ISSN-1996-918X

Pak. J. Anal. Environ. Chem. Vol. 27, No. 1 (2026) 489707c

http://doi.org/10.21743/pjaec/2026/489707c

Organic Residue Analysis of Late Bronze Age Ceramics from Aşağıseyit Höyük by GC-MS, a Key Settlement in Western Anatolia

Erim Konakçı[1] and İsmail Tarhan[2*]

[1]*İzmir Demokrasi University, Faculty of Science and Letters, Department of Archeology, İzmir, Turkey.*

[2]*Selçuk University, Faculty of Science, Department of Biochemistry, Konya, Turkey.*

*Corresponding author Email:ismtarhan@gmail.com

Received 15 September 2025, Revised 12 December 2025, Accepted 14 January 2026

Academic Editor: Najma Memon

Abstract

In the present study, gas chromatography–mass spectrometry (GC–MS) was used to investigate organic residues (ORs) preserved in the sherds recovered from the Late Bronze Age layers of Aşağıseyit Höyük, one of the important ancient settlements of Western Anatolia, in order to identify the types of food processed in these vessels. According to the results, 8 out of the 50 sherds analyzed gave interpretable results according to their OR compositions. The sherds of E-18, E-24, E-30, and E-36 may have been used in processes involving the carcass meat of a ruminant animal. The sherds of E-38, E-39, and E-41 may also contain a ruminant animal or a dairy product. The ORs obtained from E-38 and E-39 of these sherds also indicate plant oils. These results show that the relevant vessels may have been used for cooking or storing animal products together with plant oils. It can be said that the OR in sample E-43 is predominantly a residue of plant oil and that this vessel may have been used mostly for materials containing plant oils.

Keywords: Aşağıseyit Höyük, GC-MS, Late Bronze Age, Organic Residue, Western Anatolia.

Introduction

One of the fundamental pillars of the human fight for survival on a social, biological, and individual level is nutrition. Determining dietary patterns is therefore crucial to understanding human cultural history. Potteries, which are commonly discovered in archaeological excavation sites, can reveal crucial information on the dietary practices, economic systems, customs, and cultures of the societies. Important details about the daily activities, commerce, and rituals of these communities can be uncovered using the right analytical techniques to analyze the organic residues (ORs) that can be found on the surface or inside these vessels [1, 2].

The organic molecules in food interact with the pottery in which they are cooked, transported, and stored, and are absorbed in the pores on the ceramic walls. The appropriate ORs can be retained for thousands of years thanks to the inorganic porous matrix of unglazed ceramic containers, which can offer a protective environment for the biomolecules and breakdown products of absorbed organic chemicals [3, 4]. By allowing ORs to penetrate deeply into unglazed ceramic vessels, their porous structure offers robust defense against microbial attacks and advanced decomposition that could arise from water leaks during the burial process [5, 6].

Aşağıseyit Höyük is located approximately 1.5 km southeast of the Aşağıseyit neighborhood within the borders of Çal district, Denizli province (Fig. 1a). The mound is situated at an elevation of 828 meters above sea level, on a natural hill that extends northwest-southeast. The main settlement area of the mound covers approximately 6 hectares. During the surface surveys conducted in the lowland section of the Upper Menderes Basin, Aşağıseyit Höyük was discovered (Fig. 1b). Subsequent investigations were carried out on a survey project in the plateau and mountainous regions of the Upper Menderes Basin [7, 8], and archaeological excavations started at the site in 2021.

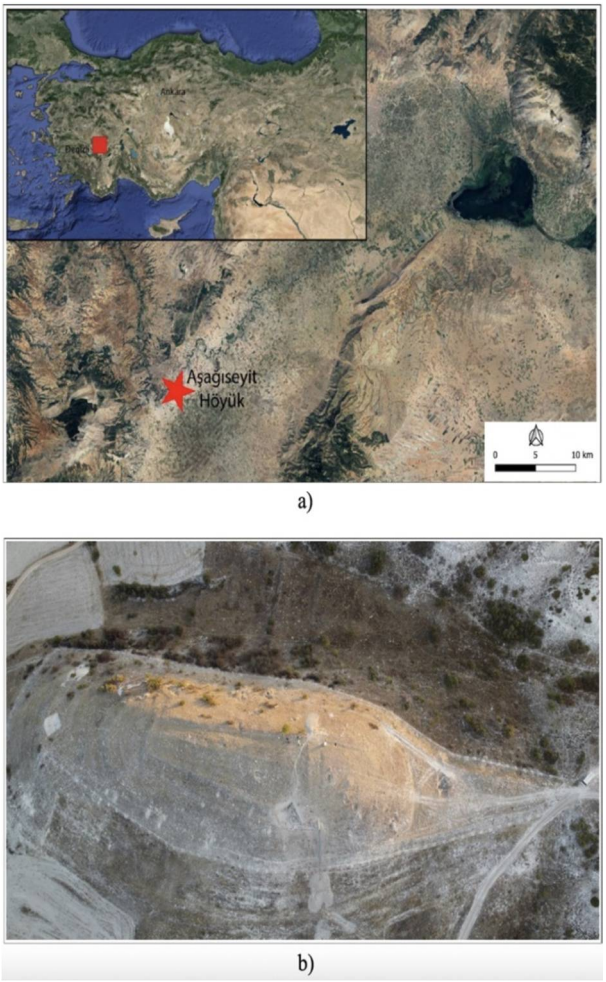

Figure 1. **The location of Aşağıseyit Höyük, a) Geographical location, b) The aerial view**

In the excavations carried out so far in Aşağıseyit Höyük, layers dating to Roman, Hellenistic, Late Bronze Age, and Early Bronze Age have been unearthed (Table 1). In addition, considering the data obtained from surface surveys [8], the existence of the following layers within the settlement can be suggested. The areas examined within the Late Bronze Age layers show that the spaces were residential, and the pottery identified in four different architectural phases belonging to these layers constitutes the focus of the presented study.

Table 1. **The Stratigraphy of Aşağıseyit Höyük.**

Layer		Period
Layer I		Roman Period (5th AD)
Layer II		Hellenistic
A		
B		
Layer III		Iron Age (8-6th BC)
Layer IV		Late Bronze Age
A		
B		
C		
D		
I.	Layer V	Middle Bronze Age
II.	Layer VI	Early Bronze Age III
III.	Layer VII	Early Bronze Age II
A		
B		
C		
Layer VIII		Early Bronze Age
Layer IX		Late Chalcolithic Period

Aşağıseyit Höyük is situated at a highly significant transitional point that connects the mountainous and plateau-dominated topography of the Çal Basin with the lowland plains formed by the Çivril and Baklan basins. This location provides access to Bekilli in the north, Uşak in the northeast, Güney and Alaşehir in the west, and subsequently the Gediz Basin, as well as the Lykos Valley and Buldan in the southwest. The site's position on natural trade routes not only underscores its economic significance but also highlights its strategic importance in a military context.

The Late Bronze Age finds uncovered during the excavations at Aşağıseyit Höyük contribute to the evaluation and refinement of the newly proposed chronological framework recently developed through the Beycesultan excavations [9]. Moreover, given its location in a highland area, Aşağıseyit Höyük offers valuable insights into the socio-economic structures of smaller contemporary settlements situated on the periphery of a central site such as Beycesultan. Within this framework, the material culture of Aşağıseyit Höyük—particularly the characteristics of its artifacts, production processes, and the nature of stored and consumed goods—provides critical data for understanding the broader cultural dynamics of the Late Bronze Age in Western Anatolia.

More accurate information about foods made or stored in a vessel may be possible using OR analysis. The development of this technique has therefore piqued the interest of scientists in recent years. In this study, fifty unglazed Late Bronze Age (1550–1200 BC) sherds that were discovered during the excavation of the Aşağıseyit Höyük site, which were amorphous in structure and believed to have been used to preserve certain meals, were subjected to OR analysis. Gas chromatography-mass spectrometry (GC-MS) was used to identify all identifiable chemicals, mostly lipids, that were extracted from the samples using the current acid-catalyzed direct extraction-methylation approach. The intended functions of the sherds and the meals they might contain were deduced from the ORs found during analyses.

Materials and Methods

Chemicals and Reagents

All reagents and solvents used in this study were of analytical grade. Methanol (CH_3OH), sulfuric acid (H_2SO_4, 95–98%), hexane (C_6H_{14}), and n-tetratriacontane ($C_{34}H_{70}$) used as the internal standard were purchased from Sigma-Aldrich (Merck KGaA, Darmstadt, Germany). Sulfuric acid was used in its concentrated protonated form for acid-catalyzed hydrolysis and methylation, while fatty acids present in the ceramic residues were converted into their corresponding fatty acid methyl esters (FAMEs) prior to GC–MS analysis.

The Sherd Samples

The selection of samples for analysis in the Late Bronze Age layers was guided by the understanding that choosing unslipped sherds would enhance the potential for yielding reliable results. Consequently, a significant portion of the analyzed sherds comprised pots (Fig. 2 and Table 2) and they have been found from the 4b layer of the settlement. This layer was selected in particular because it represents the most extensively excavated area of the site, and due to the high number of pots uncovered there in relation to spatial contexts. In the selection of sherds for analysis, form groups that were commonly encountered in the Late Bronze Age were prioritized in addition to the specified physical characteristics. This methodology aims to contribute to the identification of the functional uses of

similarly shaped examples found in various settlements.

Within the framework of the project at Aşağıseyit Höyük, a total of 50 unglazed sherds in various forms from the Late Bronze Age layers were selected and subjected to the extraction method available in the literature, and organic residue analysis (ORA) was performed with the GC-MS device. As in many studies within the scope of the study, unglazed sherds that were likely to have been used in cooking or storage were selected and samples were taken from the rim and body areas of the relevant artifacts, which are more likely to contain OR, and subjected to ORA.

The sherds analyzed in the project were manually and digitally drawn, and subsequently, detailed descriptions were transferred to a digital format. Some of these studies were conducted at the Aşağıseyit Höyük Excavation House, while others took place at İzmir Demokrasi University. All of the sherds in question date back to the early and subsequent periods of the Late Bronze Age. Before organic residue analyses, measurements, and paper drawings of the sherds in question were made. The drawings and measurements reflecting the physical properties were also transferred to the computer environment. In addition, photographs of the sherds were taken from all angles.

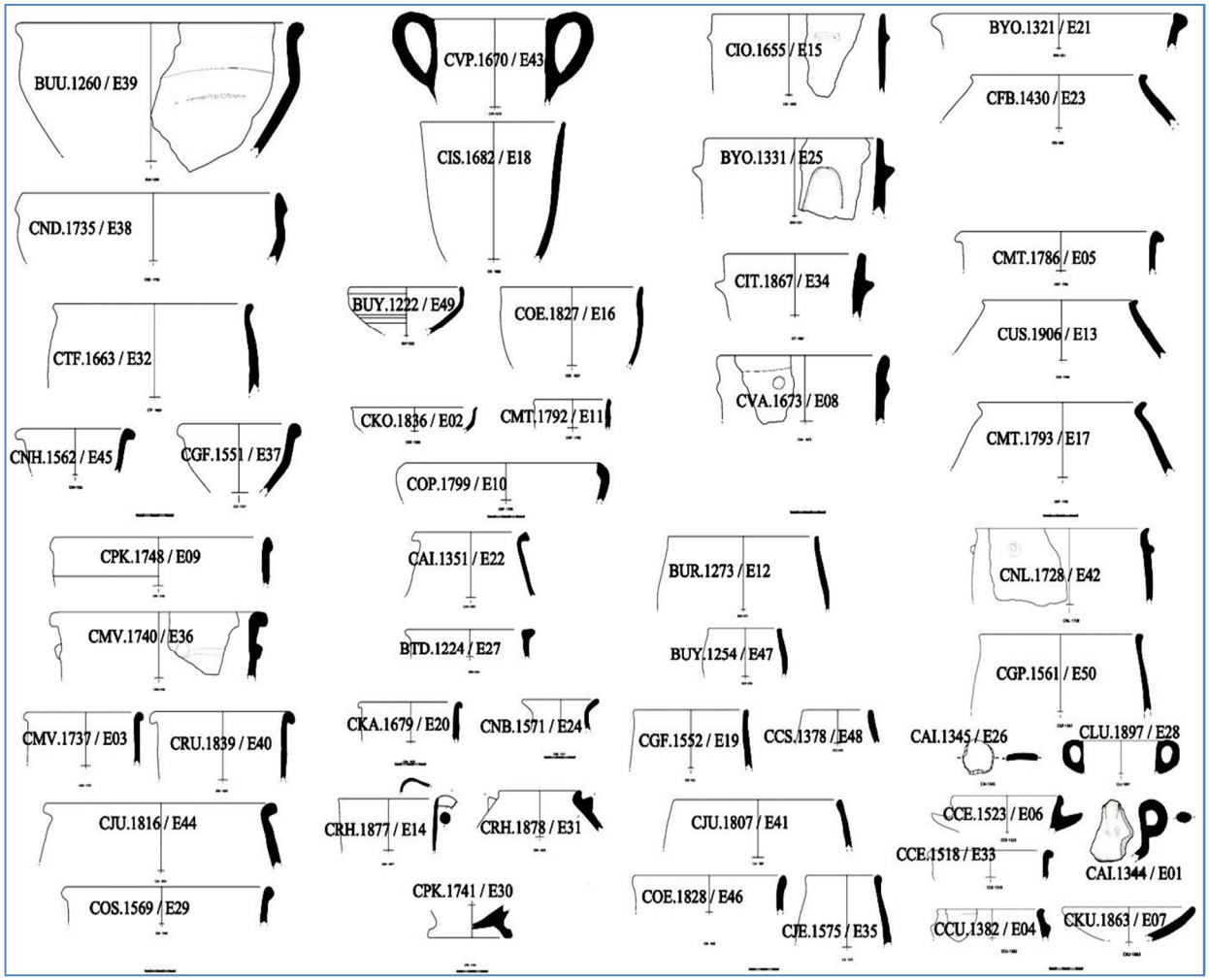

Figure 2. **Late Bronze Age sherds selected for the organic residue analysis, sample code/analysis code**

Table 2. **Total organic residue concentrations and information of the sherds.**

Sherd	Layer	GC-MS Analysis code	Form	Organic residue (µg/g, residue/sherd powder)
CAI.1344	4B	E-01	Cup-shaped Jar	22.31
CMV.1737	4B	E-03	Pot	26.36
CCU.1382	4B	E-04	Pot	42.00
CMT.1786	4B	E-05	Pot	23.65
CCE.1523	4B	E-06	Pot	47.81
CKU.1863	4B	E-07	Bowl	31.35
CVA.1673	4B	E-08	Pot	17.89
CPK.1748	4B	E-09	Pot	149.45
COP.1799	4B	E-10	Pot	304.24
CMT.1792	4B	E-11	Pot	104.48
BUR.1273	4B	E-12	Pot	181.06
CUS.1906	4B	E-13	Pot	216.37
CRH.1877	4C	E-14	Quatrefoil Cups	322.49
CIO.1655	4B	E-15	Pot	808.15
COE.1827	4C	E-16	Bowl	1514.71
CMT.1793	4B	E-17	Pot	535.20
CIS.1682	4B	E-18	Bowl	2545.94
CGF.1552	4B	E-19	Pot	440.90
CKA.1679	4C	E-20	Pot	43.38
BYO.1321	4B	E-21	Pot	54.73
CAI.1351	4B	E-22	Pot	346.79
CFB.1430	4B	E-23	Jar	173.44
CNB.1571	4B	E-24	Jar	2024.11
BYO.1331	4B	E-25	Pot	524.59
CAI.1345	4B	E-26	Pot Body Fragment	153.73
BTD.1224	4B	E-27	Pot	1262.83
CLU.1897	4B	E-28	Cup Shaped Jar	39.04
COS.1569	4B	E-29	Por	86.29
CPK.1741	4B	E-30	Ring Base	658.71
CRH.1878	4C	E-31	Pot	32.28
CTF.1663	4B	E-32	Pot	46.20
CCE.1518	4B	E-33	Bowl	97.93
CIT.1867	4B	E-34	Pot	25.69
CJE.1575	4B	E-35	Pot	23.56
CMV.1740	4B	E-36	Pot	486.64
CGF.1551	4B	E-37	Jar	62.64
CND.1735	4B	E-38	Bowl	420.78
BUU.1260	4B	E-39	Bowl	306.68
CRU.1839	4B	E-40	Pot	267.19
CJU.1807	4B	E-41	Pot	68.57
CNL.1728	4B	E-42	Pot	114.99
CVP.1670	4C	E-43	Cup Shaped Jar	255.09
CJU.1816	4B	E-44	Jar	57.56
CNH.1562	4C	E-45	Bowl	27.02
COE.1828	4C	E-46	Pot	45.98
BUY.1254	4B	E-47	Pot	53.58
CCS.1378	4B	E-48	Pot	135.27
BUY.1222	4B	E-49	Bowl	104.34
CGP.1561	4B	E-50	Pot	47.51

Extraction of the Organic Residues

To recover the ORs embedded within the ceramic matrix, we applied a one-step acid-catalyzed extraction–hydrolysis–methylation protocol commonly used for archaeological pottery [6, 10-14]. First, the exterior surface of each sherd was mechanically cleaned with an engraving hand drill to remove soil-derived or handling-related contaminants [11, 12, 14-17]. Approximately 1 g of cleaned powdered ceramic was transferred into a glass centrifuge tube, and 50 µL of an internal standard solution (n-tetratriacontane, 1000 mg/L) was added. In the first step, bound and unbound ORs were released through acid-catalyzed hydrolysis. For this purpose, 1 mL of concentrated H_2SO_4 and 5 mL of methanol were added to the powdered sample. The mixture was then heated under reflux at 70 °C for 4 h. Under these conditions, ester-linked lipids such as triglycerides and wax esters were hydrolyzed, while the liberated fatty acids were simultaneously methylated by the methanol–sulfuric acid reagent to form fatty acid methyl esters (FAMEs). The tubes were vortexed once every hour to ensure complete contact between the ceramic matrix and the solvent.

After cooling to room temperature, 2 mL of hexane was added to extract the FAMEs from the reaction mixture. The sample was shaken vigorously and centrifuged for 5 min at 2500 rpm, and the upper hexane phase was collected. This extraction was repeated with an additional 2 mL of hexane. The combined hexane extracts were evaporated gently under nitrogen and re-dissolved in 100 µL hexane. The final solution was filtered through a 0.45 µm PTFE syringe filter into a glass insert vial and analyzed by GC–MS. A blank extraction was run for each batch to monitor possible laboratory contamination.

GC-MS Analysis

Splitless mode was employed, and 1 µL of the material was injected. The injection block temperature was 280 ^{o}C. The mobile phase was helium gas, with a flow rate of 1 mL/min. Separation was done on a 30 m x 0.25 mm x 0.25 µm (5%-phenyl)-methylpolysiloxane HP-5MS capillary column in the instrument, which will be used in full scanning mode. The following was the column oven program: 50 ^{o}C for the first min of the analysis, followed by 300 ^{o}C for nine min, with the temperature rising by 5 ^{o}C per min. The source temperature for the MS was 230 ^{o}C, the ionization energy was found to be 70 eV, and the m/z scan range was 40-650. Chemstation 2.00 software was used to control the device and data. 90% or more matches with the MS library were taken into consideration, compounds with 80–89% matches were extensively examined, and matches with less than 80% were not taken into account. The gadget software automatically determined the weight proportion of the substances found as a result of the pertinent extraction. GC-MS analysis was performed with the Agilent 7890N-5975C GC device, which has an MS detector capable of automatic analysis.

Results and Discussions

ORs were detected in all fifty sherds analyzed. The total OR concentration of sherds in which they were detected was calculated with the help of the added internal standard and are given in Table 2. Due to an error originating from the device, the amount and types of residues in only the ceramic-coded E-02 could not be revealed. The % composition of the ORs of the sherds detected is given in Table 3a and 3b. The representative chromatogram of the OR composition of E38 is given in Fig. 3.

Table 3a. **Organic residue compositions of the sherds (E-01 to E-30) by the GC-MS analysis.**

Organic residue %	RT (min)	C:X	E-01	E-03	E-04	E-05	E-06	E-07	E-08	E-09	E-10	E-11	E-12	E-13	E-14	E-15	E-16	E-17	E-18	E-19	E-20	E-21	E-22	E-23	E-24	E-25	E-26	E-27	E-28	E-29	E-30
Octanoic acid, methyl ester	13.466	C8:0									0.83																				
Nonanoic acid, methyl ester	16.314	C9:0																													
Decanoic acid, methyl ester	19.058	C10:0																													
Undecanoic acid, methyl ester	19.072	C11:0									1.22																				
Tetradecane	21.044	C14																													
Pentadecane	23.522	C15		1.90																											
1,4-Benzenedicarboxylic acid, dimethyl ester	23.651																														
BUTYL HYDROXY TOLUENE	23.981		2.48																												
Phenol	23.988																														
Dodecanoic acid, methyl ester (CAS)	24.292	C12:0															1.95														
Nonanedioic (azelaic) acid, dimethyl ester	24.685																														
n-Hexadecane	25.898	C16		2.29																									2.41		
Decanedioic (Sebacic) acid, dimethyl ester	27.022																														
Dihydro methyl jasmonate	27.222																														
Heptadecane (CAS); n-Heptadecane; Normal-heptadecane	28.146	C17		4.87																											
Tetradecanoic (Myristic) acid	28.691	C14:0	3.18	2.98						7.75			3.71	1.89	4.11	1.15	10.05	1.55	1.32	2.41			3.23	6.46	1.47	1.87	2.37	2.21	6.65	2.54	1.43
Octadecane	30.277	C18		1.69																											
Pentadecanoic acid, methyl ester	30.815	C15:0		1.17						1.39														1.50	0.53			1.06	1.72		0.51
Nonadecane	32.311	C19		1.67																											
9-Hexadecenoic (Palmitoleic) acid, methyl ester	32.414	C16:1													8.06							4.24						13.34	5.14		
Benzene	32.608																														
Hexadecanoic (palmitic) acid, methyl ester	32.842	C16:0	47.90	43.93	18.72	22.74	55.65	43.42	48.23	48.37	51.92	56.25	51.95	47.05	55.02	52.01	45.66	57.24	45.55	52.09	32.01	51.85	42.50	52.46	38.23	52.94	57.67	24.42	35.53	52.14	33.84
Eicosane	34.249	C20		2.02																											
Heptadecanoic (margaric) acid, methyl ester	34.779	C17:0																													1.37
Sulfur	35.483									11.32	4.97																				
9-12 Octadecanoic (linoleic) acid, methyl ester	36.096	C18:2			39.89																					1.06		7.59			
Heneicosane	36.110	C21		2.46																											
9-Octadecenoic (oleic) acid, methyl ester	36.158	C18:1	3.88		32.49		7.91		8.31	5.82	3.18		1.70	3.37	15.66	7.17	12.44	3.49	2.45	2.89	4.27	15.49	17.15		2.22	5.90	11.34	45.68	6.79	12.79	3.58
Octadecanoic (staeric) acid, methyl ester	36.613	C18:0	40.40	35.00	3.12	8.31	27.67	25.64	27.72	24.00	33.73	38.76	40.63	47.69	13.99	39.67	29.90	37.72	50.68	42.61	22.34	19.03	37.12	39.57	57.55	38.23	28.61	5.69	30.89	29.61	58.88
Docosane	37.903	C22																													
Tricosane	39.606	C23																													
Eicosanoic (Arachidic) acid, methyl ester	40.082	C20:0																													0.39
Pentacosane	42.833	C25																													
Docosanoic acid, methyl ester	42.916	C22:0							4.52																				8.75	2.91	
14-.BETA.-H-PREGNA	42.937																														
Tetracosanoic acid, methyl ester	46.246	C24:0																													
Heptacosane	48.563	C27																				4.97									
Hexacosanoic acid, methyl ester	49.011	C26:0																													
Other			2.16	0.02	5.78	68.95	8.77	30.94	11.22	1.35	4.15	4.99	2.01	0.00	3.16	0.00	0.00	0.00	0.00	0.00	41.38	4.42	0.00	0.01	0.00	0.00	0.01	0.01	2.12	0.01	0.00
C16:0/C18:0			1.19	1.26	6.00	2.74	2.01	1.69	1.74	2.02	1.54	1.45	1.28	0.99	3.93	1.31	1.53	1.52	0.90	1.22	1.43	2.72	1.14	1.33	0.66	1.38	2.02	4.29	1.15	1.76	0.57

Table 3b. **Organic residue compositions of the sherds (E-31 to E-50) by the GC-MS analysis**

Organic residue %	RT (min)	C:X	E-31	E-32	E-33	E-34	E-35	E-36	E-37	E-38	E-39	E-40	E-41	E-42	E-43	E-44	E-45	E-46	E-47	E-48	E-49	E-50	Max	Min	Average	Pre-comment
Octanoic acid, methyl ester	13.466	C8:0																								
Nonanoic acid, methyl ester	16.314	C9:0													1.02											
Decanoic acid, methyl ester	19.058	C10:0									0.43				0.69											
Undecanoic acid, methyl ester	19.072	C11:0																								
Tetradecane	21.044	C14														1.05										
Pentadecane	23.522	C15														1.41										
1,4-Benzenedicarboxylic acid, dimethyl ester	23.651														5.03	2.63										Pollutant
BUTYL HYDROXY TOLUENE	23.981								1.52																	Pollutant
Phenol	23.988		4.13	2.83										1.21	1.05	2.71	4.86				1.47					Pollutant
Dodecanoic acid, methyl ester (CAS)	24.292	C12:0								0.85	0.91				0.83						2.30	5.49				
Nonanedioic (azelaic) acid, dimethyl ester	24.685									0.73	0.53				0.90											
n-Hexadecane	25.898	C16							1.33							2.05										
Decanedioic (Sebacic) acid, dimethyl ester	27.022														2.16											
Dihydro methyl jasmonate	27.222		1.77																							Pollutant
Heptadecane (CAS); n-Heptadecane; Normal-heptadecane	28.146	C17														0.95										
Tetradecanoic (Myristic) acid	28.691	C14:0	7.10	2.90	4.49		3.58	2.02	3.25	4.69	9.05	3.29	6.26		3.62	2.55	3.10	2.98	2.71	1.45	10.83	7.34				
Octadecane	30.277	C18													0.83	0.80			0.62							
Pentadecanoic acid, methyl ester	30.815	C15:0	3.78	1.40	1.93			0.94	1.47	0.82	4.60		1.62		0.79	0.84			0.84		0.92	0.97				
Nonadecane	32.311	C19													1.30											
9-Hexadecenoic (Palmitoleic) acid, methyl ester	32.414	C16:1	8.83	2.93	6.07		17.04		3.82	0.54	6.61		4.13		0.75	1.71	1.59		19.58		8.89	12.08				
Benzene	32.608														1.50											Pollutant
Hexadecanoic (palmitic) acid, methyl ester	32.842	C16:0	44.96	41.60	48.27	55.12	47.66	37.98	45.23	49.74	44.16	53.64	48.88	54.84	31.86	38.56	53.92	54.86	40.38	31.68	42.32	35.04	57.67	18.72	45.27	
Eicosane	34.249	C20													2.24	1.13										
Heptadecanoic (margaric) acid, methyl ester	34.779	C17:0						1.77		1.24	1.20		1.06													
Sulfur	35.483			12.67											4.98	2.09										Pollutant
9-12 Octadecanoic (linoleic) acid, methyl ester	36.096	C18:2				7.04			1.72				2.21						3.76	25.69			39.89	1.06	11.12	
Heneicosane	36.110	C21																								
9-Octadecenoic (oleic) acid, methyl ester	36.158	C18:1	8.56	5.47	4.92	10.70	5.44	1.63	5.60	12.87	5.50	7.00	7.07	2.83	12.64	3.68	3.94	3.85	7.57	32.17	7.64	9.16	45.68	1.63	8.96	
Octadecanoic (staeric) acid, methyl ester	36.613	C18:0	20.87	26.43	31.06	27.13	20.04	55.66	27.42	27.42	12.11	34.03	27.29	41.12	16.56	21.42	25.52	35.70	23.31	9.00	11.21	15.20	58.88	3.12	29.45	
Docosane	37.903	C22													0.92											
Tricosane	39.606	C23													0.92											
Eicosanoic (Arachidic) acid, methyl ester	40.082	C20:0								0.50	0.62															
Pentacosane	42.833	C25													0.80											
Docosanoic acid, methyl ester	42.916	C22:0		2.15	3.26		6.24		8.64		1.18	2.04	1.47			1.12	5.02	2.61	1.23		1.19	3.21				
14-.BETA.-H-PREGNA	42.937														1.63											Pollutant
Tetracosanoic acid, methyl ester	46.246	C24:0		1.62							1.21															
Heptacosane	48.563	C27																					4.97	4.97	4.97	
Hexacosanoic acid, methyl ester	49.011	C26:0									0.50															
Other			0.00	0.00	0.00	0.01	0.00	0.00	0.00	0.60	11.39	0.00	0.01	0.00	6.98	15.30	2.05	0.00	0.00	0.01	13.23	11.51	68.95	0.00		
C16:0/C18:0			2.15	1.57	1.55	2.03	2.38	0.68	1.65	1.81	3.65	1.58	1.79	1.33	1.92	1.80	2.11	1.54	1.73	3.52	3.78	2.31	6.00	0.57		

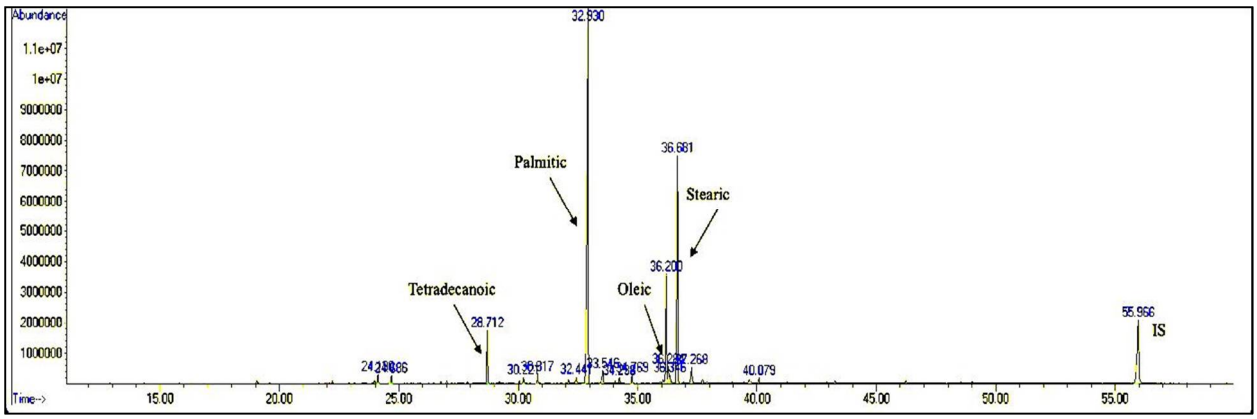

Figure 3. **The representative GC-MS chromatogram of the OR composition of E38**

When Table 2 was examined, E-18 stands out with the highest OR concentration (2545.94 µg/g). When all sherds were considered, an average of 313.91 µg/g OR was determined. The acid-catalyzed method used was found to be a useful approach to obtain ORs in the sherds. OR concentrations well above 5 µg/g were obtained from all sherds; this is considered sufficient to make reliable interpretations [18, 19]. This result indicates that the sherds examined have undergone a relatively effective preservation process concerning ORs.

Located approximately 40 km from Aşağıseyit Höyük and considered the central settlement of the Çivril plain and the region, Beycesultan has revealed numerous forms, such as chalices and fruit stands, that likely reflect the consumption habits of elite classes in its Late Bronze Age pottery repertoire. This suggests that the elite class met their daily needs through pottery with specific functions. Despite Aşağıseyit Höyük's proximity to Beycesultan, the significantly lower number of forms like chalices and fruit stands found in the excavations at Aşağıseyit Höyük compared to Beycesultan is an issue that warrants attention. If this discrepancy is not related to the nature of the excavated areas, it could indicate a class distinction in the consumption habits during the Late Bronze Age. This suggests that there may have been certain standards for the everyday use of these vessels.

Over the course of long-term soil breakdown processes, ORs are exposed to many biochemical situations. ORs either completely break down or provide hints through their decomposition products in these processes, which are entirely based on probabilities. Some of them are able to withstand decomposition because of their stable, extremely hydrophobic structures and excellent preservation,. Because of their long hydrophobic hydrocarbon chains and stable saturated structures, palmitic and stearic acids seem to be the fatty acids that are most resistant to breakdown processes. Therefore, practically all archaeometric ORA investigations include these two fatty acids. The highest OR rates are found in stearic and palmitic acids, according to Table 3. Additionally, unsaturated oleic and linoleic acids were found, which have a rather stable structure. Concentrations ranging from 18.72-57.67%, 3.12-58.88%, 1.63-45.68%, and 1.06-39.89% were detected for palmitic, stearic, oleic, and linoleic acids, respectively. When evaluating the fatty acids detected in ORs, the ratios of some fatty acids clarified in previous studies should be taken into consideration rather than the composition. Some interpretations are accepted based on the most stable of these, palmitic and stearic acids. It

has been reported that if the stearic acid ratio in the detected ORs is higher than that of palmitic acid, it is likely to be the carcass meat fat of a ruminant animal [20]. When Table 3 is evaluated, it is seen that the sherds E-18, E-24, E-30, and E-36 have a ratio suitable for this definition. Similar results were obtained on this subject with the only study conducted in Western Anatolia, but examining the Neolithic period [21]. On the other hand, if palmitic acid with a higher ratio than stearic acid is present in the presence of pentadecanoic acid, heptadecanoic (margaric) acid, and oleic acid, it has been stated that the relevant ORs may be the milk product or fat of a ruminant animal [20]. E-38, E-39, and E-41 can be evaluated within this scope. This finding presents a plausible scenario concerning both the settlement and the socio-economic organization of the period. The Late Bronze Age represents a time when extensive plateaus and plains were utilized for pastoralism. Historical records of the Hittites indicate that from the reign of I. Hattuşili to the later years of the Hittite Empire, Hittite kings conducted military campaigns in Western Anatolia, acquiring thousands of animals as spoils of war and transporting them back to the Hittite core region [22]. Among the animal bones found at Aşağıseyit Höyük, there is a substantial presence of bones from both large and small ruminants. These findings and the results of the analyses suggest a coherent picture regarding both the settlement and the socio-economic organizational model of the period.

Due to long-term burial conditions, intact mono-, di-, and triglycerides are rarely preserved in archaeological ceramics. In the samples, the original acylglycerol structures were hydrolyzed prior to analysis and subsequently during the acid-catalyzed methanolysis step applied in our extraction protocol. As a result, any surviving bound or unbound lipids were converted into their corresponding free fatty acids and fatty acid methyl esters. Therefore, the “total organic residue” values reported in this study represent the degraded lipid fraction dominated by saturated and unsaturated fatty acids rather than intact mono-, di-, or triglycerides.

When examining the typology of vessels likely used for storing or cooking carcass meat, we identify several forms: a pot with a thickened rim and a knobby decoration, a deep pot with a simple outward-turned rim, a necked pot with an outward-flaring mouth, and a bowl on a pedestal. Among these sherds, the examples illustrated in Fig. 2 can be considered suitable forms for the preservation and long-term storage of meat. However, the bowl pedestal shown in Fig. 2 stands out as a form more appropriate for daily consumption rather than for the long-term preservation of carcass meat products.

The presence of palmitic acid in higher ratios than stearic acid, in conjunction with pentadecanoic acid, heptadecanoic (margaric) acid, and oleic acid, suggests that the associated ORs could originate from dairy products or fat derived from ruminant animals. Considering the aforementioned types of ruminants present in the settlement, the detection of dairy products in the Late Bronze Age settlement can be anticipated.

Two of the identified ORs were found in pots with thickened rims and sharp shoulders, which are among the most commonly encountered and characteristic forms of the Late Bronze Age ceramic repertoire (Fig. 2). These types of pots can be easily used for the production, serving, and consumption of not only oils but also yogurt and similar products. It is highly probable that the ORs found in another form—pots with simple rims, and semi-spherical bodies are

associated with the storage of dairy products rather than oil products (Fig. 2).

Dicarboxylic acids are the most stable breakdown products of unsaturated fatty acids, particularly in soils with arid climates [23]. Table 3 demonstrates that sebacicdicarboxylic acids were found in the analyzed sherds in addition to azelaic acid. The plant oil is most likely indicated by the presence of dicarboxylic acids [24]. Because of its electrons and its position in the center of the chain, the double bond between oleic acid's ninth and tenth carbons has a tendency to break in anaerobic conditions, producing two nine-carbon azelaic acids. On the other hand, the relevant OR is probably a plant oil residue if the palmitic acid content is significant. Together with azelaic or sebacic acid, palmitic acid—roughly twice as much as oleic and stearic acid—is present in the ORs of E-38, E-39, and E-43. Thus, it may be concluded that the ORs found in these sherds are probably plant-derived oil residues.

Although archaeobotanical studies on the soils of the Late Bronze Age layers at Aşağıseyit Höyük are still in their early stages, initial observations have identified remains of barley, wheat, bitter vetch, and other legumes, as well as fruits such as grapes, indicating that the settlement hosted a rich capacity for plant production and/or consumption during the period. Therefore, considering the intense production capacity in the settlement and the region, the presence of plant oil residues in the sherds can be regarded as an expected outcome. Another important point to note is that these residues were found in vessels also associated with the storage of dairy products. It appears that the thickened-rim, sharp-shouldered bowls, which can be defined as the most characteristic form of the Late Bronze Age, hosted a variety of productions (Fig. 2). This finding is significant as it suggests that the most commonly found forms in this process were not only used for storage but also demonstrated a wide diversity of uses. Another form that has been identified with plant oil residues is the simple, outward-flaring rimmed, single-handled transfer vessel (Fig. 2). This form is highly suitable for both storing liquid products and transferring them elsewhere. The existing n-alkane structures detected within the scope of the analyses are dominated by even carbon numbers and are rich in C16 and C18 [25], increasing the possibility of petroleum-derived pollution.

A comparison of the OR results obtained in this study with those published previously for Early Bronze Age ceramic bottles from the Küllüoba Höyük site [26] reveals both methodological consistency and notable differences in the chemical profiles, reflecting distinct vessel functions and cultural practices across periods. In both analyses, GC-MS successfully recovered well-preserved lipid assemblages dominated by common degradation products such as palmitic, stearic, and oleic acids, as well as dicarboxylic acids including azelaic and sebacic acid compounds widely considered as robust indicators for plant-derived oils. This overlap demonstrates that plant oils were a persistent component of vessel use in western Anatolia from the Early to Late Bronze Age. However, while the Küllüoba bottles frequently exhibited complex mixtures of plant oils with additives such as conifer resin (evidenced by dehydroabietic acid) and, in some cases, markers potentially associated with dairy, medicinal or aromatic substances, the Aşağıseyit Höyük assemblage is more strongly dominated by simpler lipid profiles linked to ruminant carcass fats, dairy products, and plant oils without clear evidence for resinous or aromatic additives.

These differences likely reflect the distinct functional and cultural contexts of the two vessel groups. The small, fine-walled, imported bottles from Küllüoba — many

associated with ritual feasting pits — appear to have contained high-value, possibly scented or medicinal preparations, consistent with textual and archaeological evidence for long-distance exchange of aromatic oils during the third millennium BCE. In contrast, the sherds from Aşağıseyit Höyük, largely representing utilitarian Late Bronze Age cooking and storage vessels, show lipid signatures more typical of routine domestic food processing, including carcass fats, ruminant milk products, and plant oils used in everyday culinary activities. Thus, while both studies demonstrate the effectiveness of GC-MS for recovering ancient ORs, the molecular distinctions between the two datasets provide valuable insight into shifting culinary, economic, and ritual practices from the Early to Late Bronze Age in western Anatolia.

The results of this study are not only significant for understanding past culinary and domestic practices but also provide valuable insights for chemists interested in the molecular behavior of ORs over long-term burial. The clear degradation patterns observed—such as triglyceride hydrolysis, the preferential preservation of saturated fatty acids, and the oxidative formation of dicarboxylic acids—illustrate the predictable chemical pathways by which complex organics transform within a ceramic matrix. These findings contribute to a broader understanding of ancient lipid diagenesis, offering a natural laboratory for studying hydrolysis–oxidation mechanisms, adsorption processes, and the long-term stability of organic compounds in porous mineral substrates.

From an environmental science perspective, the study highlights how soil chemistry, moisture fluctuations, and microbially mediated processes shape the fate of organic molecules over millennia. The selective retention of fatty acids in the ceramic matrix demonstrates the capacity of mineral surfaces to preserve ORs despite extensive environmental alteration. These results have broader implications for understanding organic–mineral interactions in soils, the persistence of environmental lipids, and the mechanisms governing the long-term sequestration or transformation of organic pollutants. By integrating analytical chemistry with archaeological context, this research provides a multidisciplinary framework relevant to both heritage science and environmental geochemistry.

The results obtained that have the potential to provide meaningful data are compiled in Table 4. It is possible to make the following evaluations according to the relevant table.

Table 4. **Characteristics of organic residues obtained from the sherds as a result of GC-MS analysis.**

Ceramics	Molecular properties	Comment
E-18, E-24, E-30, and E-36	Stearic acid content is higher than palmitic acid	Carcass meat fat of a ruminant animal
E-38, E-39, and E-41	Palmitic acid, which has a higher ratio than stearic acid in the presence of pentadecanoic acid, heptadecanoic (margaric) acid, and oleic acid	Fat or dairy product from a ruminant animal
E-38, E-39, and E-43	Presence of oleic acid, azelaic acid, and about twice as much palmitic acid as stearic acid	Plant oils

- It is seen that the ORs detected in the E-18, E-24, E-30, E-36, E-38, E-39, E-41, and E-43 sherds out of the 50 sherds analyzed gave interpretable results according to their OR compositions.
- The sherds of E-18, E-24, E-30, and E-36 may have been used in processes involving the carcass meat of a ruminant animal.
- The sherds of E-38, E-39, and E-41 may also contain a ruminant animal or a dairy product. The ORs obtained from E-38 and E-39 of these sherds also indicate plant oils. These results show that the relevant vessels may have been used for cooking or storing animal products together with plant oils.
- It can be said that the OR in sample E-43 is predominantly a residue of plant oil and that this vessel may have been used mostly for materials containing plant oils.
- Table 3 shows that some molecules are interpreted as pollution. As a result of modern human activities, the soil is exposed to high levels of pollution. The most prominent of these pollutions is plastic and fossil fuel use. Petrochemical pollution that reaches the soil through air and water can be absorbed in structures located underground, such as archaeological ceramics. These molecules interpreted as pollution have high water solubility properties and the probability of being a residue that could originally be found in the structure of the relevant sherd is very low.

Conclusions

In this presented study, ORA was used to determine the intended use of the Late Bronze Age ceramics of Aşağıseyit Höyük, one of the important ancient settlements of Western Anatolia. The ORs found in the sherds that allow interpretation were evaluated, and inferences were made by considering other studies in the literature regarding the purpose of use of the relevant sherds. It should be noted that these evaluations, especially for palmitic and stearic acid, performed only on ratios, will be more comparable with isotope analysis.

Conflict of Interest

The authors report no conflict of interest.

Acknowledgments

The authors would like to thank the Scientific and Technological Research Council of Türkiye (TÜBİTAK) for providing financial support for this study under 1002 - A Short term Support Module (Grant Number: 223K040).

References

1. L. Blanco-Zubiaguirre, M. Olivares, K. Castro, J. A. Carrero, C. García-Benito, J. Á. García-Serrano, J. Pérez-Pérez and J. Pérez-Arantegui, *Anal. Bioanal. Chem.,* 411 (2019) 6711. doi: 10.1007/s00216-019-02044-1.
2. J. Dunne, K. M. Grillo, E. Casanova, H. L. Whelton, R. P. Evershed, *J. Archaeol. Method Theory,* 26 (2019) 619. doi: 10.1007/s10816-018-9384-0.
3. F. A. Hansel, I. D. Bulland R. P. Evershed, *Rapid Commun. Mass Spectrom., 25* (2011) 1893. doi: 10.1002/rcm.5038.
4. F. A. Hansel, M. S. Copley, L. A. S. Madureiraand R. P. Evershed, *Tetrahedron Lett.*, 45 (2004) 2999. https://doi.org/10.1016/j.tetlet.2004.01.111.
5. S. N. Dudd, R. P. Evershed and A. M. Gibson, *J. Archaeol. Sci.*, 26 (1999) 1473. https://doi.org/10.1006/jasc.1998.0434.
6. V. Papakosta, R. H. Smittenberg, K. Gibbs, P. Jordan and S. Isaksson, *Microchem. J.,* 123 (2015) 196.

https://doi.org/10.1016/j.microc.2015.06.013
7. E. Abay, B. Temür, E. Konakçı and B. Semiz, *Mediterr. Archaeol. Archaeom.,* 20 (2020) 107. doi: 10.5281/zenodo.3605666.
8. E. Konakçı, Yüzey Araştırmaları Verilerine Göre Aşağıseyit Höyük Yerleşiminde İskân Süreci, in: Chapter title? B. Tunçsiper, D. İnan (Eds.) Beşeri Bilimler Ekseninde Güncel Araştırmalar: Kuramlar, Kavramlar, Uygulamalar, Kriter Yayınevi, İstanbul, (2020) 15-33.
9. F. Dedeoğlu and A. Abay, *Arkeoloji Dergisi,* 19 (2014) 1. doi: ??? or weblink
10. O. E. Craig, H. Saul, A. Lucquin, Y. Nishida, K. Taché, L. Clarke, A. Thompson, D. T. Altoft, J. Uchiyama, M. Ajimoto, K. Gibbs, S. Isaksson, C. P. Heron and P. Jordan, *Nature,* 496 (2013) 351. doi: 10.1038/nature12109.
11. M. Leclerc, K. Taché, S. Bedford, M. Spriggs, A. Lucquin and O.E. Craig, *J. Archaeol. Sci.: Rep.,* 17 (2018) 712. https://doi.org/10.1016/j.jasrep.2017.12.019.
12. S. Mileto, E. Kaiser, Y. Rassamakin and R.P. Evershed, *J. Archaeol. Sci.: Rep.,* 13 (2017) 67. https://doi.org/10.1016/j.jasrep.2017.03.028.
13. E. Oras, A. Lucquin, L. Lõugas, M. Tõrv, A. Kriiska and O.E. Craig, *J. Archaeol. Sci.*, 78 (2017) 112. https://doi.org/10.1016/j.jas.2016.11.010
14. V. Papakosta, E. Oras and S. Isaksson, *J. Archaeol. Sci.: Rep.,* 24 (2019) 142. https://doi.org/10.1016/j.jasrep.2019.01.003.
15. L. Drieu, M. Horgnies, D. Binder, P. Pétrequin, A.-M. Pétrequin, K. Peche-Quilichini, T. Lachenal and M. Regert, *Archaeometry*, 61 (2019) 1081. https://doi.org/10.1111/arcm.12479.
16. F. Kherbouche, J. Dunne, S. Merzoug, S. Hachi and R.P. Evershed, *Quatern. Int.,* 410 (2016) 50. https://doi.org/10.1016/j.quaint.2016.01.005.
17. O. P. Nieuwenhuyse, M. Roffet-Salque, R. P. Evershed, P. M. M. G. Akkermans, and A. Russell, *J. Archaeol. Sci.,* 64 (2015) 54. https://doi.org/10.1016/j.jas.2015.10.002
18. R. P. Evershed, M. S. Copley, L. Dickson and F. A. Hansel, *Archaeometry,* 50 (2008) 101. https://doi.org/10.1111/j.1475-4754.2007.00368.x.
19. E. A. Reber, M. T. Kerr, H. L. Whelton and R. P. Evershed, *Archaeometry,* 61 (2019) 131. https://doi.org/10.1111/arcm.12403.
20. M. Regert, *Mass Spectrom. Rev.*, 30 (2011) 177. https://doi.org/10.1002/mas.20271.
21. İ. Tarhan, Ş. Çelikten and Z. Derin, *J. Archaeol. Sci.: Rep.,* 61 (2025) 104954. https://doi.org/10.1016/j.jasrep.2024.104954
22. E. Konakçı, The Lykos Valley During The Second Millenium BC, Landscape and History in the Lykos Valley, Cambridge Scholars Publishing, United Kingdom (2017) page number?. weblink?
23. M. Regert, H. A. Bland, S. N. Dudd, P. F. V. Bergen and R. P. Evershed, *Proc. Biol. Sci.,* 265 (1998) 2027. doi:10.1098/rspb.1998.0536.
24. M. Roffet-Salque, J. Dunne, D. T. Altoft, E. Casanova, L. J. E. Cramp, J. Smyth, H. L. Whelton and R. P. Evershed, *J. Archaeol. Sci. Rep.*, 16 (2017) 627. doi:https://doi.org/10.1016/j.jasrep.2016.04.005.
25. H. L. Whelton, S. Hammann, L. J. E. Cramp, J. Dunne, M. Roffet-Salque and R. P. Evershed, *J. Archaeol. Sci.,* 132 (2021) doi: 10.1016/j.jas.2021.105397.
26. İ. Tarhan, M. Massa, M. Türkteki and S. Türkteki, *Archaeometry,* 65 (2023) 833. https://doi.org/10.1111/arcm.12852.