# Insights into the use of late Halaf vessels. Organic residues in pottery from Tell Halula (Syria)

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*Abstract*

Halaf pottery is one of the core elements defining Late Neolithic Middle Eastern societies. This article presents the preliminary results of organic residue analyses performed on a small set of Late Halaf painted and plain Fine wares from Sector 49 in Tell Halula (Syria). Data obtained from lipids embedded in the clay matrix suggests the existence of type-content variation possibly related to food display and commensality practices.

*Keywords*

Late Halaf, Organic Residue Analysis, Pottery use, Acidified Methanol extraction, Tell Halula

## 1. Introduction and objectives

At the onset of the sixth millennium before the common era (BCE), innovations in pottery technology and changes in the production processes introduced what archaeologists have named the Halaf traditions.[1] Except for large settlements with continuous occupation sequences, this Late Neolithic society inhabited small sites with short and intermittent occupations with sometimes an absence of built structures, suggesting the shelters were of perishable materials. This picture supports a shifting settlement pattern coherent with communities that relied on animal husbandry to accumulate wealth and status, and control production relations.[2] The recovery of clay sealings suggests the existence of a certain degree of private property structured in an egalitarian social order[3] when burial treatment is considered. Amidst this situation, Halaf pottery was extensively decorated and produced per distinct stylistic conventions, which could have symbolised group membership and many small communities participating in a wider cultural framework. Therefore, it has been considered that many painted vessels would have been used for the display and consumption of foods and drinks, while others would have been involved in storage and food preparation.[4] Consequently, culinary practices resulting in the consumption of products from animal husbandry in feasting contexts could have played a significant role in the production and reproduction of the Halaf social structure.

On the study of pottery use, several pieces of evidence such as use-wear or the existence of macroscopic and microscopic remains (phytoliths and pollen) attached to the inner surface of the pot can offer valuable information. At the molecular scale, the analysis of hydrophobic organic matter (lipids, resins and waxes) preserved in clay pores (putative Organic Residue Analysis, ORA) is another one of the complementary tools archaeologists apply to understand pottery use. Since the beginnings of this analytical technique pioneered by Condamin et Al.,[5] methods for the detection of fats preserved inside pottery and interest in this area of investigation have been steadily growing. When taking only into account sites from the Middle East, less than 10 papers on organic residues in pottery have been published before 2006.[6] Nevertheless, just between 2008 and 2009 at least 7 new studies and 1 Ph.D. have been written[7] and, from that date onwards, the slow but steady methodological improvement[8] has facilitated at least 19 more papers published exploring the organic remains preserved inside pottery.[9]

One striking characteristic shared by the over 30 studies published to date is the scarcity of vessels which reveal significant quantities of lipids. As an example, an extensive project by Evershed and colleagues comprising 2225 pottery vessels from 23 sites in south-eastern Europe and the Middle least revealed that only 12% of them contained lipidic residues.[10] Such a figure is significantly lower than the results obtained in other regions of the world, such as the western Mediterranean or northern Europe.[11] Low preservation and degradation factors can be related to pottery age, choice of clay and the seasonal variations in temperature and precipitation that characterise the climate in the Middle East.[12] Alternatively, residues might not be originally present, thus suggesting that vessels did not participate in activities incorporating lipids in the clay matrix. But, were prehistoric Near Eastern societies less dependent on organic products such as fats, resins and waxes? Is a high recovery of organic residues implausible in a period and region where environmental, temporal and technological factors might not be favourable to their preservation?

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1 Nieuwenhuyse 2009.

2 Akkermans, Schwartz 2003.

3 Frangipane 2007; Gómez-Bach, Cruells, Molist 2018.

4 Nieuwenhuyse 2013; Hopwood, Mitra 2012.

5 Condamin et Al. 1976.

6 Copley et Al. 2005a; Shimoyama, Ichikawa 2000; Kimpe et Al. 2004.

7 Tükerkul-Biyik, Özbal 2008; Mathe et Al. 2009; Namdar et Al. 2009.

8 Correa-Ascencio, Evershed 2014.

9 ex: Özbal et Al. 2011; Mayyas et Al. 2013; Chovanec et Al. 2015; Steele, Stern 2017.

10 Evershed et Al. 2008b; Thissen et Al. 2010.

11 Breu 2017.

12 Gregg, Slater 2010.

Taking these limitations into account, organic residue analyses from the Late Halaf layers in Tell Halula aim at providing, for the first time, insights into the products possibly cooked, displayed and consumed in Late Halaf Fine wares. To overcome preservation difficulties, more aggressive extraction methods developed by Correa-Ascencio and Evershed[13] and successfully used in pre-Halaf pottery[14] will be applied to a small selection of sherds from Sector 49 in Tell Halula.

## 2. Archaeological context and sampling strategy

Tell Halula is a site in the Syrian Euphrates valley. It conceals a settlement presenting a wide chronology that extends from the first phases of the pre-ceramic Neolithic (PPNB) to the ceramic periods (Halaf), in which the consolidation of the first agro-pastoral communities can be studied.[15]

Samples for this study have been collected from Sector 49, which comprises a wide excavation space featuring an open-air area with exterior floors associated with negative structures. Habitats are constructed of mudbrick and stone and have had four occupation phases dated to the Late Halaf period.[16] The chronological adscription and the technological strategies involved in the ceramic production (more than 3700 ceramic minimum number of individuals) detected in this context have been published elsewhere.[17] These studies reveal an assemblage including small containers with features intended for the individual consumption of liquids or semi-solid foods and medium/large pieces ideal for storage and transport of liquid and solid provisions. Dish covers, collar-rimmed jars (with an inside depression in the neck) and large bowls, amongst others, all show cooking practices requiring either covered[18] or uncovered pots, such as boiling and stewing. Given that previous research suggests fats in aqueous solutions accumulate in the upper part of the vessel, for this study, rims (n=6), necks (n=1), upper body wall sherds (n=4) and one base were sampled.[19]

A sample of 12 ceramic vessels from Sector 49 including straight walled bowls (n=2), closed bowls (n=2), hemispherical bowls with a flat base (n=4), hole-mouth bowls (n=1), cream bowls (n=2) and pots (n=1) was selected for this study (see Table 1). Although other types such as Plant Tempered ware and Mineral Tempered ware (both coarse wares) were present in the assemblage, monochrome painted Late Halaf Fine ware (n=9) and Plain Fine ware (n=3) were exclusively chosen. Complementary with previous research,[20] this approach intends to maximise data gained from vessels related to said Halaf feasting and commensality activities.

## 3. Materials and methods

Lipids trapped in the clay pores within the ceramic fabric were extracted following the technique developed by Correa-Ascencio and Evershed.[21] After discarding the vessel's inner surface, 1g of ceramic powder was sampled using an electric drill with a tungsten bit. After adding 4ml of methanol to the ceramic powder, the mixture was subjected to an ultrasonic bath for 15 minutes and acidified with 0.8ml of concentrated sulphuric acid. The mixture was heated at 70° C for 4 hours and then left to cool. Lipids were extracted from the acidified methanol mixture with 2ml of hexane (x3). The hexane extracts were desulphurised with copper pellets and then vacuum dried. Finally, the samples were dissolved again in 100ml of isooctane and transferred to gas-chromatography (GC) vials, which had been spiked with 10mg of n-tetratriacontane as the internal standard.

To analyse each sample, 1μl was injected in splitless mode to a 782A Agilent Gas Chromatograph coupled to a Flame Ionisation Detector (FID) and

13 Correa-Ascencio, Evershed 2014.

14 Breu et Al. 2018.

15 Molist, Vicente 2013.

16 Gómez-Bach 2011.

17 Gómez, Cruells, Molist 2014; Gómez- Bach 2013; Gómez-Bach et Al. 2012.

18 Gómez-Bach 2013.

19 Charters et Al. 1993.

20 Nieuwenhuyse et Al. 2015; Hopwood, Mitra 2012.

21 Correa-Ascencio, Evershed 2014.

Table 1 – Ceramic and biomolecular characteristics of the analysed sherds

| Id | Sherd shape | Ware type | TLE[a] | P/S[b] | Vessel shape | Molecules | Residue interpretation |
|---|---|---|---|---|---|---|---|
| 7 | Base | Plain Fine Ware | 98.2 | 2.01 | Not determined | FFA: $C_8$-$C_{26}$; Branched FA: C15, C17; Diacids: C9 | Degraded animal fat |
| 10 | Rim | Plain Fine Ware | 34.95 | 1.3 | Straight walled bowl | FFA: $C_9$-$C_{28}$; Diacids: C9 | Degraded animal fat |
| 12 | Rim | Painted Fine Ware | 10.68 | 1.1 | Hemisferic flat bowl | FFA: $C_9$-$C_{18}$; Alkanes; Pthalate plasticisers | Degraded animal fat |
| 13 | Neck | Painted Fine Ware | 7.3 | 1.3 | Pot | FFA: $C_9$-$C_{18}$; Alkanes; Pthalate plasticisers | Degraded animal fat |
| 16 | Rim | Painted Fine Ware | 3.79 | 1.17 | Hole-mouth bowl | FFA $C_{12}$-$C_{18}$; Alkanes; Pthalate plasticisers | Non significant residue |
| 18 | Wall | Painted Fine Ware | 6.2 | 1.7 | Cream bowl | FFA: $C_{12}$-$C_{18}$; Alkanes | Degraded animal fat |
| 20 | Wall | Painted Fine Ware | 6.68 | 1.04 | Closed bowl | FFA: $C_{14}$-$C_{18}$; Alkanes | Degraded animal fat |
| 25 | Rim | Painted Fine Ware | 4.73 | 1.62 | Hemisferic flat bowl | FFA: $C_9$-$C_{18}$; Pthalate plasticisers | Non significant residue |
| 32 | Rim | Plain Fine Ware | 6.6 | 2.31 | Straight walled bowl | FFA: $C_9$-$C_{18}$; Alkanes; Phtalate plasticisers | Degraded animal fat |
| 35 | Wall | Painted Fine Ware | 5.41 | 1.55 | Cream bowl | FFA: $C_8$-$C_{24}$; Alkanes; Phtalate plasticisers | Degraded animal fat |
| 46 | Wall | Painted Fine Ware | 8.32 | 1.49 | Hemisferic flat bowl | FFA: $C_9$-$C_{26}$; Alkanes, Pthalate plasticisers | Degraded animal fat |
| 52 | Rim | Painted Fine Ware | 8.1 | 0.92 | Hemisferic flat bowl | FFA: $C_{12}$-$C_{18}$; Alkanes; Phthalate plasticisers | Degraded animal fat |

[a] Total Lipid Extract (µg·g-1)
[b] Palmitic acid/Stearic acid relative abundance ratio

eluted through an HP-1 capillary column (60 m length, 250 µm internal diameter, 0.25 µm film thickness) using hydrogen as the carrier gas. The oven temperature was initially set at 50ºC for 1 minute and then increased by 6ºC/min to 320ºC, where it stayed for 20 minutes.

When the compounds were not identifiable by their retention time, samples were then analysed by mass spectrometry. Consequently, 1µl was injected into an Agilent 7890A GC coupled to an Agilent 5975C Mass Spectrometer. The GC was fitted with a DB-5MS column measuring 30m length x 250µm internal diameter x 0.25µm film thickness. The GC injector was operated in splitless mode and helium was used as the carrier gas. The oven temperature was set at 50ºC for 2 minutes and then was increased by 15ºC/min until reaching 170ºC. Next, the temperature was further increased by 5ºC/min until reaching 320ºC and maintained for 6 minutes. The Mass Spectrometer was run in electron impact mode and masses were acquired in full scan mode between 50 to 800 m/z. Detected molecules were identified using the NIST 4.0 library.

## 4. Results

Out of the 12 samples, 83% contained lipid residues above the 5µg/g threshold commonly used to detect archaeologically significant amounts of residues.[22] Developed as a highly aggressive procedure able to retrieve lipids that hitherto were strongly bound with the clay matrix, the acidified methanol extraction[23] might help explain the high success rate compared to earlier studies. Previously, this technique had been successfully applied to pre-Halaf pottery[24] and, for the Late Neolithic in the Middle East, it has resulted in a 50% increase in the total quantity of residues recovered.

In terms of the recovered fats, all 12 potsherds contained octadecanoic acid (C18:0) and palmitic acid (C16:0) with a series of other minor fatty acids such as tetradecanoic acid (C14:0), pentadecanoic acid (C15:0), heptadecanoic acid (C17:0)

[22] Nieuwenhuyse et Al. 2015.

[23] Correa-Ascencio, Evershed 2014.

[24] Breu et Al. 2018.

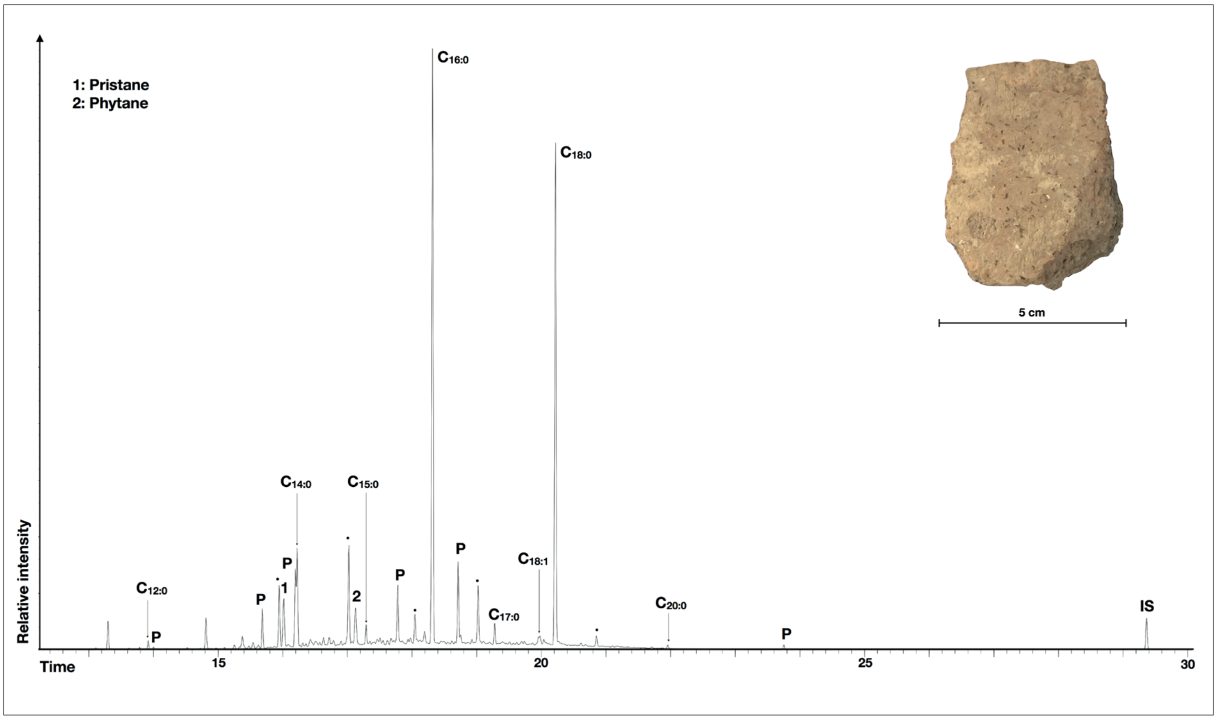


Figure 1
Chromatogram showing a common degraded animal fat resulting from the hydrolysis of triacylglycerols; P: phthalate plasticisers, IS: Internal Standard

and eicosanoic acid (C20:0). In consequence, although there is a certain variability between samples, it is not clear whether this can be explained by differences in vessel content. Long-chain ketones (16-hentriacontanone, 16-tritriacontanone and 18-pentatriacontanone)[25] and ω-(o-alkylphenyl) alcanoic acids[26] indicate thermal-specific degradation related to specific cooking techniques. Moreover, a range of specific isoprenoid acids (pristanic acid and phytanic acid)[27] are regarded as biomarkers which can suggest fish-related oils. Given that the Euphrates river is less than 2km away from the site, freshwater resources could have been easily accessible by Tell Halula's inhabitants. Nevertheless, none of these biomarkers were detected, suggesting either they were never in contact with the analysed sherds, or the difficult preservation conditions in the Middle East made their detection difficult. Furthermore, the ratio between the abundance of Palmitic acid versus the abundance of Stearic acid has been used to interpret the possible origins of the recovered lipidic residues. In the case of the sherds analysed from Tell Halula, samples 18 and 7 presented Palmitic/Stearic ratios (P/S) higher than 1 (1.7 and 2.1 respectively) and an almost equal amount of oleic acid with octadecanoic acid (fig. 1). Sample 32 presented the highest P/S ratio in the assemblage (2.3) with barely any traces of oleic acid. Although similar profiles have been assigned to fats of plant origin,[28] in this case, the inner variability of animal and dairy fats across different seasons or foraging strategies[29] could still explain the obtained octadecanoic acid relative quantities.

Therefore, the detected fatty acid profiles can be interpreted as being most coherent with hydro-

25 Evershed et Al. 1995.

26 Hansel et Al. 2004.

27 Evershed et Al. 2008a.

28 Dunne et Al. 2016; Copley et Al. 2005b.

29 Chilliard et Al. 2007; Liu et Al. 2017.

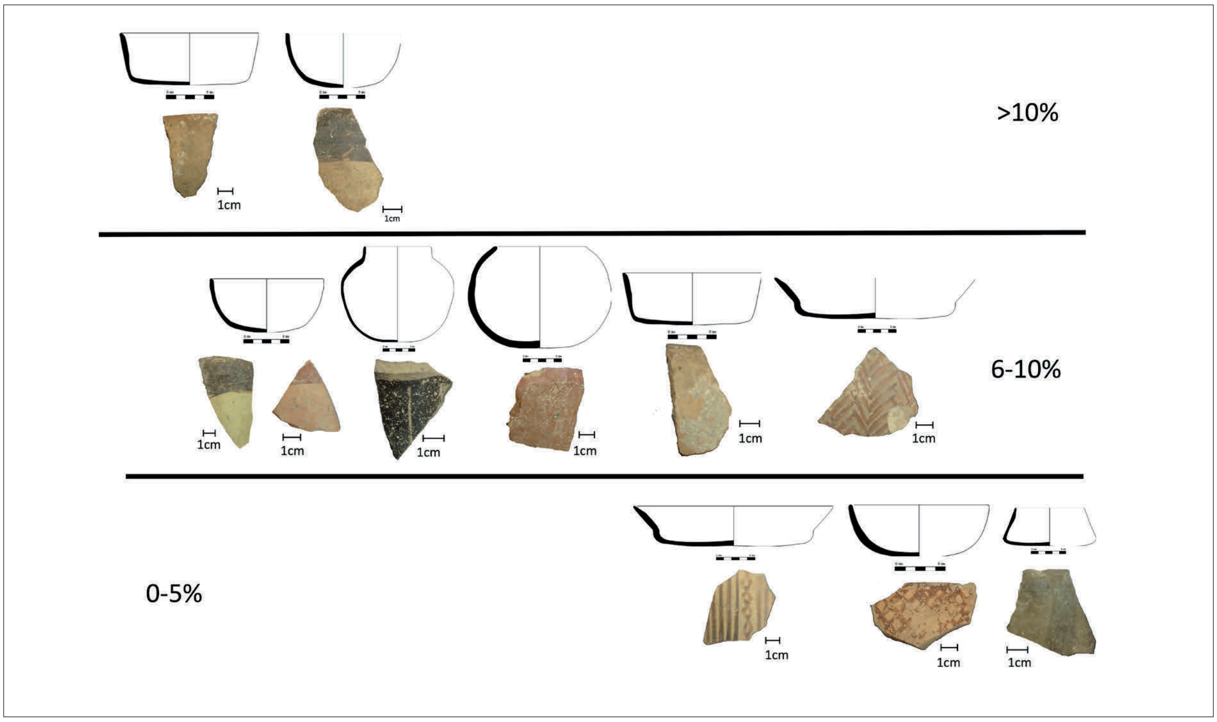


Figure 2
Photos and drawings from the analysed sherds ordered by the relative amount of lipids recovered

lysed triacylglycerols from animal fats. In 7 cases, the detection of phthalate esters, molecules associated with modern plastics, is coherent with an assemblage excavated in 2007 and until now stored in plastic bags. No other molecules attributed to modern contamination, such as cholesterol and squalene, were found. Further investigation of these fats for the presence of dairy products is conditional on the acquisition of compound-specific isotopic data from the palmitic and stearic fatty acids.

Although the small biomolecular variation seems to suggest content homogeneity between vessels, certain potsherds present infrequently high and low quantities of fatty acids. Quantification of the amount of archaeological residue recovered after analysis is a highly complex task carrying a significant amount of uncertainty. The presence of phthalate esters and co-elusions from other modern contaminats, and the possible unaccounted losses during laboratory sample preparation, can significantly increase the margin of error associated with the total lipid extract (TLE) value. Consequently, archaeological interpretations based on the TLE must take these limitations into account. In the attempt to minimize uncertainties, TLE absolute values were not used to report lipid quantities in this study. Instead, samples from Tell Halula were classified into 3 groups related to the relative amount of lipids recovered as a percentage the sum of the TLEs from all 12 analysed samples (the assemblage's total). The first category incorporated samples yielding 0 to 5% of the total amount of lipids in the assemblage, samples with 6 to 10% were placed in the second group and, finally, the third group integrated samples containing more than 10% of the total amount recovered. Given that every compared sherd was sampled in a similar position (the upper body close to the rim) it can be assumed that residues would have been generated as a result of similar pottery uses.

One possibility that could describe why different quantities of lipids would be found in the analysed assemblage could be the vessel's involvement in repeated food preparation episodes. The vessel shapes associated with each group are presented in fig 2. Cream bowls and the Hole-Mouth bowl are associated with the lowest amounts of lipids, while one of the two analysed straight walled bowls alone yielded 34% of the total quantity. Other shapes such as the pot and the closed bowl similarly presented significant amounts of residues. As shown in previous studies,[30] the incorporation of lipids into the ceramic matrix is improved when a certain amount of heat helps mobilise them outside the actual food. Additionally, other factors such as boiling water and/or other fats in liquid form may promote the transfer into the clay matrix. Therefore, it could be speculated that non-cooking pottery types would less frequently encounter the ideal conditions for residue incorporation, thus absorbing lower amounts of fats comparatively. Nevertheless, this pattern associating tableware with fewer lipid quantities needs to be carefully evaluated given that this case study only analysed 12 samples out of the thousands that were excavated in the site.

## 5. Discussion and conclusions

As outlined by the research objectives in section 1, it can be concluded that the acidified methanol extraction has successfully recovered significant amounts of lipids in 83% of the analysed pottery. Nonetheless, the use of this aggressive extraction must be cautiously evaluated given that it imposes several limitations to the interpretability of the results when the preservation of organic matter is good. In the case of the Middle East, previous extensive research has already demonstrated that recovered organic matter is scarce and poorly preserved. Therefore, chemical reactions such as hydrolysis generated by sulphuric acid only minimally compromise the integrity of the sample. This is because the organic matter has already been fully hydrolysed by the passage of thousands of years under an environment with high temperature and humidity fluctuations. In the case of Late Halaf analyses from Tell Halula, it seems that this choice of method has facilitated the acquisition of interpretable results.

Although many studies have investigated the possible uses of pre-Halaf and early Halaf pottery with great success,[31] research on organic residue analyses from the second half of the sixth millennium BCE has only been practiced in archaeological sites located at the edges of the area of the Halaf tradition (the south and north of the Taurus mountain range, the north of the Jesireh and the plain ending in the Zagros foothills). On the Mediterranean coast (Israel and Jordan), the sites of Newe Yam, Tabaqat al-Bûma, al-Basatîn[32] and Ein Zipori[33] have offered insights into the uses of ceramic types such as cups, blows and jars with various degrees of success and, in northern Iran, analyses from Hajji firuz Tepe might suggest resinated wine was already consumed at this early age.[34]

Specifically, 14 out of the 80 samples analysed in the aforementioned sites[35] yielded mainly animal fats. Where isotopic analyses were performed (al-Basatîn), results suggested the presence of mixtures of non-ruminant (probably porcine) and ruminant fats, although the researchers did not rule out the presence of dairy products.[36] Furthermore, an amphoriskos and two jars from the Late Neolithic/Early Chalcolithic layers in Ein Zipori presented residues interpreted as originating from olive oil and other undetermined plant oils.[37] Many of the sherds analysed from these sites are associated with the Wadi Rabah cultural group, which is contemporary with the Halaf phenomenon.

In Turkey, residue analyses were practiced on pottery from Çayönü found in the layers directly above the PPN occupations and reported as chalcolithic[38]. These represent the closest parallels to the analyses presented in this paper. Out of 18 sherds, six contained lipid residues which were interpreted as

[30] Charters et al. 1997; Evershed 2008; Hammann, Cramp 2018.

[31] E.g.: Nieuwenhuyse et al. 2015; Hendy et al. 2018; Evershed et al. 2008b; Gregg 2009.

[32] Gregg 2009.

[33] Namdar et al. 2015.

[34] McGovern et al. 1996.

[35] Samples were extracted using conventional solvent extractions, Evershed et al. 1990 and microwave-assisted extraction protocols, Gregg, Slater 2010.

[36] Gregg et al. 2009.

[37] Namdar et al. 2015.

[38] Gregg 2009.

being derived from plants and, in one case, animal fats.

Therefore, for the second half of the sixth millennium BCE, research so far suggests the combined presence of both animal (60%) and plant fats (40%). In this matter, Tell Halula stands out (similarly to the results from al-Basatîn) for only revealing animal fats. Plant residues are thus far absent possibly because of two factors.

Firstly, the quantity of fats in plants is significantly lower than fats in animals, which suggests a single use of a pot to cook an animal product would produce a comparatively higher lipid signal. Therefore, it should not be ruled out that biochemical signals from plant fats were present in Tell Halula's assemblage but hidden below strong animal fat signals. A hint supporting this line of thought can be found in some samples where the abundance of Palmitic acid is two times higher than Stearic acid.

The second factor possibly preventing the detection of plant fats is their weak resistance to degradation. Mono and polyunsaturated fats are the major compounds in plant triacylglycerols and are also more easily oxidised than saturated fatty acids, which are significantly more abundant in animal fats. In consequence, the apparent absence of plant biomarkers in such a small sample size (12 sherds) should not be interpreted as strong evidence for the absence of such products in the Halaf cooking traditions given that previous research succeeded in detecting them.[39]

Concerning possible food management practices, samples extracted from rim sherds on straight walled bowls (samples 10 and 32) and hemispheric flat bowls (samples 12 and 52) present archaeologically significant amounts of lipids in a part of the vessel not usually exposed to foodstuffs. One explanation for this phenomenon is that these vessels were repetitively filled to the top with a fat-rich food. Sauces and meat juices can be the components with the highest amount of fats, but these elements are usually liquid or viscous and they would, therefore, tend to concentrate at the lower part of the vessel body. This explanation seems to fit well with sample 7 (a base with the highest detected amount of lipids) but does not seem to work for sample 10 (a rim with the second highest amount of lipids recovered). Alternatively, it must be considered that animal fats are lighter than and not soluble in water. Therefore, in an aqueous solution, they will tend to be in the upper part of the vessel body. Experimental studies[40] based on this premise succeeded in correlating high relative amounts of fats in the rim with boiling episodes. Consequently, vessels 10, 12, 32 and 52 could have been involved in the production and consumption of boiled foods and broths, which both involve aqueous solutions, but the former should be considered highly unlikely given that painted and plain fine ware is most generally associated with food consumption rather than food preparation. More work is needed to understand the likeliness of each possibility.

When considering that vessel sizes are small (as depicted in Table 1), it seems that the presence of animal fats in them, the fact that a certain degree of heat would have been applied, and the quantity of residues found close to the rim suggests that food could have been prepared as well as consumed in relatively small portions. This interpretation suggests a possible scenario where food is used to negotiate and compete for individual positions of leadership[41] and it implies that, amongst many other possible foodstuffs, animal products were of significant importance.

In conclusion, the organic residue analyses performed on 12 Late Halaf pottery sherds from Sector 49 in Tell Halula have revealed that pottery was involved in the management and consumption of products with some animal fat in their composition. A comparison with the knowledge gathered so far on the contents in pottery from the late sixth millennium BCE in the Middle East has helped confirm the use of certain pottery shapes as tableware and suggest Late Halaf pottery in Tell Halula took part in the transformation, display and consumption of animal products.

39 Hopwood, Mitra 2012.

40 Charters et Al. 1993.

41 Nieuwenhuyse 2013.

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