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RESIDUE ANALYSIS OF GRINDING STONES FROM CHALCOLITHIC GÜLPINAR

KALKOLİTİK GÜLPINAR ÖĞÜTME TAŞLARININ KALINTI ANALİZLERİ

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Abstract

This study presents the preliminary results of residue analysis of grinding stones recovered from the Early Chalcolithic 2 and Middle Chalcolithic period settlements (Gülpınar II and Gülpınar III) in the Sanctuary of Apollo Smintheus (Smintheion), located in the southwestern corner of the modern Biga Peninsula (Ancient Troad) in north-western Anatolia. The Early Chalcolithic 2 period settlement at Gülpınar II has been dated to 5320-4940 BC, while the Middle Chalcolithic period settlement at Gülpınar III superimposing it has radiocarbon dates ranging between 4930 and 4450 BC. Excavations at Chalcolithic Gülpınar between 2004 and 2014 revealed a total of 453 ground stone tools. This work in this context residue analyze of total of 28 grinding stones that were uncovered from these two Early Chalcolithic 2 and Middle Chalcolithic cultural levels.

Chemical analysis of residue on stone tools has become a common research technique used in archaeology science. Chemical residues in and on the working surface of used grinding tools lead us to consider the context of the tool usage. The widely-used Gas Chromatography–Mass Spectrometry (GC-MS) method was selected for analyses of the grinding stones from Chalcolithic Gülpınar. The chemical analysis and preliminary results obtained from Gülpınar samples are outlined in this study. Results show evidence of the distinctive lipid organic residues *pentacosane* and *heptacosane* as minor and trace constituents in some of the essential oils of edible crops and the major fatty acids of *octadecanoic (stearic)* and *palmitic (oleic)*. Palmitic acid is also a major component of the oil found in dairy products and abundant in animal fat. These results may contribute to approaches that aim to explain how prehistoric societies carried out processing of organic and inorganic raw materials.

Keywords: Chalcolithic, Gülpınar, Ground stone, Residue Analysis, GC-MS.

Öz

Bu çalışma Kuzeybatı Anadolu Biga Yarımadası (Antik Troas Bölgesi) güneybatı ucunda bulunan Apollon Smintheus Kutsal Alanı (Smintheion) kazıları sırasında ortaya çıkan Erken Kalkolitik 2 ve Orta Kalkolitik dönem yerleşimleri buluntusu (Gülpınar II ve Gülpınar III) öğütme taşlarının öncül kalıntı analiz sonuçlarını sunmaktadır. Erken Kalkolitik 2 dönemine ait Gülpınar II yerleşimi radyokarbon tarihlemesine göre M.Ö. 5320-4940 tarihlerine sahipken bunun üzerine kurulan Orta Kalkolitik dönemine ait Gülpınar III yerleşimi M.Ö. 4930 ve 4450 arası radyokarbon tarihlerine sahiptir. Gülpınar'da 2004 ve 2014 yılları arasında gerçekleştirilen 11 yıllık kazı sezonunda toplam 453 adet sürtme taş alet ve obje gün ışığına çıkarılmıştır. Bu çalışma ise Erken Kalkolitik 2 ve Orta Kalkolitik döneme ait 28 adet öğütme taşının kalıntı izi analizinden oluşmaktadır.

Son yıllarda arkeoloji biliminde taş aletler üzerinde kimyasal kalıntı izi analizleri yapılması yaygın bir uygulama olarak görülmektedir. Prehistorik Gülpınar öğütme taşlarının analizinde yaygın olarak kullanılan Gaz Kromatografisi/Kütle Spektro-metresi (GC-MS) kalıntı analizi metodu tercih edilmiştir. Kalkolitik Gülpınar örneklerinden elde edilen analiz ve ön değerlendirme sonuçları bu çalışmanın ana hatlarını oluşturmaktadır. Analiz sonuçları yenilebilir tohum ve tahılların içeriğinde eser miktarda bulunan *pentakosan* ve *heptakosan* gibi esansiyel yağlar ile ana yağ asitlerinden olan oktadekanoik (stearik) ve palmitik (oleik) lipit organik kalıntıları belirgin ölçüde tespit edilmiştir. Palmitik asit süt ürünleri ve hayvansal kökenli yağlarda bolca bulunan ana bileşenlerinden biridir. Analiz sonuçlarının prehistorik toplumların organik ve inorganik ham maddeleri nasıl bir öğütme işlemine tabi tuttuklarını açıklamayı amaçlayan yaklaşımlara katkı sunması beklenmektedir.

Anahtar Kelimeler: Kalkolitik, Gülpınar, Öğütme Taşı, Kalıntı Analizi, GC-MS.

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Introduction

This study concerning the importance of residue analysis, which has enormous potential in the identification of ground stone artifacts, determination of assumed functions, and evaluation of prehistoric daily life. Early studies benefited from ethno-archaeological approaches to support the functional usage of ground stone tools. However, for the past few decades the organic residues obtained from heavily-used ground stone objects widely grinding stones, grinding slabs, handstones, pestles and mortars used for food processing and other grinding tasks have been neglected and not yet been studied extensively. Nevertheless, organic residue analysis has been used on pottery findings associated with cooking activities or food storage for a long time. A large number of studies on archaeological data obtained by means of this analysis confirm the presence of varied remains of organic compounds such as fatty acids and lipids (Condamin et al. 1976; Marchbanks 1989; Patrick et al. 1985; Skibo 1992; Charters et al. 1993; Carl & Evershed 1993; Mottram et al. 1999; Regert et al. 1998; Craig 2002). The results of residue analysis have proven that it is a feasible approach for identifying sustenance remains, and organic and inorganic compounds on ground stone tools.

Recently, analyze of residues on stone tools has become a significant research approach in archaeology. The current study tested the possibility that organic residues from prehistoric grinding activities might remain on ground stone tools. The research employed Gas Chromatography–Mass Spectrometry analysis (GS-MC) to help identify residue remains from grinding stones obtained from Chalcolithic Gülpınar. GS-MC has become a widely-used analytical method for residue analyses in recent years. Chemical investigation of residues on ground stone tools provides a new approach to their functional use as against the traditional view. Residue analyses of ground stone objects may offer significant insights into the diet of prehistoric Gülpınar.

Site and assemblage of ground stone objects

The Prehistoric Gülpınar is located on the southwestern tip of the Biga Peninsula in northwestern Turkey close to the village of Gülpınar (Fig. 1). The area in which the prehistoric settlement is located has often been linked with the Graeco-Roman site of Chrysa and the sanctuary of Apollo Smintheus, which was first mentioned by Homer (*Iliad* I. 37, 390, 431.). Excavations since 1980 led by a team from Ankara University have revealed a Hellenistic Sanctuary and Roman Bath complexes (Özgünel 2001; 2003). Archaeological soundings nearly two hundred meters west of the sanctuary area in 1982 yielded prehistoric remains which were subsequently dated to the fifth millennium BC (Sperling 1976; Seeher 1987: 533). Systematic prehistoric excavations initiated by a team between 2004-2014 supported by the Institute for Aegean Prehistory (INSTAP) noted significant remains of a prehistoric settlement ascribed to the poorly-understood Middle Chalcolithic period in western Anatolia (Takaoğlu 2006).

Figure 1. Map of NW Anatolia showing prehistoric sites mentioned in the text.

Prehistoric Gülpınar is represented uncommonly by a flat, extended settlement. Due to limited evidence regarding the Chalcolithic period generally, the prehistoric settlement at Smintheion is a promising site that can help shed light on this period in western Anatolian chronology. The finds are temporally placed in the timeframe of ca. 5320-4450 BC on the basis of AMS dating. Prehistoric occupation is attested in the following phases; Phase I (*Early Neolithic Period*) and Phase II (Early Chalcolithic 2) and Phase III (*Middle Chalcolithic Period*) on the basis of the conventional tripartie division of western Anatolian chronology. The Early Chalcolithic 2 period settlement at Phase II has been dated to 5320-4940 BC, while the Middle Chalcolithic period settlement at Phase III superimposing it has AMS dates ranging between 4930 and 4450 BC. (Takaoğlu 2006).

The Gülpınar ground stone objects, which were commonly used for grinding, especially cultivated crops, consist of more than 400 samples. The vast majority of the stone objects were found in Phase II and III. The assemblage consists predominantly (63%) of milling stones such as grinding stones (lower stones / stationary) and handstones (upper stones / portable), which are mostly complete and have been used, but also includes unused examples. The finds are typically concave in their longitudinal profile and plano-convex shape in section, uni-facial, and have one or more flat/concave ventral surfaces. Some specimens have worn traces, the result of extensive abrasive use. The ground stone objects are diverse in size, averaging nearly 35 cm at their greatest dimension, and they vary in plan between elliptical, rectangular, ovate and irregular shape (Fig. 2).

Furthermore, petrographic analysis shows that the main raw material source for the ground stone objects is metamorphic rock outcrops. The assemblage is mostly made from local andesite but also includes basalt, granite and serpentine. The raw materials for production were easily acquired from slopes facing the site to the east (Table 1).

Artifact Type	*Count*	*%*	*Form*	*Total*	*%*	*Phase I*		*Phase II*		*Phase III*	
Grinding Stones	**68**	22%	**1** Ovate	18	26%	-	-	5	28%	13	72%
			2 Elliptical	12	18%	1	8%	4	33%	7	58%
			3 Rectangular	14	21%	-	-	4	29%	10	71%
			4 Irregular	9	13%	-	-	3	33%	6	67%
			5 Large/Slab	15	22%	-	-	2	-	13	87%
			Total	**68**		**1**	**1,5%**	**18**	**26%**	**49**	**72%**

Figure 2. Shapes of grinding stones according to types.

Table 1. Selected ground stone tools in each geological category.

Geological Category	*Rock Type*	*Grinding Stones*		*Handstones*		**Total**	
Igneous Rocks	Andesite	53	68%	5	32%	**8**	75%
	Basalt	6	55%		45%	**1**	11%
	Granite	9	69%		31%	**3**	13%
Sedimentary Rocks	Sandstone	0	0%		6%		2%
	Total	**68**	**65%**	**6**	**35%**	**04**	**100%**

Preparation of samples for analysis

The methodology of sample preparation took place in two main stages. The first stage was selection of the artifacts by visual inspection. For Gülpınar grinding stones and handstones, visual macroscopic inspection of the ground stone tool surface easily distinguished the working (used) surface from unused areas. Typically, concave grinding stones in their longitudinal profile and on the grinding surfaces, which were polished over the entire area, especially at midpoint, by extensive abrasive use-wear, were selected for analysis. For diagnosis, ground stone objects were intentionally chosen from examples found near hearths, ovens and food preparation areas, where routine daily activities took place. Heavily-used grinding stones functioning as both upper and lower stones were selected for chemical examination. Unwashed and uncontaminated examples in particular were taken into consideration.

The second stage was the extraction of samples by drilling process. Samples were extracted from selected querns with an electric drill fitted with a diamond-embedded core bit. Two key zones, the center of the working surface and the tool's edges, were preferred for the drilling process. The center of the objects was presumed to be the working area and the margins of the objects were assumed to be unworked areas, in order to compare the results. Previous studies advised that a minimum of two holes were enough for the drilling process

(Mclaren & Evans 2002: 131). In this study, a minimum ten holes (approximately 1 cm in diameter by 0.5-1 cm deep) were chosen in order to take enough sample powder. The number of drilling pits depended on the morphology and texture of the rock. Fine-grained igneous rocks such as granite and basalt are harder surfaces to drill. This method requires a diamond-tipped coated saw hole core drill. Indeed, a 12 mm ⌀ drill bit was used for discrete holes in this process (Fig. 3). However, the actual drilling zone width could not exceed 1 mm because of the saw hole core of the bit.

Figure 3. Drilling process and sampling with diamond-tipped coated saw hole core drill bit on sample GLP 14-09.

Due to surface degradation, contaminations and calcareous covered surfaces, the first 2 mm. at least must be removed by scraping to prevent incorrect results (Evans 1990). Taking into consideration Evans' suggestion, even with tougher drill bits, the depth of holes should not exceed 3-5 mm on granite and basalt samples. However, samples of andesite can easily be drilled to more than 1 cm in depth. The drilling process obtained a powdery texture from the rock surface. For proper chemical analysis, at least 5 to 10 gr of powdered sample must be taken from each specimen. In some situations, artifacts from an unsecured context or surface findings need to be evaluated with their associated soil samples and should be taken for decomposition and secure comparison (Evans 1990; McLaren et al. 1991).

Chemical methodology

Detection of organic residues by chemical lipid analysis of pottery samples has shown the persistence of chemicals in mineral substrates (Condamin et al. 1976; Condamin & Formenti 1978; Evershed et al. 1992; Regert et al. 1998). Despite the potential, the same analytical technique for the study of ground stones has been little used. Initially, only grinders and mortars for grain-processing were studied (Formenti & Procopiou 1998; Quigg et al. 2001; Burton 2003; Buonasera 2007).

Chemical analysis of organic residues obtained from archaeological findings can give us information about the prehistoric diet, functions of related structures, or help distinguish between hunter-gatherers and early farming societies. Residual problems, contamination, and handling of samples during and after excavations may affect the accuracy of chemical analysis (Hillman et al. 1993; Malainey et al. 2000). The length of time that passes following the sample extraction process is critical for stable results. Experiments have shown that organic residues extracted by chemical solvents are stable for nearly five years, but this is not valid for fatty acid components. According to McLaren and Evans (2002), it is strongly recommended that rapid GC-MS analysis be carried out after the extraction process to prevent the fatty acid methyl esters being lost or masked by other components (Mclaren & Evans 2002:132).

Residue samples have up to now been extracted with chemical solutions and by several analytical methods (McLaren et al. 1991; Malainey 1999a; Malainey 1999b; Colombini et al. 2009). In the extraction process, water, methanol and chloroform are commonly used (Kates 1986; Christie 1973; Buonasera 2005). The key stage of analysis is extraction of organic components from the samples. In this study, the extraction process lasted for at least 48 hours in a mixture of *hexane, chloroform* and *propan-2-ol* chemical solvents. Following the extraction process, chemical scanning was conducted by GC-MS. Using GC-MS, fatty acids are detectable, which are the major constituents of fats and oils occurring in plants, cereals, animals and other edible species (Quigg et al. 2001).

Instrumentation

The application of Gas Chromatography combined with Mass Spectrometry (GC-MS) technique is commonly used for analysis and quantification of volatile and semi-volatile organic compounds. Using Mass Spectrometry (MS) to identify the various components from their mass spectra combined with Gas Chromatography (GC) is useful for identification of organic components by separating complex mixtures (James & Martin 1952). Each sample has a nearly unique mass spectrum that can be compared with existing GC-MS database libraries and identified by its chemical linear formula. More recently, the application of the GC-MS technique to archaeological findings, such as absorbed lipid residues in pottery, has proved its potential in identifying food species and individual compounds (Malainey 1999b; Mottram et al. 1999). Mechanical grain processing is followed by a chemical transformation of substances inherent to the grain and a change in nutritional value (Stahl 1989: 173).

Close visual examination of the prehistoric Gülpınar ground stone objects did not yield visible residues such as ochre or plaster. Although lacking visible traces, it was considered that invisible residues might be present on our examples. Consequently, the GC-MS chemical scanning technique was selected for the residue analysis instead of histological and morphological methodologies, which allow identification only of the visible traces of organic compounds (Biers & McGovern 1990; Evershed et al. 1990).

All analysis was conducted at Canakkale Onsekiz Mart University, Science and Technology Research Center (COBILTUM). Lipid extracts were analyzed with Thermo Scientific™ Finnigan Trace DSQ Single Quadrupole GC-MS combined with a Finnigan TRACE DSQ mass spectrometer. A Zebron ZB-5MS capillary column [30 m x 0.25 mm (i.d.); film thickness, 0.25 µm] was used. Helium was used as the carrier gas and flow rate was 1.2 mL / min. The maximum interface temperature was 300°C and the ion source temperature was 200°C. Samples (2 µl) were auto injected and the injection temperature was 275°C. The column temperature was ramped up from 10 to 150°C at 30°C per minute and held for 5 minutes, then from 150 to 280°C at 8°C per minute and maintained for 35 minutes. Xcalibur® 2.0 Thermo Electron mass spectrometry data software was used as the instrumentation method. Peak areas were integrated using Thermo Electron software and peaks were identified from the Wiley 7, NIST02 and PMWtox3N mass spectral database and libraries.

Results

Twenty-eight samples from Prehistoric Gülpınar were analyzed for their organic residue. The majority of the grinding stones contained no residue of lipids. GC-MS analyses showed that two objects contained measurable trace amounts of lipid and the single remaining sample contained distinct inorganic chemical compounds. The total amount of fatty acids found was calculated by summing the areas of each sample's peak area (Tab. 2).

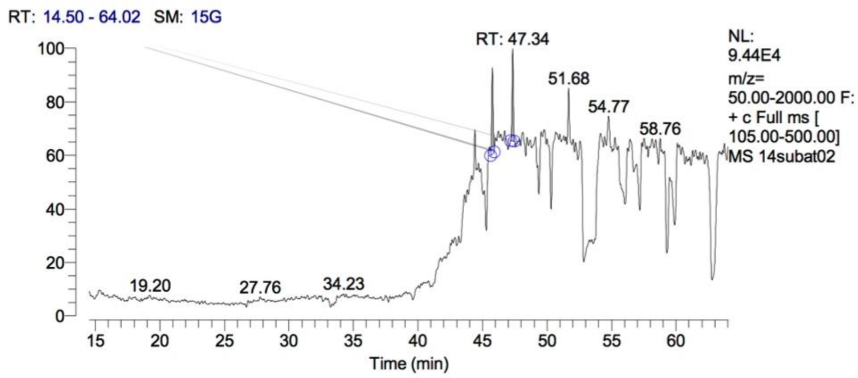
GC-MS analysis results, identifying organic residues consisting of fatty acid compositions found in the chemical composition of essential oils, were determined only in samples GLP 13-01 and GLP 13-02. The grinding surface of a discoidal shaped handstone (GLP 13-01) from level III had the highest proportion of fatty acids (Fig. 4).

Table 2. Organic and inorganic chemical compounds identified on Gülpınar samples.

	Sample No.	*Identified Compounds*	*Peak Area %*
1	GLP 13-03	*8,9,10,11,12,13,14,24,25,26,27,28,29,30-Tetradecahydro-7H,23H - dibenzo[b,p][1,12,15,26]tetraoxycyclooctacosin-6,15,22,31-tetraone*	24.41
		7-deuterio-8-(3-thienyl)-bicyclo-[4.2.0]oct-2-ene-7-ol	14.66
		(E)-2-ethylidene-3-oxo-GA9 16á, 17-epoxide methyl ester	38.56
		1,2-Benzenedicarboxylic acid, bis(2-ethylhexyl) ester (CAS)	22.37
2	GLP 13-04	*Bis[(2,4,6-Tri-tert-butylphenyl)amino]n-butylbromosilane*	100
3	GLP 13-05	*N-(2-Tributylstannyl-2-propen-1-yl)pioperidine*	36.36
		3,4-Dimethyldibenzothiophene	38.05
		1-(3-deuterio-3-methylbutyl)-2,3,5,6-tetramethylbenzene	25.59
4	GLP 13-01	*Pentacosane (CAS)*	50.36
		Heptacosane (CAS)	49.64
5	GLP 13-02	*6-METHYL-CYCLODEC-5-ENOL*	7.28
		Nonanoic acid (CAS)	7.99
		2-hydroxy-4,4-dimethyl-3-(1-methyl-3-oxobut-1-enyl)cycloheptanone	7.13
		Glycylglycylglycylglycine	8.25
		Palmitic acid page 1210 in PMW part 3	19.15
		2(3H)-Furanone, 5-dodecyldihydro- (CAS)	3.20
		2(3H)-Furanone, 5-dodecyldihydro- (CAS)	3.47
		Octadecanoic acid (CAS)	27.92
		4H-1-Benzopyran-4-one, 2-(3,4-dimethoxyphenyl)-5,7-dihydroxy- (CAS)	11.87
		Stigmast-5-en-3-ol, (3á,24S)- (CAS)	3.75
6	GLP 13-06	*(Z)-3-Iodopropenamide*	100.00
18	GLP 14-378	*GLYCEROL-1,2,3-D3, TRIS-O-(TRIMETHYLSILYL)-*	100.00
19	GLP 14-387	*No Data*	
20	GLP 14-389	*9-(3'-phenylpyrazolyl)-9-borabicyclo[3.3.1]nonane*	80.00
		1,5,8,12-Tetraaz-14-selenatricyclo[10.3.2.1(5,8)]octadecane-13,15,18-trione	20.00
21	GLP 14-391	*3-(o-Azidophenyl)propanol*	100.00
22	GLP 14-393	*BENZOFLEX*	100.00
25	GLP 14-438	*ethyl 1-(2-(3,4-dimethoxyphenylethyl)aminomethyl)-5,6-dihydro-8,9-dimethoxypyrrolo[2.1-a]isoquinolin-2-carboxylate*	100.00
28	GLP 14-443	*3(5)-(4'-Chlorophenyl)- 4-nitroso-5(3)-phenylaminopyrazole*	100.00

Figure 4. Working surfaces of samples GLP 13-01, GLP 13-02 and GLP 13-05.

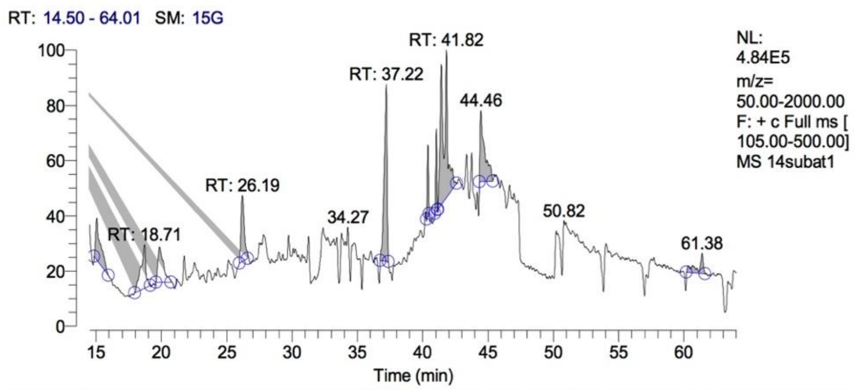
Pentacosane (50.36%) and *heptacosane* (49.64%) were identified in sample GLP 2013-01 as minor and trace constituents in some of the essential oils (Fig. 5). The major fatty acids identified from sample GLP 2013-02 were *octadecanoic (stearic) acid* (27.92%), *palmitic (oleic) acid* (19.15%) and *glycylglycylglycylglycine* (8.25%) (Fig. 6). Surprisingly, the results of analysis also showed evidence of distinct inorganic residue traces. Traces of *Tributylstannyl*, a typical metal compound of tin (Sn), and *Dimethyl Dibenzothiophene,* known as sulfur (S), were identified on the working surface of sample GLP 13-05 (Fig. 7). One of the constituents characterized in sample GLP 13-05 was 4,6-dimethyldibenzothiophene, evaluated as a sulfur compound (Valla 2007).



RT	Name	Library	Molecular Weight	Molecular Formula	Cas #	%
47.34	Heptacosane (CAS)	WILEY7	380	C27H56	593-49-7	49.64
45.76	Pentacosane (CAS)	WILEY7	352	C25H52	629-99-2	50.36

Key: RT: Retention time, Cas: Chemical abstract service

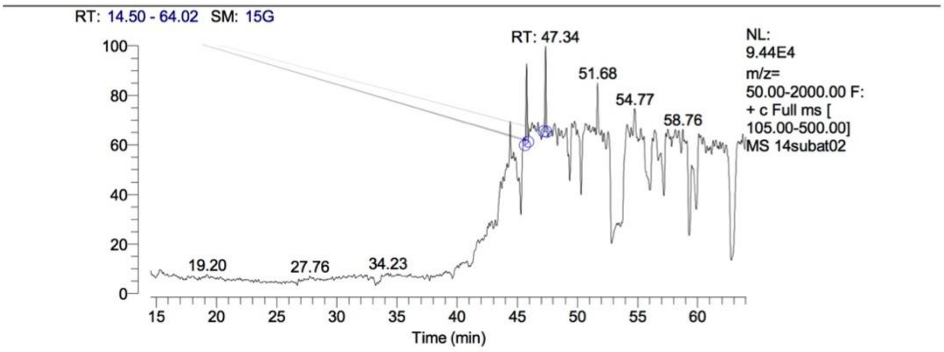
Figure 5. Constituents identified in sample GLP 13-01.



RT	Name	Library	Mol. W.	Mol. Formula	Cas #	%
40.40	2(3H)-Furanone, 5-dodecyldihydro- (CAS)	WILEY7	254	C16H30 O2	730-46-1	3.20
41.05	2(3H)-Furanone, 5-dodecyldihydro- (CAS)	WILEY7	254	C16H30 O2	730-46-1	3.47
61.38	Stigmast-5-en-3-ol, (3á,24S)- (CAS)	WILEY7	414	C29H50 O	83-47-6	3.75
19.87	2-hydroxy-4,4-dimethyl-3-(1-methyl-3-oxobut-1-enyl)cycloheptanone	WILEY7	238	C14H22 O3	125257-74-1	7.13
15.06	6-METHYL-CYCLODEC-5-ENOL	WILEY7	168	C11H20 O	NA	7.28
18.71	Nonanoic acid (CAS)	WILEY7	158	C9H18O 2	112-05-0	7.99
26.19	**Glycylglycylglycylglycine**	NIST02	246	C8H14N 4O5	637-84-3	**8.25**
44.46	4H-1-Benzopyran-4-one, 2-(3,4-dimethoxyphenyl)-5,7-dihydroxy- (CAS)	WILEY7	314	C17H14 O6	4712-12-3	11.87
37.22	**Palmitic acid** (page 1210 in PMW part 3)	PMWtox3 N	256	C16H32 O2	57-10-3	**19.15**
41.82	**Octadecanoic acid (CAS)**	WILEY7	284	C18H36 O2	57-11-4	**27.92**

Key: RT: Retention time, Cas: Chemical abstract service

Figure 6. Constituents identified in sample GLP 13-02.



RT	Name	Library	Molecular Weight	Molecular Formula	Cas #	%
33.61	1-(3-deuterio-3-methylbutyl)-2,3,5,6-tetramethylbenzene	WILEY7	181	C15H23D	116761-77-4	25.59
14.50	N-(**2-Tributylstannyl-2-propen-1-yl**)pioperidine	WILEY7	415	C20H41NSn	NA	36.36
30.63	3,4-**Dimethyldibenzothiophene**	WILEY7	212	C14H12S	23654-30-0	38.05

Key: RT: Retention time, Cas: Chemical abstract service

Figure 7. Constituents identified in sample GLP 13-05.

Discussion of evidence

Residue analyses applied to ceramic samples have shown that these analytical methods have remarkable potential, with ground stone artifacts also, to identify the actual function and use of these objects. These studies demonstrated that organic residues can adhere to the working surface of ground stones during their primary use and survive over a long-time span (Buonasera 2007). These results are helpful in identification of the diverse functions of ground stones other than traditional milling activities. Only limited and pioneering efforts to identify the organic residues on ground stone objects have been made so far yet remarkable achievements have been published (Yohe et al. 1991; Fullagar et al. 1996; Formenti & Procopiou 1998; Procopiou et al. 2002; Baysal & Wright 2005: 308).

Residues are the chemical compounds of materials remaining as lipids, such as fatty acids, that transfer or adhere to tools during use (Fullagar & Matherson 2014). In general, plants, wild cereals and legumes contain some proportion of fatty acids (Harwood et al. 1984; Gunstone 1999). Fatty acids appear in more volatile compounds, giving a flour smell and taste to food by the fermentation process. The released fatty acids can become attached to the mineral material of grinding stones in the form of fatty acid (Procopiou et al. 2002). Heavily-ground cereals, seeds or plants are digested away and preserve only traces in the texture of the grinding stone surface (Hillman et al. 1993). Ethnographic studies have noted that grinding tools were sporadically used in unusual ways for processing foods. Pulverized animal bones were mixed with several foods (Bean & Saubel 1972) and this explains the presence of animal residues such as fatty acids remaining on ground stone tools (Yohe et al. 1991).

The major chemical composition of cereals, crop legumes and weeds is characterized by essential oil compounds such as pentacosane and heptacosane and fatty acids like palmitic acid and octadecanoic (oleic/stearic) acid, which were all identified to a lesser degree on the Gülpınar samples. The results derived from prehistoric Gülpınar datasets (Tab.3) were considered link with archaeobotanical data obtained from other prehistoric settlements nearby, namely, Kumtepe A (Chalcolithic) and Kumtepe B (Early Bronze Age) and also Early (Troy I-III), Middle (Troy IV-V) and Late Bronze Age (Troy VI-VIIb) levels at Troy (Kromer 2003; Riehl 1999).

Pentacosane and heptacosane were identified on sample GLP 13-01 which are possible compounds of the essential oils of *Malva sylvestris L.* (common mallow), *Cicer arietinum L.* (chickpea), *Berula erecta* (parsley family), *Teucrium cf.botrys L.* (mint family) and *Lamiaceae* (mint family). Pentacosane and heptacosane are both volatile constituents of the *Berula erecta* (parsley family) species (Lazarević et al. 2010). However, archaeobotanical studies have not yet revealed enough seed samples from Neolithic / Chalcolithic Kumtepe A levels, and they have only been found in Bronze Age levels at Troy.

Table 3. Organic compounds of selected samples from prehistoric Gülpınar.

Sample No.	***Compound Name***	**%**	***Possible Compound of essential oils of edible species***	***Seed samples from***
GLP 13-01	***Pentacosane***	50.36	*Malva sylvestris L. (c. mallow)*	Troy VIII (700-480 BC)
			Cicer arietinum L. (chickpea)	Troy VI-VII (1700-1050 BC)
			Berula erecta (parsley family)	Troy VI-VII (1700-1050 BC)
	Heptacosane	49.64	*Teucrium cf.botrys L. (mint family)*	Troy IV-V (2200-1250 BC)
			Berula erecta (parsley family)	Troy VI-VII (1700-1050 BC)
			Lamiaceae (mint family)	Troy IV-V (2200-1250 BC)
GLP 13-02	***Glycylglycyl glycylglycine***	8.25	*Olea europaea L.* (olive)	Troy VIII (700-480 BC)
	Palmitic Acid	19.15	*Linum usitassimum L. (flax)*	Troy IV-V (2200-1250 BC)
			Linum cf. strictum L. (upright yellow flax)	Kumtepe A (ca. 4800-4500 BC) Kumtepe B (ca. 3400-3200 BC)
			Lathyrus ciceral sativus (grasspea)	Chalcolithic Kumtepe A
	Octadecanoic (Stearic) Acid	27.92	*Lens culinaris Medik. (lentil)*	Kumtepe A (ca. 4800-4500 BC) **Gülpınar (ca. 5300-4300 BC)**
			Vicia ervilia (L.) Wild. (bittervelch)	Kumtepe A (ca. 4800-4500 BC) **Gülpınar (ca. 5300-4300 BC)**
			Ficus carica L. (fig)	Kumtepe A (ca. 4800-4500 BC)

Palmitic (stearic) acid is one of the chief components of seeds oils obtained from *Lathyrus ciceral sativus* (grass pea), *Linum usitassimum L.* (flax) and *Linum cf.strictum L.* (upright yellow flax) identified on sample GLP 13-02. Flax is one of the major prehistoric crop-plants, which has been cultivated since the 7th millennium BC (van Zeist 1985). Investigation of the fatty acids in flax seed oil (*Linum usitatissimum L.*) has determined that linoleic and linolenic acids were predominant. Flax seed oil contains also palmitic and stearic acids in lesser quantities (Kikalishvili et al. 2014).

Wild oily flax seeds were used in oil production (Stewart 1976) and it has also been suggested that they were used for textile production on the basis of fiber residues recovered from Neolithic Catalhöyük (Ryder 1965; Barber 1991). Ertuğ (2000) stated that if flax is harvested by sickle it is intentionally cultivated for its oily seeds. Ethnographic studies have shown that the oil obtained from flax seeds had multiple usage in animal husbandry, cooking, lighting or folk medicine (Ertuğ 2000). For instance, linseed oil was used to rub on the hides of cattle to provide a protective shield against insects and prevent cracked skins during harsh weather conditions. Preliminary archaeozoological studies have revealed that the faunal remains of *Bos taurus* (cattle) from Gülpınar show us that animal husbandry was an important activity of the prehistoric settlers.

In order to establish the nutritional as well as medicinal properties of *Cicer arietinum L. (chickpea)*, GC-MS analysis determined pentacosane and octadecatrienoic acids in both leaves (Gatade et al. 2013). The species *Lathyrus ciceral sativus* (grass pea) also contains linoleic - palmitic acid types (Bağcı et al. 2004). Mass finds of *Lathyrus ciceral sativus* (grass pea) seeds from prehistoric settlements are widely known from Anatolia, the Balkans and Greece (Renfrew 1977; Halstead & Jones 1980; Miller 1991; Nesbitt 1996). Moreover, vast amounts of *Lathyrus* seeds were also found in the Chalcolithic layers of Kurucay Höyük in SW Turkey (Nesbitt 1996).

In contrast, *Lathyrus* is widely assumed to be a weed among other bitter vetch species, which adapted itself as a consequence of cultivation or soil management from Neolithic to the Late Bronze Age (Riehl 1999: 65). *Lathyrus* seeds from Kumtepe fall within the range of Bronze Age samples found at Dimini according to seed-size differences (Kroll 1979). Nevertheless, according to the seed sizes of the Kumtepe samples, the *Lathyrus* seeds evaluated, as bitter vetch is more suitable.

Gas chromatography, used for analyzing the main fatty acids of *Olea europaea L.* (olive), has shown us that the composition of the olive contains palmitic and oleic acids (León et al. 2004), which were identified in sample GLP 13-02. Furthermore, the volatile components of fig fruit were also identified through GC-MS and showed the presence of oleic acid (Soni et al. 2014).

Overall, the results supported the presence of organic residues on the working surface of the ground stone tools retrieved from Chalcolithic Gülpınar. Minor portions of carbonized grains and seeds from Phase II and III were identified during excavations. Einkorn wheat (*Triticum monoccocum*) and emmer wheat (*Tricutum dicoccum*) were the dominant cereals found at the settlement. Small amounts of a crop legume mixture of bitter vetch (*Vicia ervilia*) and lentil (*Lens culinaris*) were also obtained from soil samples using the flotation technique (Takaoğlu 2006: 311).

The oily seeds of *Vicia* (bitter vetch) contain palmitic and stearic acids, which are the major component of well-known fatty acids (Kökten et al. 2010). The oily seeds of *Vicia* species such as *Vicia ervilia (L.) Wild.* (bitter vetch) also have large amounts of octadecanoic (oleic) acid, also identified on sample GLP 13-02 (Akpınar et al. 2001). Furthermore, *Lens culinaris Medik. (lentil)* seeds contain fatty acids such as dodecenoic and octadecanoic acids (Gallasch et al. 2000).

Recent archaeobotanical studies in the Troad have revealed that the range of cereal and legume species recovered from Gülpınar exhibits strong links with Kumtepe IA samples, supported by the results of our residue analysis (Riehl 1999: 27). In contrast to the vast majority of samples from the Bronze Age levels of Troy and Kumtepe B, Kumtepe A is represented with only 7 samples, which limits us in revealing the archaeobotanical profile of that site.

The use of grinding stones and hand stones at prehistoric Gülpınar has primarily been attested for food preparation. All ground stone finds reflecting the daily activities of Prehistoric Gülpınar settlers indicate that they practiced an agro-pastoral sedentary economy based on farming and herding, as suggested by archaeozoological and archaeobotanical evidence. The proposed archaeozoological and archaeobotanical evidence at Gülpınar was elucidated by the residue analysis in this study. Residue analysis has not previously been widely carried out on ground stone tools. Additional improved analyses should be conducted in order to identify the actual function of ground stone objects rather than assumed functions. Study of the preparation and processing of cultivated crops related to grinding activities, combined with chemical analysis, provides a new approach for learning about the dietary systems of prehistoric societies. Furthermore, this kind of study may provide us with a valuable record of the grinding processes and technologies of daily life in prehistoric societies.

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