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ORGANIC RESIDUES IN ANCIENT POTTERY SHERDS FROM SITES IN JORDAN

Abdulraouf. S. Mayyas

Department of Conservation Science Queen Rania Faculty of Tourism and Heritage,
The Hashemite University, P. O. Box. 330127, Zarqa 13133, Jordan.
(A_S_Mayyas@hotmail.com)

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ABSTRACT

This paper discusses the analysis of organic residues preserved in the fabric of twelve pottery sherds (two Bronze Age and ten Iron Age) excavated from three sites: Jneneh, Sahab and Tell Abu al-Kharaz. Gas chromatography-mass spectrometry was used for separation and identification of organic constituents. Conventional solvent extraction was used for the extraction of residues preserved in their fabrics. The analysis showed that five sherds out of the twelve preserve significant organic constituents derived from plant and animal sources. The results inform that plant oil (most likely olive oil) could have been introduced into four Iron Age vessels (three from Jneneh and one from Tell Abu al-Kharaz) while animal fat of unknown source could have been introduced into one Iron Age vessel from Sahab. Evidences of the availability and exploitation of plant materials, such as oils and animal fats during the Iron Age were also presented in this paper.

KEYWORDS: Bronze Age, Iron Age, Pottery, Organic Residue, Lipid, Biomarker, Gas Chromatography - Mass Spectrometry, Extraction.

1. INTRODUCTION

Analysis of organic residues preserved in ancient pottery vessels has the capacity of supplying archaeologists with hidden data preserved in the fabric of these vessels. Ancient pottery vessels were exploited in the past for different uses such as storing, processing and serving different materials. As a result, chemical constituents of organic materials can penetrate to the pores of these vessels and retain there. During use and/or burial, however, degradative processes, such as thermal, chemical and microbial degradation can affect the distributions and chemical structures of these constituents (Evershed et al., 1992; Dudd et al., 1998, 1999). Consequently, organic residues are usually present as a complex mixture of organic molecules and can be preserved for long periods of time inside the pores of pottery vessels under favorable conditions (Evershed et al., 2001; Evershed, 2008). These molecules are called "biomarkers", which are characteristic of the original natural material used in the vessel. In the laboratory, organic constituents can be extracted using organic solvents and then separated, characterised and identified using trace analytical techniques, such as gas chromatography-mass spectrometry (GC-MS). Residues of natural materials including plant oils and animal fats preserved in the fabrics of ancient pottery vessels were detected and reported by many researchers (Passi et al., 1981; Shimoyama et al., 1995; Evershed et al., 1997a; 1999, 2002; Regert et al., 1998; Mottram et al., 1999; Kimpe et al., 2001, 2002; Colombini et al., 2005a; Copley et al., 2005; Romanus et al., 2008; Garnier et al., 2009; Mayyas et al., 2013; Koh and Birney 2017). Such studies can provide pioneering data on ancient materials used in these vessels (Heron et al., 1991; van Bergen et al., 1997; Dudd et al., 1999; Regert et al., 2001; Stern et al., 2003; Evershed 2008; Gregg et al., 2009; Baeten et al., 2010; Isaksson and Hallgren, 2012), and thereby, useful data on human activities at the site through time. This paper reports the results obtained from GC-MS analysis of absorbed organic residues extracted via conventional solvent extraction from twelve (two Bronze Age and ten Iron Age) pottery shreds excavated from three archaeological sites of Jneneh, Sahab and Tell Abu al-Kharaz with the aim of determining the type of the materials used in their fabrics and the potential uses of their mother vessels.

The archaeological site of Jneneh is a small (ca. 4.5 ha.) Iron Age II settlement dated to the 8th century B.C. The site located in the north central Jordan at the western bank of Wadi az-Zarqa in the north western periphery of the modern city of Zarqa (grid ref. 250.88E 165.25N). It was first occupied during the Early Bronze Age (I/II and IV) in a form of an unfortified settlement unlike the nearby heavily fortified Early Bronze Age II-III settlement of Khirbet al-Batrawy (Nigro et al., 2008; Nigro and Sala, 2009; Nigro, 2009, 2010). Then the site was settled during the Iron Age II as a small open town with a fortified acropolis.

Sahab site is situated about 12 km south-east of Amman and about 15 km to the west of the Jordanian desert. The Tell is located in a transitional zone between the highlands and the desert. Six main occupational levels have been recognized at the different areas of the site, starting from the Chalcolithic period, and extending through the Bronze Age, Iron Age, Roman and Byzantine periods, up to the Islamic Age (Ibrahim, 1972). The main occupational history of the site, however, extends from the late Neolithic/Chalcolithic period (5th and 4th millennia B.C.) to the late Iron Age (6th century B.C.) (Ibrahim, 2016a). The site is the largest, and one of the last major pre-classical settlements on the borders of the desert of Eastern Jordan.

Tell Abu al-Kharaz is a large tell site (ca. 12 ha.), located in the east central Jordan Valley, ca. 4 km east of Jordan River and 6 km south-southwest of Pella, Jordan (PGC: E 206 196.54 and N 200 623.07.). The natural tell is rising up to ca. 60 m above the surrounding areas. Excavations revealed that the site was occupied from the Chalcolithic to the Islamic periods. It is most likely that Tell Abu al-Kharaz is identical with *Jabesh Gilead*: this city is mentioned frequently in the Old Testament (Fischer, 2012: 165). However, the main settlements were belong to Early Bronze Ages IB and II, Late Middle Bronze Age, Late Bronze Ages I and II and Iron Ages I and II. One of the most interesting find in Tell Abu al-Kharaz is the Iron Age I town (phase IX 1200-1050 B.C) (Fischer, 2012). Evidence demonstrates a wealthy Iron Age I society which had contacts all over the Eastern Mediterraneanin the 12th and 11th centuries BC (Fischer, 2012). Pottery vessels excavated from each of the three sites are diverse and characterised by different forms, sizes, fabrics and decorations that infer the presence of different chronological periods. Pottery sherds subjected to this study, however, belong to the Bronze and Iron Ages and were described in Table 1 and shown in Table 2.

2. RESEARCH METHODOLOGY

2.1. *Materials and sampling*

All solvents and reagents used in this research were ultrapure or HPLC grade. Dichloromethan, methanol were used as solvens. *n*-tetratriacontane (C_{34}) was used as internal standard (IS) and N,O-bis(trimethylsilyl)trifluoroacetamide (BSTFA) in 1% v/v trimethylchlorosilane (TMCS) was used as deri-

vatising agent. The glassware used for sampling was cleaned. All glassware and one sherd of unused modern pottery vessel utilized as a control sample were heated in an oven at 550 °C for 5 hours before use. The available archaeological pottery sherds and the control sherd were stored in aluminum foil and then were sampled and analysed alongside. The twelve pottery sherds lack visible organic residues on their surfaces (Tables 1 and 2).

Powdered pottery samples were taken individually from the interior (from the internal surface to the core) of the sherds using a Dremel electric drill fitted with a tungsten abrasive bit. Each sample was taken after having scraped and discarded the outer surface layer using a scalpel blade, to avoid contamination (Evershed et al., 1999, 2002). About 0.5 g of each powder was transferred into a clean glass vial. The powder was subjected to conventional solvent extraction and derivatisation procedures adopted from Regert et al., (1998); Evershed et al. (1999, 2002) and Copley et al. (2005).

2.2. *The Analytical Technique of GC-MS*

Gas chromatography-mass spectrometry (GC-MS) analysis was carried out using a Varian CP-3800 Gas Chromatograph coupled to Varian Saturn 2200 Ion trap Mass Spectrometer. The GC was equipped with a split/splitless injector. Splitless mode was used. Helium was used as a carrier gas, with a constant head pressure of 1 psi and a flow rate of 1 ml/min at 50 °C. The inlet of the GC and the transfer line of the MS were maintained at 320 °C and 350 °C, respectively. The temperature of the oven was programmed from 50 °C (2 min isothermal) to 340 °C (20 min isothermal) at a rate of 10 °C/min. A FS-Supreme-5ms/HT column of 30m length and 0.25mm internal diameter coated with a stationary phase film of 0.1µm thickness was used. The column was directly inserted into the ion source. Electron impact (EI) spectra were obtained at 70 eV with full scan from *m/z* 50 to 700 amu. Because there were no significant GC signals in the analysed samples at higher retention times (above 35 min) the chromatograms shown in this paper are displayed in the range of 10 – 35 min retention time. This allows for a better presentation and more clear identification of the relevant compounds.

3. RESULTS AND DISCUSSION

Chemical constituents obtained from GC-MS analysis of organic residues preserved in the twelve sherds are shown in Table 3 and discussed herein.

3.1. *Organic extracts of sherds Jn3, Jn5, Jn13 and TK11.*

GC-MS analysis of the solvent soluble extracts of the interiors of sherd Jn3 and Jn5 revealed the preservation of informative lipid constituents (Fig. 1). These are saturated $C_{14:0}$, $C_{16:0}$, $C_{18:0}$, $C_{20:0}$, $C_{22:0}$ and $C_{24:0}$, monounsaturated $C_{18:1}$ and diunsaturated $C_{18:2}$ fatty acids, in addition to the plant sterol, sitosterol. $C_{x:y}$ is a monocarboxylic fatty acid of carbon chain length x and degree of unsaturation y. The total amount of the lipids retrieved from the two sherds is 143 and 132 µg lipid per one gram of dry pottery powder, respectively. On the other hand, GC-MS analysis of the solvent soluble extracts of the interiors of sherds Jn13 and TK11 showed some differences in their lipid profiles (Figs. 2 and 3) compared with that of the first two sherds. The extract of TK11 contains saturated $C_{9:0}$, $C_{12:0}$, $C_{16:0}$ and $C_{18:0}$ fatty acids, monounsaturated $C_{18:1}$ fatty acid and monoacylglycerols, MAG-$C_{16:0}$ and MAG-$C_{18:0}$, in addition to the sitosterol (Fig. 2). The only difference between the two extracts is that the extract of sherd Jn13 contains two *n*-alcohols, AL_{16} and AL_{18}, and lacks to MAG-$C_{18:0}$ and sitosterol plant biomarker (Fig. 3) compared with the extract of sherd TK11 (Fig. 2). The total amount of the lipids retrieved from each sherd did not exceed 130 µg lipid per one gram of dry pottery powder.

Saturated $C_{16:0}$ and $C_{18:0}$ fatty acids have a wide distribution and are abundant in plant oils and animal fats (Dudd and Evershed, 1998). Monounsaturated $C_{18:1}$ fatty acid is also commonly present in most plant and animal lipids (Dey and Harborne, 1997: 238; Copley et al., 2005; Gunstone et al., 2007: 4; Koh and Birney 2017). Very small proportions of $C_{18:0}$ and higher contents of $C_{18:1}$ and $C_{18:2}$ are present in vegetables and their oils, cereals and fruits (Kimpe et al., 2004). In case of vegetable oil residues detected in archaeological pottery vessels $C_{16:0}$ and $C_{18:1}$ were found in high content while $C_{18:0}$ is present in very low content (Kimpe et al., 2004). The presence of higher contents of $C_{18:1}$ and $C_{16:0}$ acid compared with the other acids, particularly $C_{18:0}$, and the occurrence of the major plant sterol (sitosterol) in Jn3 and Jn5 extracts suggests a plant origin, possibly plant oil. Olive oil is proposed since $C_{18:1}$ is present in high abundance (Koh and Birney 2017). It is worth noting that both of Jn3 and Jn5 sherds were excavated from the same season, square and locus (Table 1).

Plasticizers (P) are modern contaminants resulting from plastics. They could also have been introduced into the samples from plastic bags during storing of the pottery sherds or during laboratory preparations.

Table 1. General description of the pottery sherds analysed in this study.

#	Sherd code	Data of Excavation (Site, Season, Area, Square, locus and other data)	Part of the vessel analysed	Vessel description	Chronology
1.	JoMuS2	Sahab 1980 Area: GIII Square: 12 Locus: L11 Pail 16	Body/Base	Large storage jar with everted rim, coarse ware, low fired, wheel manufactured. Inclusion: limestone with grey and red grits, straw casts. Exterior and interior slip.	Middle Bronze Age
2.	JoMuS7	Sahab 1980 Area: GIII Square: 12 Locus: L4 Box 61, Pail 6	Body/Base	Cooking pot, coarse ware, low fired, wheel manufactured. Inclusion: limestone with grits. No slip and but with black mark of burning on the exterior surface	Iron Age*
3.	JoMuS9	Sahab 77 Area BO Square 19 5/41	Body/Base	Body/base part of possibly a jar*, coarse ware, low fired, wheel manufactured. Exterior slip.	Iron Age II
4.	JoMuS14	Sahab 80 Area: GII Square: 6 13, 165	Base	Base of bowl*, coarse ware, low fired, wheel manufactured. Exterior and interior slip.	Middle Bronze Age
5.	Jn3	Jneneh 2011 Square: IA Locus: L015 Locus type: Ash layer (fire place)	Body	Small jar/jug , medium coarse ware, low fire. Inclusions: Limestone. Three Black bands on a creamy slip. Medium thick sherd	Iron Age II
6.	Jn5	Jneneh 2011 Square: IA Locus: L015 Locus type: Ash layer (fire place)	Body	Large storage jar, coarse ware, low fire. Inclusions: Limestone. Self-sip. Thick sherd	Iron Age II
7.	Jn7	Jneneh 2011 Square: IA Locus: L015 Locus type: Ash layer (fire place)	Body	Large storage jar, coarse ware, low fire. Inclusions: Limestone. Self-sip. Thick sherd	Iron Age II
8.	Jn13	Jneneh 2011 Square: IB Locus: L008	Body	Large storage jar, coarse ware, low fire. Inclusions: Limestone. Self-sip. Sherd of medium thickness	Iron Age II
9.	TK6	Karaz 2010 Area: TLVA Locus: L273 No: 2 Phase IX, Area 9	Body	Krater, hard-fired, light brown fabric, light grey core, coarse, multicoloured inclusions, light yellow slip.	Iron Age IB
10.	TK9	Karaz 2010 Area: TLIIB Locus: L267 No: N1395 Phase IX, Area 9	Body	Jar, hard-fired, light yellowish-brown fabric, coarse, multicoloured inclusions, light yellow slip, brownish-red decoration, finger impression above one handle.	Iron Age IB
11.	TK10	Karaz 2010 Area: TLIIIB Locus: L206 No: 3 Phase X, Area 9	Body	Storage jar, hard-fired, light greyish-brown fabric, large white inclusions, light yellow patches of paint.	Iron Age IB/IIA
12.	TK11	Karaz 2010 Area: TLIIIB Locus: L237 No: 2 Phase IX, Area 9	Body	Krater, medium-hard-fired, yellowish-grey fabric, medium-coarse, mainly grey inclusions, self slip.	Iron Age IB

* Personal Contact with Luma Haddad on 8th November 2017

Table 2. Photos of the pottery sherds analysed in this study.

#	Sherd code	Photo	
		Interior	*Exterior*
1.	JoMuS2		
2.	JoMuS7		
3.	JoMuS9		
4.	JoMuS14		
5.	Jn3		
6.	Jn5		

Continue to Table 2. Photos of the pottery sherds analysed in this study.

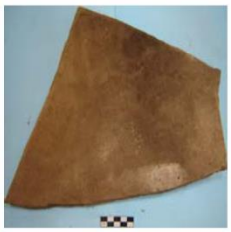
#	Sherd code	Photo	
		Interior	*Exterior*
7.	Jn7		
8.	Jn13		
9.	TK6		
10.	TK9		
11.	TK10		
12.	TK11		

Table 3. Molecular weights and chemical formulas of trimethylsilylated (TMS) organic constituents (common names in parentheses) retrieved via simple extraction from the interiors of the twelve pottery sherds

#	Organic Constituent	Name	Trimethylsilylated (TMS)	
			Molecular Weight (MWt)	Chemical Formula
1.	$C_{9:0}$	Nonanoic acid (Pelargonic acid) - TMS	230	$C_{12}H_{26}O_2Si$
2.	$C_{12:0}$	Dodecanoic acid (Lauric acid) - TMS	272	$C_{15}H_{32}O_2Si$
3.	$C_{14:0}$	Tetradecanoic acid (Myristic acid) - TMS	300	$C_{17}H_{36}O_2Si$
4.	$C_{15:0}$	Pentadecanoic acid (Pentadecylic acid) - TMS	314	$C_{18}H_{38}O_2Si$
5.	$C_{16:0}$	Hexadecanoic acid (Palmitic acid) - TMS	328	$C_{19}H_{40}O_2Si$
6.	$C_{17:0}$	Heptadecanoic acid (Margaric acid) - TMS	342	$C_{20}H_{42}O_2Si$
7.	$C_{18:0}$	Octadecanoic acid (Stearic acid) - TMS	356	$C_{21}H_{44}O_2Si$
8.	$C_{18:1}$	*cis*-9-octadecenoic acid (Oleic acid) - TMS	354	$C_{21}H_{42}O_2Si$
9.	$C_{18:2}$	*cis*,*cis*-9,12-octadecadienoic acid (Linoleic acid) - TMS	352	$C_{21}H_{40}O_2Si$
10.	$C_{20:0}$	Eicosanoic acid (Arachidic acid) - TMS	384	$C_{23}H_{48}O_2Si$
11.	$C_{22:0}$	Docosanoic acid (Behenic acid) - TMS	412	$C_{25}H_{52}O_2Si$
12.	$C_{24:0}$	Tetracosanoic acid (Lignoceric acid) - TMS	440	$C_{27}H_{56}O_2Si$
13.	AL_{16}	Hexadecanol (Palmityl or Cetyl alcohol) - TMS	314	$C_{19}H_{42}OSi$
14.	AL_{18}	Octadecanol (Stearyl alcohol) - TMS	342	$C_{21}H_{46}OSi$
15.	MAG-$C_{14:0}$	1-tetradecanoyl-glycerol (1-monomyristin) - TMS	446	$C_{23}H_{50}O_4Si_2$
16.	MAG-$C_{16:0}$	1-hexadecanoyl-glycerol (1-monopalmitin) - TMS	474	$C_{25}H_{54}O_4Si_2$
17.	MAG-$C_{18:0}$	1-octadecanoil-glycerol (1-monostearin) - TMS	502	$C_{27}H_{58}O_4Si_2$
18.	10-OH-$C_{18:0}$	10-hydroxy-octadecanoic acid - TMS	444	$C_{24}H_{52}O_3Si_2$
19.	*β*-Sitosterol	24-ethylcholest-5-en-3β-ol (Sitosterol) - TMS	486	$C_{32}H_{58}OSi$
20.	Cholesterol	Cholest-5-en-3β-ol (Cholesterol) - TMS	458	$C_{30}H_{54}OSi$
21.	DAG32	1,2-dihexadecanoyl-glycerol (1,2-dipalmitin) - TMS 1,3-dihexadecanoyl-glycerol (1,3-dipalmitin) - TMS	640	$C_{38}H_{76}O_5Si$
22.	DAG34	Mixture of 1,2- and 1,3-isomers of (hexadecanoyl-octadecanoyl-glycerol) - TMS	668	$C_{40}H_{80}O_5Si$
23.	DAG36	1,2-dioctadecanoyl-glycerol (1,2-distearin) - TMS 1,3-dioctadecanoyl-glycerol (1,3-distearin) - TMS	696	$C_{42}H_{84}O_5Si$

$C_{x:y}$: monocarboxylic fatty acid of carbon chain length x and degree of unsaturation y, AL_x: *n*-alcohol of carbon chain length x, OH: hydroxyl, MAG-$C_{x:y}$: monoacylglycerol of $C_{x:y}$ fatty acid moiety, and (DAG X): diacylglycerol with carbon chain length x.

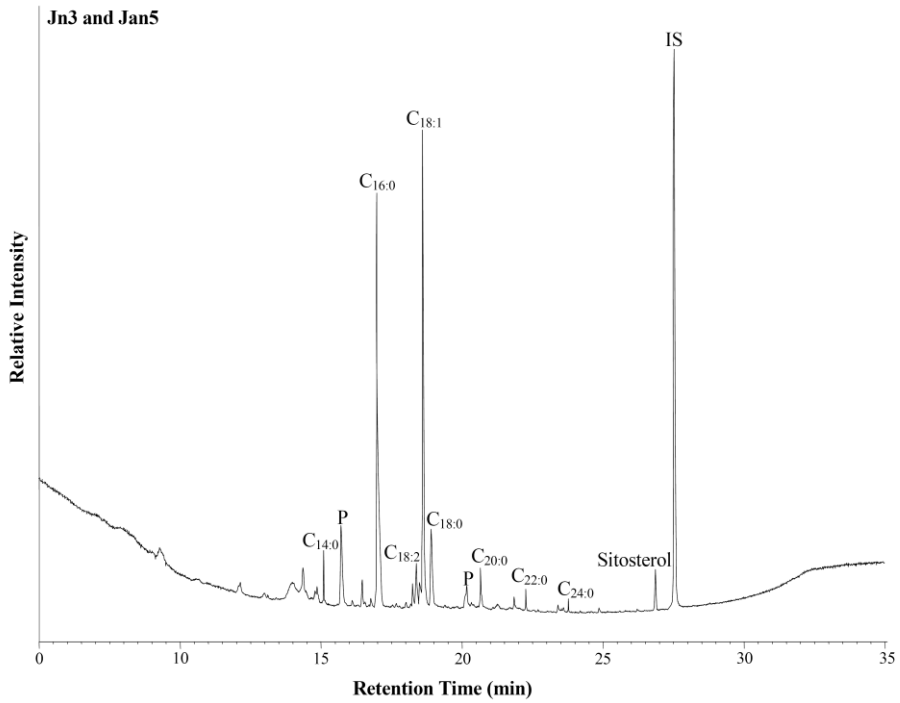


Figure 1. Partial (0-35 min) total-ion-chromatogram (TIC) of the trimethylsilylated total lipid extract of the organic residue obtained via conventional solvent extraction from the interiors of sherds Jn3 and Jn5. ($C_{x:y}$): monocarboxylic fatty acid of carbon chain length x and degree of unsaturation y, (P): plasticizer and (IS): the internal standard (n-tetratriacontane; C_{34}).

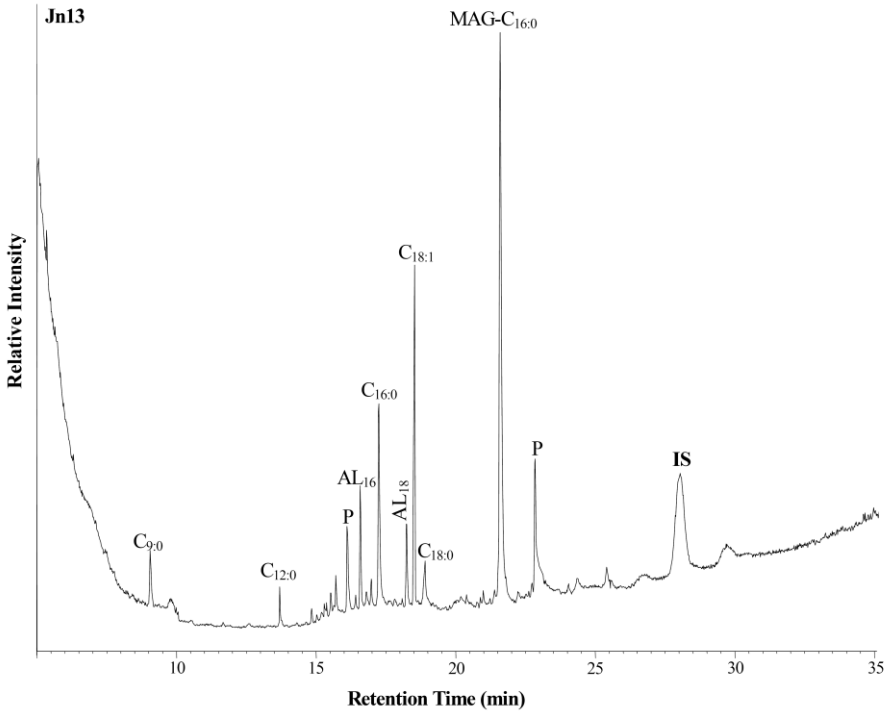


Figure 2. Partial (0-35 min) total-ion-chromatogram (TIC) of the trimethylsilylated total lipid extract of the organic residue obtained via conventional solvent extraction from the interior of sherd Jn13. ($C_{x:y}$): monocarboxylic fatty acid of carbon chain length x and degree of unsaturation y, (MAG-$C_{x:y}$): monoacylglycerol with $C_{x:y}$ fatty acid moiety, (P): plasticizer, and (IS): the internal standard (n-tetratriacontane; C_{34}).

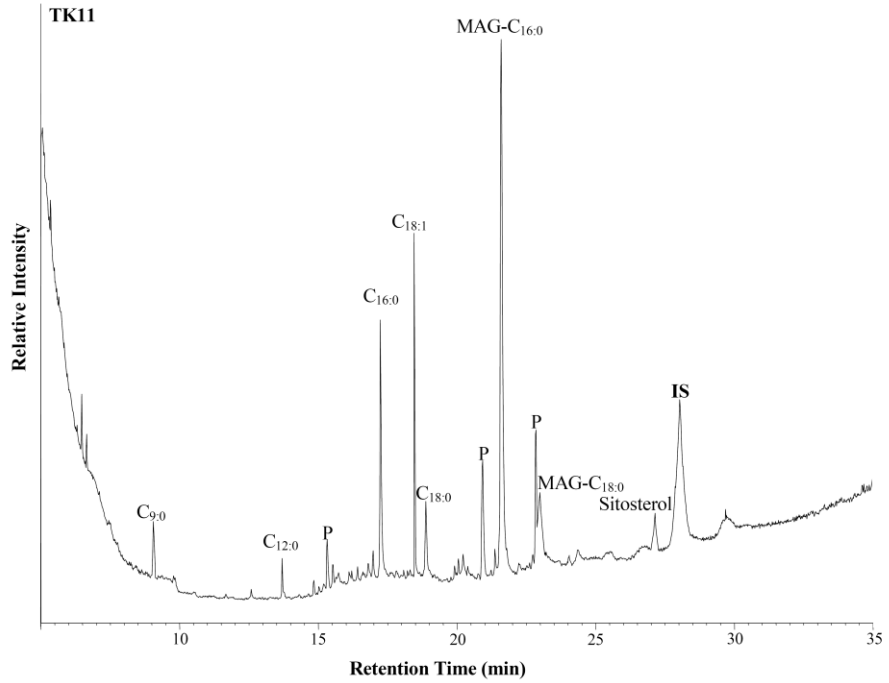


Figure 3. Partial (0-35 min) total-ion-chromatogram (TIC) of the trimethylsilylated total lipid extract of the organic residue obtained via conventional solvent extraction from the interior of sherd TK11. ($C_{x:y}$): monocarboxylic fatty acid of carbon chain length x and degree of unsaturation y, (MAG-$C_{x:y}$): monoacylglycerol with $C_{x:y}$ fatty acid moiety, (P): plasticizer, and (IS): the internal standard (n-tetratriacontane; C_{34}).

It is known that short chain fatty acids are vulnerable to leaching more than the long chain fatty acids. The presence of $C_{9:0}$ in the extracts of Jn13 and TK11 could be attributed to the oxidation of the unsaturated moieties of $C_{18:1}$ (oleic) fatty acids present in the original organic residues (Shimoyama et al., 1995; Evershed et al., 1999; Reber and Evershed, 2004a,b; Colombini et al., 2005b; Copley et al., 2005; Boran et al., 2006; Spangenberg et al., 2006; Mayyas et al., 2013; Koh and Birney 2017). The oxidation process of the unsaturated moieties of $C_{18:1}$ of archaeological residues usually yields $C_{9:0}$ and other oxidation products (Mayyas et al., 2013). Oxidation products however, are absent in the extract, most probably as a result of leaching during burial. The two monoacylglycerols (MAG-$C_{16:0}$ and MAG-$C_{18:0}$) have originated from the hydrolysis of the mother triacylglycerols of $C_{16:0}$ and $C_{18:0}$ fatty acids present in the original raw material used in the pottery vessels. Similar fatty acids profiles were obtained from pottery vessels tested by Knappett et al. (2005) and Giorgi et al. (2010). Their investigations suggest that the lipid profiles of the residues have originated from plant oil. Therefore, the occurrence of these lipid profiles in the extracts of the interiors of the four sherds (Jn3, Jn5, Jn13 and TK11) suggests a residue of plant origin, possibly plant oils.

Archaeologically, sherd Jn3 is a body part of a small storage jar/jug and sherd Jn5 is also a body part of large storage jar from Jneneh site. The shapes of these vessels indicate that they could have been used as liquid containers. They were found inside a fireplace. Since the main function of these vessels is most probably liquid container it seems that their context is a secondary. Sherd Jn13, however, is a body of small storage jar, thin and of outside slip. It was uncovered in a small storage room (B1:R3) together with several variant sizes storage jars. It could have been used for storage liquid materials. The occurrence of plant biomarkers, of most likely oil, in the fabrics of the three Iron Age II sherds from the site of Jneneh supports that the vessel Jn3 was used as liquid container and the two vessels Jn5 and jn13 were used as storage jars. Similar results on other sherds from Jneneh site were obtained by Mayyas and Douglas (2015). Storage rooms with large storage jars (pithos) found *in situ* embedded in their floor were uncovered at the site. These results may infer that the Iron Age II settlement at Jneneh was used for domestic purposes, which can be proved from diversity of pottery ware found at the site. Its location in a fertile land at the western bank of Zarqa River, and the existence of a large quantity of storage jars, as well as grinding stones, led to conclusion that the economy of the society of Jneneh was primarily based on agriculture (Mayyas and Douglas, 2015).

Sherd TK11 is also a broken part of Krater from the site of Tell Abu al-Kharaz. It was excavated from Phase IX, Area 9, where an obviously storage and working facility room (Room 8) was found. A considerable number of storage jars leaning against the walls, cooking pots, juglet, other pottery finds, foodstuff such as chickpeas and other cereal grains were found in Room 8, suggesting that the room were used as a multi-purpose facility including a storage area for food and liquids (Fischer, 2012; Fischer and Burge, 2013). Similar kraters, were excavated from another room (Room 2), Phase IX, Area 9, which was used for storage of liquids and foodstuffs, and in which olive pits and dried olive oil remains in one of the kraters were found (Fischer, 2013: 275, 287). It was pointed out that the arable land, with alluvial/colluvial soil around the site, is fertile, and there is evidence of the cultivation of barley, wheat, millet, chickpeas, legumes, grapes and olives during the Iron Age at the site of Tell Abu al-Kharaz (Fischer 2013: 468). Kraters are vessels for mixing, serving and storing liquids/foodstuffs (Fischer, 2013: 407). Therefore, vessel TK11 is an Iron Age IB krater that could have been used for mixing, serving and/or storing liquid materials, most likely olive oil.

Diagnostic biomarkers of the exact source of the lipid preserved in these vessels however were not detected as a result of the degradation of organic residues during burial and post-excavation over time.

3.2. Organic extracts of sherd JoMuS7

GC-MS analysis of the solvent soluble extract of the interior of sherd JoMuS7 also revealed informative lipid constituents (Fig. 4). The sherd preserves a high abundance of monoacylglycerols (MAG-16 and MAG-18) containing 16 and 18 long-chain acyl carbon atoms, moderate abundance of the principal fatty acids ($C_{16:0}$ and $C_{18:0}$), low abundance of $C_{18:1}$, $C_{15:0}$ (branched), $C_{17:0}$ (branched and unbranched), cholesterol, and diacylglycerols (DAG-32, DAG-34 and DAG-36) containing 32, 34 and 36 long-chain acyl carbon atoms. The total amount of the lipids retrieved from the sherd is about 160 µg lipid per one gram of dry pottery powder.

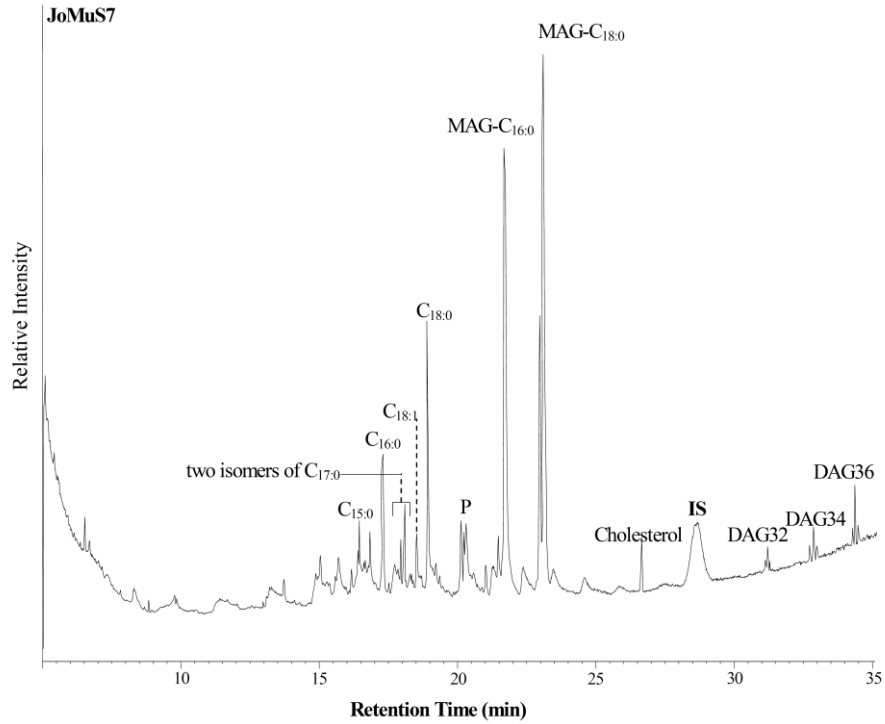


Figure 4. Partial (0-35 min) total-ion-chromatogram (TIC) of the trimethylsilylated total free lipid extract of the absorbed residue from the interior of sherd JoMuS7. ($C_{x:y}$): monocarboxylic fatty acid of carbon chain length x and degree of unsaturation y, (MAG-$C_{x:y}$): monoacylglycerol with $C_{x:y}$ fatty acid moiety, (DAG X): diacylglycerol with carbon chain length x, (P): plasticizer, and (IS) internal standard (n-tetratriacontane; C34).

A high quantity of $C_{18:0}$ is present in animal fat compared to plant materials (Kimpe *et al.* 2004). Degraded animal fat, which is most commonly present in archaeological pottery vessels is characterised by a higher quantity of saturated fatty acids, primarily $C_{18:0}$ and $C_{16:0}$ (Dudd and Evershed, 1998; Evershed et al., 1997b, 1999), and smaller content of monounsaturated $C_{18:1}$ (Kimpe et al., 2004). The extract of sherd JoMuS7 is dominated by free fatty acids, principally $C_{18:0}$ and $C_{16:0}$, with $C_{18:0}$ being the most abundant, while $C_{18:1}$ is present in low abundance, suggesting an animal fat input. In ruminant animal fats, however, $C_{18:1}$ is present with more than one positional isomer of low contents while $C_{18:0}$ is present in high content (Evershed et al., 1999). These positional isomers appear in the fats of ruminant animals, such as sheep and cattle, as a result of biohydrogenation of unsaturated dietary fats in the rumen (Dudd and Evershed 1998; Evershed et al., 1997b, 1999). In the monogastric animals, such as pigs, only a single isomer, Z-9-octadecenoic acid is present (Evershed et al., 1997b). Single isomer of $C_{18:1}$ was detected in the extract of sherd JoMuS7.

Branched-chain fatty acids are present in high abundance in ruminant fats due to bacterial synthesis in the gut, and they are either absent or present in negligible quantity in non-ruminant fats (Dudd et al., 1999). The occurrence of odd-carbon number chain $C_{15:0}$ (branched) and $C_{17:0}$ (branched and unbranched) fatty acids with observable abundance may suggests a ruminant origin (Evershed et al., 1997b; Dudd and Evershed, 1998; Evershed et al., 1999; Mottram et al., 1999; Evershed et al., 2002; Craig et al., 2004, 2005; Giorgi et al., 2010; Regert, 2011; Šoberl et al., 2014; Mayyas et al., 2017). Although they are characteristic of the fats of ruminant animals branched-chain fatty acids could also be present as a result of microbial contamination in vessels (Mottram et al., 1999). Fatty acids with odd-carbon chain lengths have also been found in securely identified porcine fats (Mukherjee et al., 2007).

Mono- and di- acylglycerols, both contain $C_{16:0}$ and $C_{18:0}$ acyl moieties (shown in Figure 4), are known degradation products of the original triacylglycerols, which could have been present in the raw animal fat used in the mother vessel (Regert et al., 1998; Dudd et al., 1999). The occurrence of di- and mono- acylglycerols, with saturated acyl moieties of $C_{18:0}$ and $C_{16:0}$, is in conjunction with the high abundance of the free $C_{18:0}$ and $C_{16:0}$ and is indicative of a degraded animal fat (Copley et al., 2005; Mayyas et al., 2017). The laboratory decay of animal fat supports these results (Evershed et al., 2002). The presence of di- and mono- acylglycerols in addition to the free fatty acids in these extracts attests to the degradation of the original triacylglycerols during

use and/or during subsequent burial at the archaeological site. The animal fat biomarker, cholesterol, detected in the extracts also supports the animal origin (Kimpe et al., 2002, 2004; Salvini et al., 2008; Giorgi et al., 2010, Mayyas et al., 2017).

The absence of squalene in the extracts of the archaeological sherd and the absence of both squalene and cholesterol in the extract of the modern sherd exclude contamination from handling (Knappett et al., 2005; Marangou and Stern, 2009; Heron et al., 2010). Thermal biomarkers of mid-chain ketones were not detected in the extract indicating that the pot might not exposed to temperatures above 300°C (Reber and Hart, 2008). Plant biomarkers, including sterols and unsaturated fatty acids, excluding $C_{18:1}$, were also not detected in this extract.

The common occurrence of these lipid constituents in sherd JoMuS7 indicates that the animal fat is the source. But it was not possible to identify whether the lipid source is ruminant or non-ruminant. Both sources are suggested for this residue. Biomarkers of animal fats preserved in archaeological pottery vessels were detected and reported by many researchers, such as Evershed et al. (1999), Kimpe et al. (2002), Regert et al. (2003b), Spangenberg et al. (2006), Giorgi et al. (2010), Gerbault et al. (2013) and Mayyas et al. (2017). It should be emphasised that degradation of organic residues in archaeological vessels during use/or burial is one of the most important issues that must be considered in such study and in particular the degradation of $C_{18:1}$ fatty acid which was discussed and reported by many researchers (Mayyas et al., 2017).

Although the input of organic residue from plant materials in JoMuS7 sherd can not be excluded the results of this study provide evidence that an animal fat was most probably used inside the mother pot of this sherd, possibly for cooking activity.

Archaeologically, cooking pots were excavated from Sahab from the Iron Age contexts (Ibrahim, 2016b: 199, 204, 209, 213) but this sherd were not investigated anywhere (Personal communication with Ibrahim on 4th Nov 2017). Therefore, no archaeological data were published concerning this sherd. However, sherd JoMuS7 is a thick body/base part of cooking pot with black burning marker on the external surface indicating that the mother vessel was used for cooking/processing activity. No secondary fire during the Iron Age was noted by Ibrahim (2016b: 177-263) supporting that the original vessel most probably was used for cooking/processing activity. This result can also enforced with the archaeological finding of large quantity of bones of domestic animals, mainly ruminant, such as goats, sheep and cattle found at the site of Sahab during the Iron Age (Ibrahim, 1972 1974, 1975). In addition, animal fat, potentially of ruminant origin, was detected in Iron Age lamps from the site of Sahab (Mayyas et al., 2017).

3.3. Organic extracts of other sherds

GC-MS analysis of the other seven pottery sherds (described in Table 1 and shown in Table 2) showed very low abundances of some lipid constituents (Table 4) that can not be attributed to any significant source.

Table 4. Organic constituents retrieved via simple extraction from the interiors of the other seven pottery sherds

#	*Sherd Code*	*Organic constituents obtained via simple extraction of the interiors*
1.	JoMuS2	$C_{12:0}$, $C_{16:0}$, $C_{18:0}$, 10-OH-$C_{18:0}$
2.	JoMuS9	$C_{9:0}$, $C_{12:0}$, $C_{16:0}$, $C_{18:0}$, $C_{20:0}$, MAG-$C_{16:0}$
3.	JoMuS14	$C_{12:0}$, $C_{16:0}$, $C_{18:0}$, $C_{18:1}$, $C_{20:0}$, MAG-$C_{16:0}$, MAG-$C_{18:0}$
4.	TK6	$C_{14:0}$, $C_{16:0}$, $C_{18:0}$, $C_{18:1}$, $C_{20:0}$, $C_{22:0}$, AL_{16}, MAG-$C_{16:0}$, MAG-$C_{18:0}$, Other hydroxyl fatty acids
5.	TK9	$C_{16:0}$, MAG-$C_{16:0}$, MAG-$C_{18:0}$
6.	TK10	$C_{14:0}$, $C_{16:0}$, $C_{18:0}$, $C_{18:1}$, $C_{20:0}$, $C_{22:0}$, AL_{16}, MAG-$C_{14:0}$, MAG-$C_{16:0}$, MAG-$C_{18:0}$, Other hydroxyl fatty acids
7.	Jn7	$C_{12:0}$, $C_{14:0}$, $C_{16:0}$, $C_{18:0}$, AL_{16}, MAG-$C_{16:0}$, MAG-$C_{18:0}$

$C_{x:y}$: monocarboxylic fatty acid of carbon chain length x and degree of unsaturation y, AL_x: *n*-alcohol of carbon chain length x, OH: hydroxyl, and MAG-$C_{x:y}$: monoacylglycerol of $C_{x:y}$ fatty acid moiety.

The low abundance of these constituents could be attributed to high level of degradation during burial and post-excavation over time or they could be contaminats from burial environment compared with those detected in the other five sherds.

The results of the analysis of organic residues, retrieved via conventional solvent extraction, showed that plant oil, very likely olive oil was introduced into the interiors of four Iron Age vessels from the sites of Jneneh and Tell Abu al-Kharaz and animal fat was introduced into the interior of one Iron Age vessel from the site of Sahab. These five vessels however, can be classified according to their use as follows:

1- Container (vessel Jn3) and storage jars (vessels Jn5 and Jn13) from Jneneh site were used for holding and storing plant liquid material, most likely olive oil, during the Iron Age II.

2- Krater (vessel TK11) from Tell Abu al-Kharaz site was used for mixing, serving and/or storing plant liquid material, most likely olive oil, during the Iron Age IB.
3- Cooking/processing pot (vessel JoMuS7) from Sahab site was used for cooking/processing animal fat during the Iron Age.

The four vessels (represented by Jn3, Jn5, Jn13 and TK11 sherds) have slip while the only vessel (represented by JoMuS7 sherd) has not. Sherd JoMuS7 is thick and has black burning marker on the exterior supporting that vessel JoMuS7 was exposed to fire possibly during cooking/processing.

Evidences of the availability and exploitation of plant products, such as oils, and animal fats during the Iron Age were discussed above. For Jneneh site, in addition to the archaeological evidences, the chemical study of organic residues preserved in Iron Age II pottery vessels from Jneneh provided comparable data on the possible use of plant oil, such as olive oil, in vessels excavated at Jneneh site (Mayyas and Douglas, 2015). For Tell Abu al-Kharaz site, the land around the site is characterized by an arable land with alluvial/colluvial soil; therefore, it is a fertile land. Evidences on the availability of major cereal crops including barley and wheat and cultivation of millet and chickpeas, legumes, grapes and olives during the Iron Age at the site of Tell Abu al-Kharaz are present (Fischer, 2013: 468). For Sahab site, it was extensively settled and the settlement had reached its largest extension during the Early Iron Age (Ibrahim, 1972, 1978). This supports the availability of large quantity of bones of domestic animals at the site of Sahab during the Iron Age (Ibrahim, 1972 1974, 1975). In addition, the chemical data obtained by Mayyas et al. (2017) provided further evidence of the on the availability of domestic animals and most likely use of their products at the site of Sahab during the Iron Age.

However, future work should be supported with compound specific carbon isotope analysis (^{13}C analysis).

4. CONCLUSIONS

This research project aimed at analysing organic residues preserved in the fabric of twelve (two Bronze Age and ten Iron Age) pottery sherds from three sites. Based on the biomarkers detected in these sherds, it was possible to generate useful data from five of these sherds. The results inform that plant material, most likely olive oil, has been preserved in the fabrics of four sherds (Jn3, Jn5 and Jn13 from the site of Jneneh and TK11 from the site of Tell Abu a-Kharaz) and animal fat has been preserved in the fabric of one sherd (JoMuS7 from the site of Sahab). These sherds belong to vessels described in Table 1 and shown in Table 2. These five vessels could have been used during the Iron Age as: container (Jn3), storage jars (Jn5 and Jn13), mixing, serving and/or storing krater (TK11) for plant liquid material, very likely olive oil, and cooking pot (JoMuS7) for cooking/processing animal fat. These results are in conjunction with the archaeological evidences on the cultivation of plant materials and domestication and exploitation of animal fat during the Iron Age in Jordan. Biomarkers from the other seven sherds however are not diagnostics because they have diminished abundances that can not infer any significant source of organic residues.

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