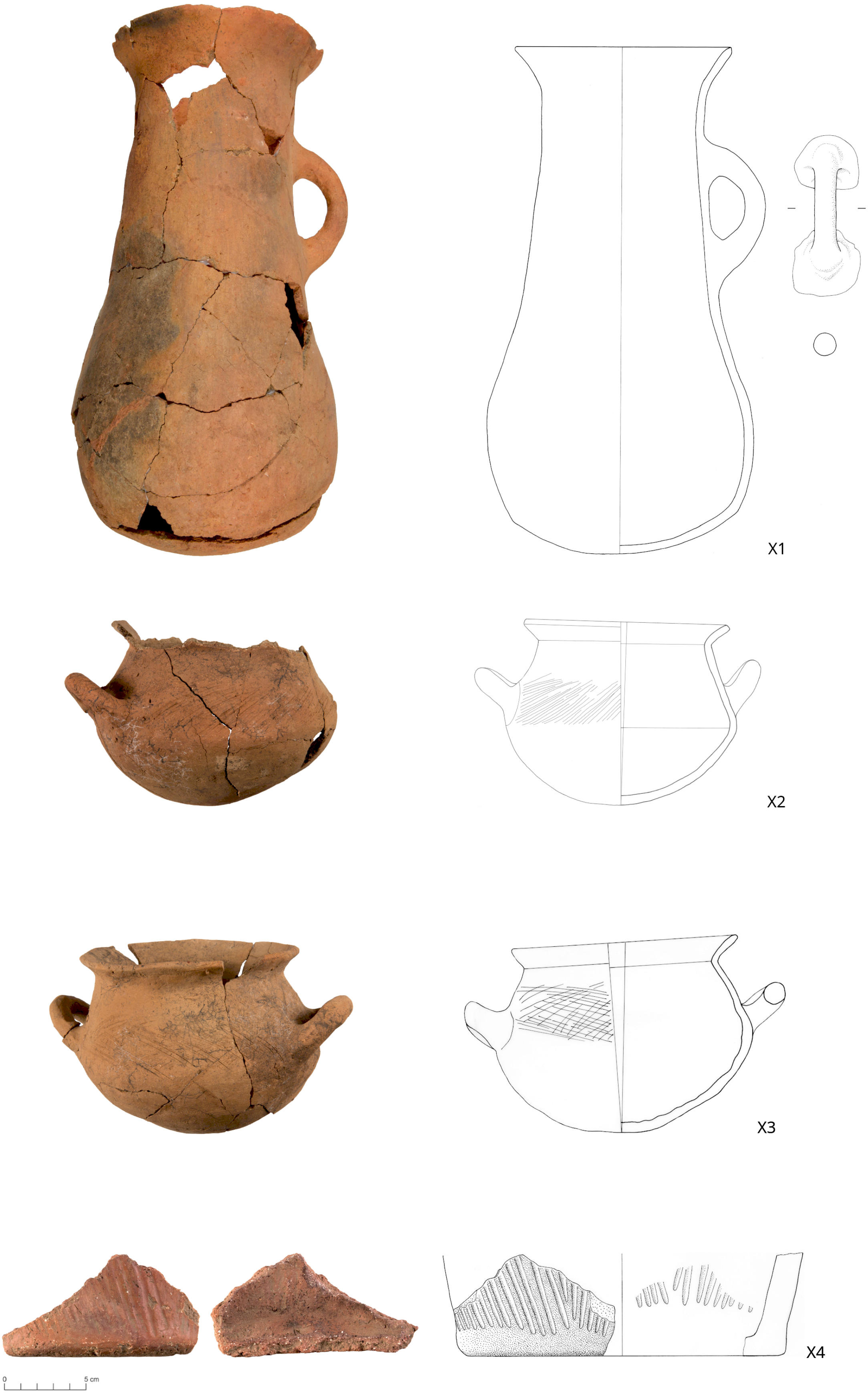

Fig. 7: Objects X1–X4.

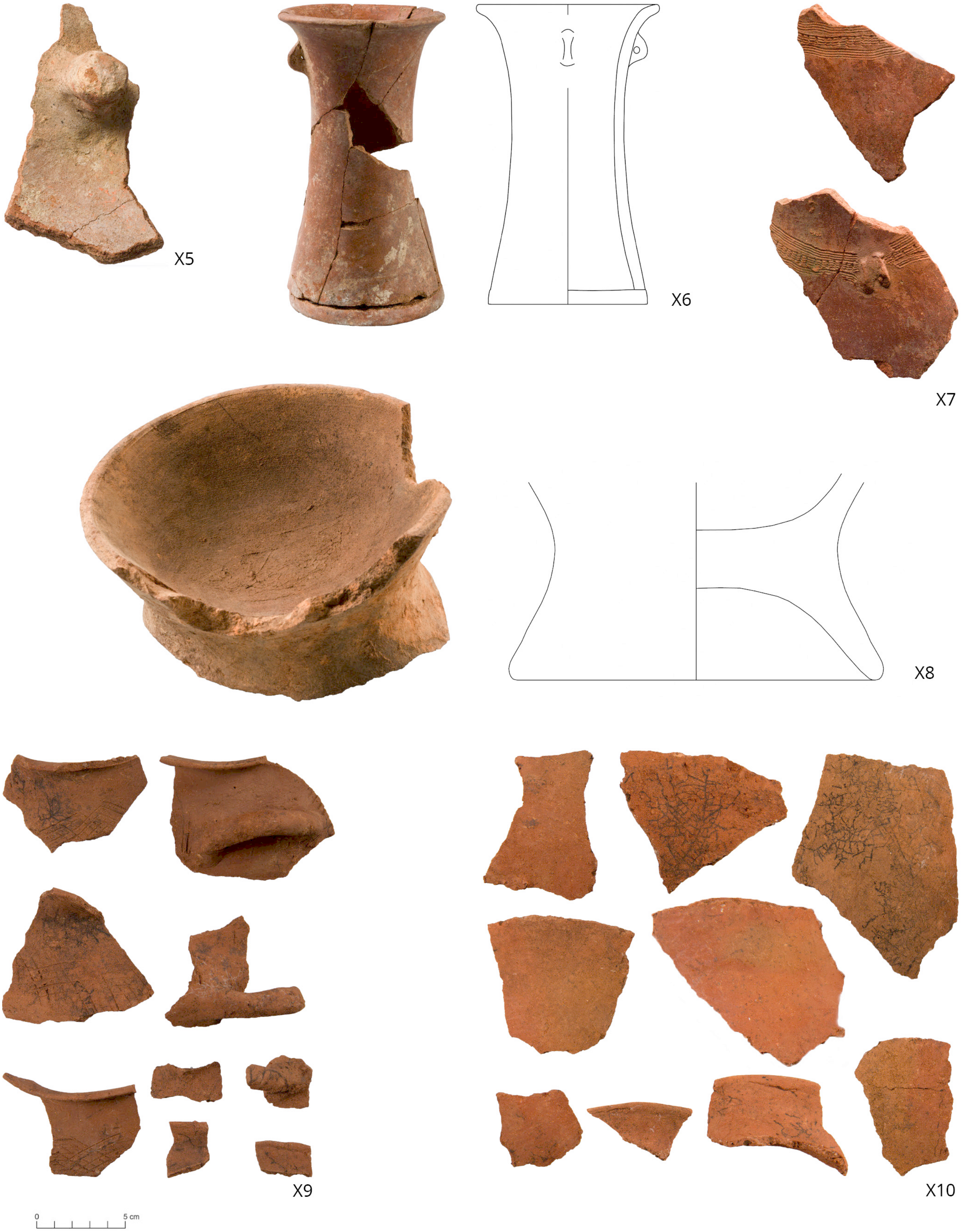

Fig. 8: Objects X5–X10.

Fig. 9: Objects X13–X16.

Fig. 10: Object X17.

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