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FIRST RESULTS OF ORGANIC RESIDUE ANALYSIS ON CERAMIC VESSELS (JIYEH AND CHHÎM, LEBANON) BY HIGH PERFOMANCE LIQUID CHROMATOGRAPHY WITH TANDEM MASS SPECTROMETRY

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ABSTRACT

The aim of this paper is to present the first results of organic residue analysis of pottery from two Lebanese archaeological sites, Jiyeh and Chhîm. The standard approach in investigations of this type consists of mass spectrometry and gas chromatography technique, which enabled the detection of organic fractions absorbed by pores of the pottery. In the present paper, new method of liquid chromatography and tandem mass spectrometry has been used in order to identify organic fractions. Sixteen fragments of vessels from different forms, periods and archaeological contexts have been analysed. The selected samples came from three vessel categories, kitchen vessels for cooking and store liquids, transport amphorae and storage *pithoi*, dated from Persian to Byzantine period. The procedure of sample extraction and HPLC-MS/MS analysis allowed to find vessels with the potential traces of the use for food product preparation and/or storage and separate it from different vessels (without traces of organic residues). Organic substances have been detected in the majority of the investigated samples.

KEYWORDS: organic residue analysis, liquid chromatography, tandem mass spectrometry, cooking vessels, amphorae, *pithoi*, Porphyreon, Chhîm.

1. INTRODUCTION

Analyses of the organic residue preserved in ceramic vessels lie within the scope of such fields as biomolecular archaeology and archaeometry. They are also influenced by such disciplines as biology, genetics, chemistry, physics, zoology or botany, and they explore issues linked, among other things, to ancient DNA analyses, isotopic analyses or the study of lipids, carbohydrates or proteins preserved in ancient material (Brown and Brown 2011). However, studies of the traces of organic substances found on pottery have a much longer tradition and include both the analyses of substances preserved as surface residual crust and that of residue absorbed by the walls of ceramic vessels (Oudemans 2007; Steele 2013; Mukherjee *et al*. 2008; Evershed 2008; Craig, Collins 2002). Pioneer attempts at recognizing such surface deposits as wood tar were undertaken as early as in the 19th century (among others, by Reichenbach, Kekule, Landolt) and focused primarily on the observation of the physical properties of the preserved remains, e.g. their melting points (after Pietrzak 2010). Attempts to identify the contents of pots were also conducted at the beginning of the 20th century, among others by Marcellin Berthelot (1906), who identified oleic, palmitic and stearic free fatty acids from two Gallo-Roman vessels. In turn, the bituminous substances found during L.C. Woolley's excavations in Ur became the subject of the examination conducted by Hackford *et al*. (1931) and Forbes (1936), who distinguished the particular fractions by using simple separation and filtration techniques. In 1933, Grüss determined the origin of some blackish matters on a Hallstatt vessel and labelled them as remains of overcooked milk, using so-called wet chemistry techniques (Grüss 1933). However, the turning point in research into organic residues came with the introduction of mass spectrometry and gas chromatography, which enable the identification of organic fractions not visible on the surface of the artefacts but absorbed by their pores. The first trials were conducted in the 1970s and 1980s (including by Thornton *et al*. 1970; Condamin *et al*. 1976; Mills and White 1977; Rottländer and Hartke 1982; Evans and Hills 1982; Shackley 1982), but it became widely used in the 1990s, when a specialized analytic protocol was developed enabling the examination of even a small amount of organic residue, primarily preserved lipids and waxes (Evershed 1993; 1998; Evershed *et al*. 1990). This included a series of steps, beginning with the acquisition of a pottery sherd subsample through lipid extraction, the evaporation of the obtained mixture, its saponification, re-evaporation, dissolution with chloroform, and then its screened by gas chromatography (GC) and mass spectrometry (MS) or gas chromatography-combustion-isotope ratio mass spectrometry (GC/C/IRMS) (e.g. Evershed 2008) or a liquid chromatography with a mass spectrometry (among others, Guasch-Jane *et al*. 2004; Hurst *et al*. 2002). The obtained results were then compared to reference material consisting of known compounds and identified using such means.

Among the many problems linked to recognition of the particular organic compounds and their classification, the most important include degradation of the original organic substances and contamination usually occurring as a result of post-deposit processes or during the acquisition of the archaeological material and its later storage (Evershed 2008; Oudemans 2006). Distinguishing between exo- and endogenic substances is usually limited to the mechanical removal of a thin layer from the surface of the vessel before subjecting the material to further analysis (Evershed 2008; Correa-Ascencio; Evershed 2014; Copley *et al*. 2003) or – more rarely – a separate analysis of the substances taken from the outer and inner vessel surfaces (Stern *et al*. 2000; Steele *et al*. 2007), and from LM stirrup jars (Koh & Birney 2017).

The objective of the research undertaken by our team is identifying whether organic residue has been preserved in the pores of the vessels found during excavations at Chhîm and Jiyeh, Phoenician sites, and conducting an attempt at their identification by using an innovative analytic protocol incorporating liquid chromatography and tandem mass spectrometry. The proposed analytical procedure includes also a new way of sampling the potsherds enabling to discern between the organic compounds of prehistoric origin and the modern contamination.

2. THE ARCHAEOLOGICAL MATERIAL AND CONTEXT

The analysed pottery material comes from two archaeological sites, Jiyeh and Chhîm, situated in ancient Phoenicia, currently in central Lebanon.[1] These sites are located *ca*. 30 km south and south-east of Beirut (Fig. 1).

[1] Excavations have been conducted since 1996 by the Polish Archaeological Mission, ***Polish Centre of Mediterranean Archaeology***, University of Warsaw, supervised by T. Waliszewski, in cooperation with the Lebanese antiquity services from the Direction General des Antiquites et Musees. Urszula Wicenciak is participating in these projects since 1999 and she is in charge of the pottery studies at the Jiyeh and Chhîm.

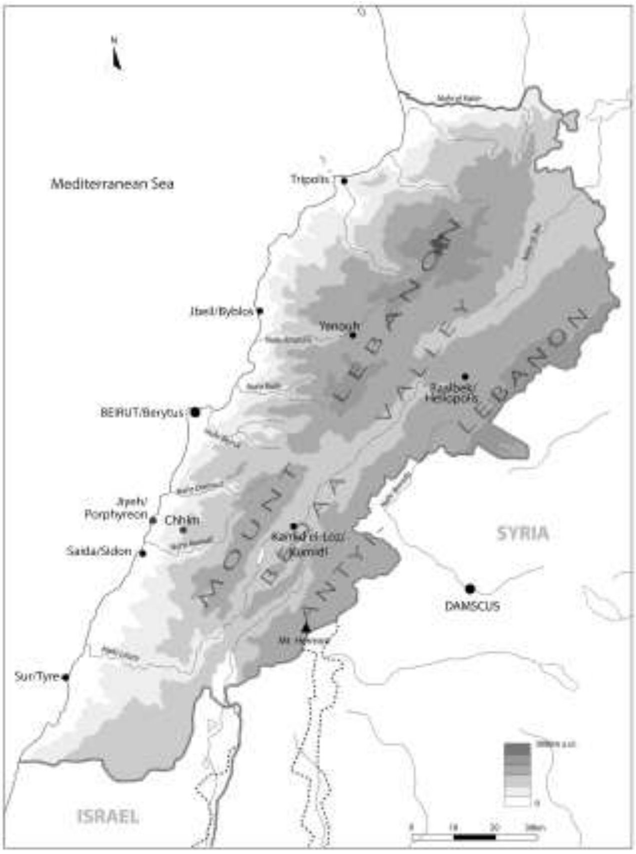

Figure 1. Map of Lebanon (U. Wicenciak)

The remains of one of these ancient settlements is located on the outskirts of the modern-day village of Chhîm in the Chouf Mountains (a Lebanese mountain range) (Waliszewski, Wicenciak 2015a). The present-day coastal village of Jiyeh, about 10 km west of Chhîm, houses the remains of buildings thought to be linked to the ancient settlement of Porphyreon (Waliszewski, Gwiazda, 2015; Waliszewski, Wicenciak 2015b).

The results of archaeological research supported by analysis of the material from these sites indicate that both places had been settled with some interruptions from the Late Bronze Age to the end of the Byzantine period. In addition, studies into the written sources, the character of the buildings and the archaeological material with reference to the neighbouring coastal metropoles, Berytus and Sidon, incline towards putting forward the hypothesis that both settlements were located within the area under the administration of Sidon (Wicenciak 2016; Gwiazda 2014). The settlements differed in terms of their purpose: agricultural in the case of Chhîm – focusing on olive cultivation and olive oil production (Waliszewski 2014), and handcraft in Porphyreon – concentrated primarily on the production of pottery (Wicenciak 2014; 2016) and glass vessels, metallurgy, and other forms of craftwork (Gwiazda 2014).

3. SAMPLING

Sixteen fragments of vessels diverse in terms of their chronology, find context, pottery workshop and function were subjected to analyses using liquid chromatography. The aim of choosing diversified material is to broaden the potential of identifying organic residue in the pores of the vessels which could have had contact with food. That is why the selected samples came from three vessel categories: **1**. kitchen vessels for cooking and those used to store liquids (samples: 4-6; 11-16); **2**. transport amphorae (samples: 1-3, 8, 10); **3**. pithoi – vessels used primarily for storing products[2] (samples: 7, 9, 10) [Table 1]. The chronology of the sherds is not as important as the will for testing a new method which could be used for archaeological research. Moreover, the selection of different type of pots in terms of technology, morphology and function would be potentially useful in the tracing of the relation between the presence of a particular type of organic substances and specific category of pots. Additionally, the taking such varied material gives a chance in testifying the proposed analytical method and better demonstrate its limitations and potentials.

As the idea to conduct the analyses only developed after the termination of the excavations in the 2014 season, the sample selection was dependent on the material available within the "Polish collection" brought to Poland for research purposes with the permission of the DGA in 1999-2013.

Such methods of analysis were applied on vessel sherds from excavations conducted in the Jiyeh "production zone" (sector B) (Wicenciak 2014; 2016). Deposits containing production waste were uncovered there in 2004, i.e. vessels and fragments destroyed during the baking process (Waliszewski *et al.* 2010). Theoretically, these vessels had never been used and thus should not have borne any traces of organic substances. The sherds were to serve as a means of verifying the aptness of the applied method. This group includes kitchen vessel sherds (Samples: 4-6), as well as fragments of pots and pans, produced in Porphyreon in the Early Roman period (Early Roman Jiyeh Ware) (Fig. 2) (Wicenciak 2014; 2016).

[2] Z. Kowarska's PhD dissertation focuses on this category.

Table 1. List of vessel fragments from Chhîm and Jiyeh subjected to analysis.

Sample no	Cat. no	Site	Form	Dating
1	JY2009/874	Jiyeh/Porphyreon	Amphora	Persian/Hellenistic
2	CHM 2730	Chhîm	Amphora	Byzantine
3	JY2008/590	Jiyeh/Porphyreon	Amphora	Hellenistic
4	JY2004/132_B4	Jiyeh/Porphyreon	Cooking pot	Early Roman
5	JY2004/134_B4	Jiyeh/Porphyreon	Cooking pot	Early Roman
6	JY2004/126_B4	Jiyeh/Porphyreon	Cooking pot	Early Roman
7	JY2010/1418	Jiyeh/Porphyreon	*Pithos*	Late Roman/Byzantine
8	CHM 6117	Chhîm	Amphora	Hellenistic
9	CHM 6115	Chhîm	*Pithos*	Late Roman/Byzantine
10	CHM 2105	Chhîm	*Pithos*	Late Roman/Byzantine
11	CHM 2058	Chhîm	Cooking pot	Late Roman
12	CHM 406	Chhîm	Krater	Early Roman
13	CHM 2711	Chhîm	Cooking pot	Byzantine
14	CHM 648	Chhîm	Cooking pot	Byzantine
15	CHM 2700	Chhîm	Cooking pot	Byzantine
16	CHM 2067	Chhîm	Casserole	Byzantine

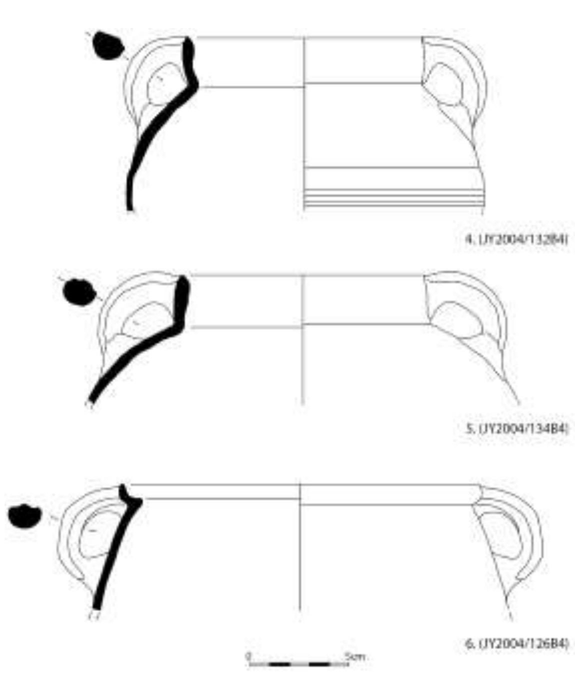

Figure 2. Kitchen vessels of Early Roman Jiyeh Ware from Porphyreon (Drawing and digitizing M. Puszkarski)

The second group of selected vessel sherds comes from excavations in the ancient village in Chhîm, i.e. from settlement contexts. The layers from which they were taken and the analogies for the individual types date these vessels to the Roman and Byzantine periods, from the 3rd to the beginning of the 7th century AD. These sherds bear traces of use, with visible traces on the outer wall surfaces of being blackened by smoke and burnt in the course of meal preparation. Macroscopic analyses of fabric indicate that these are products from two different regions/production zones. The first is located within the territory of Upper Galilea/northern Israel, in Tell Keisan or in its vicinity, *ca.* 9 km east of Akko/Ptolemais. This production was termed *Workshop X* and is considered to belong to the category of *brittle cooking ware* (Waksman *et al.* 2005; Reynolds, Waksman 2007). Both closed cooking forms (pot and casserole sherds) and open ones make up part of this group (Samples: 13-16) (Fig. 3).

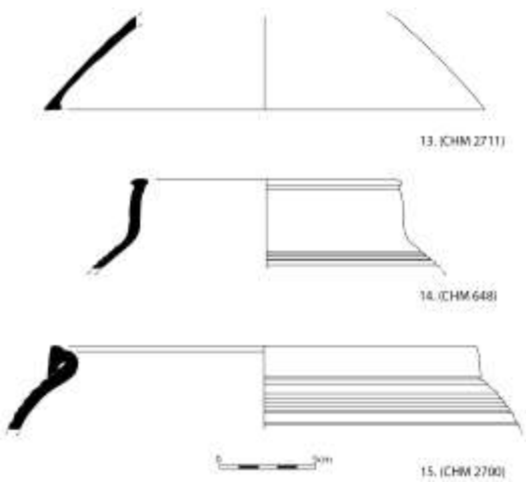

Figure 3. Kitchen vessels of Workshop X production (Drawing M. Chrapek, digitizing U. Wicenciak)

The second production zone is situated in the southern part of the Bekaa Valley, while its products are described in archaeological literature as CW 34 products (Reynolds, Waksman 2007: 59-62). This production is represented by closed cooking pot (Sample 10) (Fig. 4), and fragments of *pithoi* (Fig. 10).

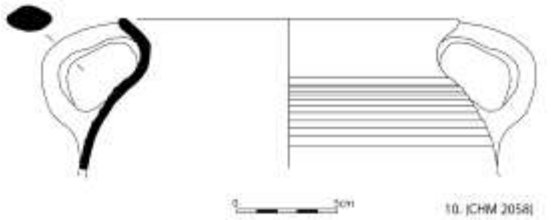

Figure 4. Cooking pot of CW 34 production (Drawing M. Chrapek, digitizing U. Wicenciak)

The category of kitchen ware includes a krater fragment, i.e. a vessel originally used to store liquids (Sample 12) (Fig. 5). The results of the macroscopic analysis indicate that this is a product of a Porphyreon workshop from the Early Roman period (Wicenciak 2014, crater type 4; Wicenciak 2016).

Figure 5. Krater of Early Roman Jiyeh Ware from Porphyreon (Drawing and digitizing M. Puszkarski)

Yet another vessel category consists of amphorae, i.e. containers for transporting and storing various kinds of products (Samples: 1, 2, 3, 8). These fragments come from Chhîm and Jiyeh, and they are diverse both in terms of their chronology and their workshop (in the sense of the place of production). One of the sherds is a fragment of a so-called *carinated shoulder amphora*, a form characteristic for the Phoenicia area produced in the Iron Age near Tyre and Sidon (Sample 1) (Fig. 6).

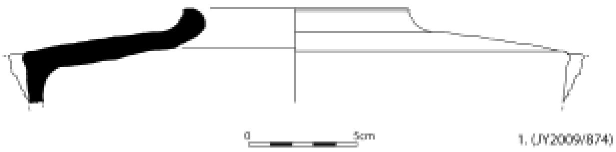

Figure 6. South Phoenician type amphora from Persian period (Drawing and digitizing M. Puszkarski)

The three subsequent samples come from amphora sherds dated to the Hellenistic period (Samples 3 and 8) (Fig. 7), the production of which is linked to the Sidon and Porphyreon region. One fragment belongs to the most popular type in the Mediterranean Basin in the Roman and Byzantine periods, i.e. *Late Roman Amphora 1* (Sample 2) (Fig. 8).

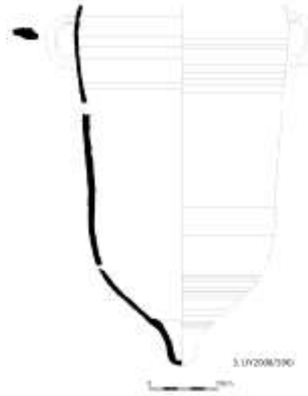

Figure 7. Sidonian type amphora from Hellenistic period (Drawing and digitizing M. Puszkarski)

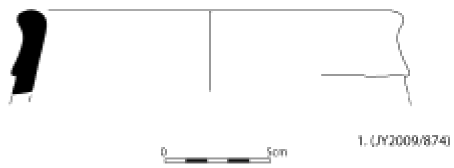

Figure 8. Late Roman Amphora 1 type (Drawing M. Chrapek, digitizing U. Wicenciak)

The third category consists of storage vessels (*pithoi*) used both in Antiquity and in modern times for the storage of food, but primarily of olive oil. The samples subjected to analyses come from excavations conducted at Chhîm and Jiyeh (Samples: 7, 9, 10).

Two storage vessel types were chosen for the analysis. The first (Sample 7) (Fig. 9) is massive, with a rim in section similar in shape to a triangle, no distinguished neck, a flat base and decorated with dots or/and wavy or straight lines made using a comb (cf. vessel reconstruction). Macroscopic observations of fabric indicate similarities to ceramic products from Amrit (Paul Reynolds – pers. comm.; Reynolds 2014: 59, fig. 6 d; Kowarska and Lenarczyk 2014: 129). The contexts of this vessel type found in Chhîm and Jiyeh, as well as in the case of analogous finds from present-day Israel and Syria, indicate their approximate dating to the Late Roman/Early Byzantine period, i.e. the 6th – 7th century AD (cf. Hayes and 'Ala' Eddine 1998-1999: 131, 134, 135, fig. 7; Florimont 1984: 29-30; Ayash and Ganor 2009: fig. 1-3; Frankel, Getzov and Aviam 2001: 68, Fig. 3.11:13; Sa'id 2009: fig. 3:16; Amir 2006: fig. 3.2-4; Calderon 2010: 183, fig. 1.1, 187, fig. 3:27, 195 fig. 7:69; Smithline 2007: fig. 14:1–5; Abu-'Uqsa 2007: fig. 4:12; Dayan et al. 2013: fig. 15:4; Mills 2014: 29-31, fig. 20, N 36, Reynolds 2014: fig. 6 d).

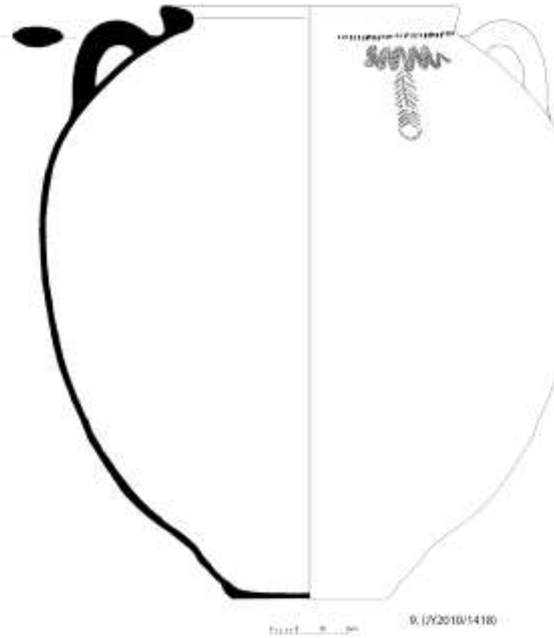

Figure 9. Late Roman pithos from Amrit(?) Drawing and digitizing Z. Kowarska)

The second type (Samples 9 and 10) (Fig. 10) is represented by a fragment of rim straight, slightly everted rim and a "spiral" base. Fabric of this type shows certain similarities with 'kaolinitic fabrics' (CW 34) (Paul Reynolds - pers. comm.; Kowarska and Lenarczyk 2014: 129, cf. Reynolds and Waksman 2007, 'CW 34'; Wicenciak 2012: 136). Analogies for this vessel type include the so-called *black pithoi* found in Upper Galilee (Frankel, Getzov and Aviam 2001, 68, Figs. 3.11:18-22) and *Golan- type pithoi* (Cf. Zingboym 2011: Fig. 4:16–17; Smithline 2006: Fig. 12:21–24; Jaffe 2010: Fig. 5:5), dated to the Late Roman/Byzantine period.

The agricultural and production activities conducted in Chhîm in the Late Roman period indicate that the storage vessels found at the site served to store the olive oil produced there (Waliszewski 2014: 234-235, 288, 419-443).

4. SAMPLE PREPARATION AND ANALYSIS

The pottery sherds selected for analysis came from well-excavated "safe" contexts. The tests were applied to distinctive vessel fragments, primarily the upper parts near the rim and the middle parts of the body, to the exclusion of any vessel sherds that had been secondarily fired or cleaned using aggressive detergents. It was also essential in the course of the tests to choose a wide range of vessel forms and adapt the sample selection to specific types of vessel form.

Following careful mechanic cleaning, pottery sherds were divided into layers in accordance with the vessel shapes: outer, mid (constituting the core of the pottery specimen) and inner layers. The outer and inner ones were separated by grinding them with a corundum grinding wheel set on a pillar drill (600-1000 rpm, Bosch), while the remaining mid layer was ground with an agate grinding mill (Retsch).

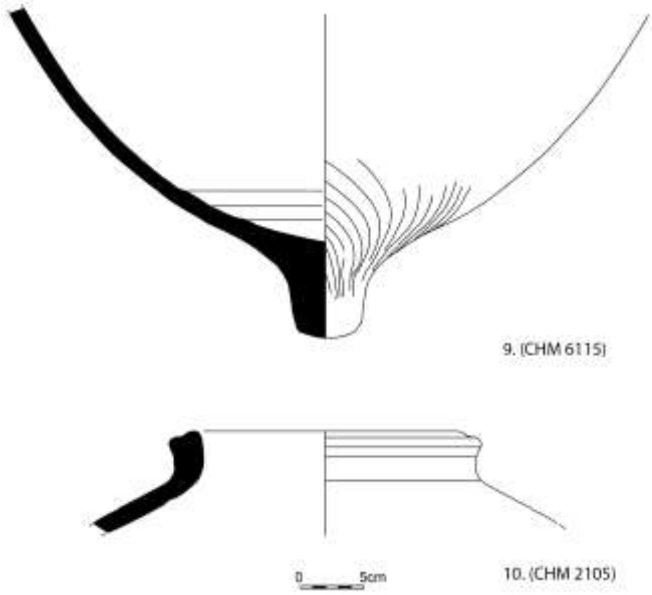

Figure 10. Base and rim of pithoi of CW 34 production (Drawing and digitizing Z. Kowarska)

1.00 g of the sample from each of the layers of the particular analysed vessel sherd was extracted onto an ultrasound cleaner for 30 minutes using 20 ml of chloroform. Next, the extract was vaporised until dry in an air stream. The dry residue was then dissolved in 1.00 ml of chloroform and the sample analysed. This was done using a WatersAquity UPLC chromatograph with a tandem mass spectrometer as a detector. The analysis conditions: a PursuitDiphenyl (150 x 2.0 mm in diameter, 3 µm) analytic pillar; room temperature; the mass range: 100-700 m/z; ESI; mobile phase: 0.1% acetonitrile: water (1:1); flow: 0.5 ml/min; injection volume: 3 ul; cone voltage: 34 V.

Sixteen pottery sherds were subjected to analysis, each of which were divided into three layers, so as to obtain 48 mass spectra. A blind test of the dissolvents used in the extraction procedure was applied to provide control samples, while additionally an analysis of modern ceramic material that has never had contact with food was also conducted. To enable the interpretation of the spectra, only signals of an intensity greater than 10% of the most intense signal were taken into account, and also signals shared with the control samples. Additionally, the signals shared with the outer layer were removed for each of the samples from all of the acquired mass spectra, as this part of the vessels was treated as potentially lacking any contact with the vessel contents. The thus reduced mass spectra became the basis for further considerations.

The chromatographic analysis has been not focused on the specific group of compound determination. At this stage of studies, the proposed procedure may be used for selection of the samples intended for the next analysis based on the recognition of the specific organic compounds to attribute to organic residues (e.g. oil, milk, dairy products). The simple procedure of sample extraction and HPLC-MS/MS analysis allowed to find vessels with the potential traces of the use for food product preparation and/or storage and separate it from different vessels (without traces of organic residues).

5. RESULTS

Out of the 48 obtained mass spectra, 10 did not contain analytic signals. For the following samples, i.e. 14 (CHM 648) and 11 (CHM 2058) (the mid and inner layers), 15 (CHM 2700) (the outer and mid layers), 2 (CHM 2730) and 6 (JY2004/126B4) (the outer layer), 3 (JY2008/590) (the mid layer) no organic residue was identified.

For many of the samples, i.e. 12 (CHM 406), 13 (CHM2711), 9 (CHM 6115), 8 (CHM 6177), 4 (JY2004/132B4) (all the layers); 7 (JY2010/1418) (the inner layer); 16 (CHM 2067), CHM 2731, 5 (JY204/134B4); 1 (JY2009/874), 10 (CHM 2105), 15 (CHM 2700), 2 (CHM 2730), 6 (JY2004/126B4), 3 (JY2008/590) (the inner layer), the obtained analytic signals were not of a very high intensity.

In turn, the highest amount of analytic signals were identified for samples 11 (cooking pot CHM 2058) (the outer surface), 10 (*pithos* CHM2105) and 2 (amphora CHM2730) (the inner surface).

<u>Sample 1 (JY2009/874)</u>

In the case of this vessel, a series of low intensity signals for the outer and inner layers were ascertained. Two analytic signals at m/z 136 (very intense) and 137 appeared in the case of the vessel's core.

<u>Sample 2 (CHM 2730)</u>

The presence of organic compound residue was not established for the outer layer of the analysed pottery sherd, while only a single analytic signal at m/z 138 was registered for the mid layer. Over a dozen analytic signals of a low intensity were registered for the inner vessel layer, grouped within a range of between m/z *ca*. 130 and *ca*. 200.

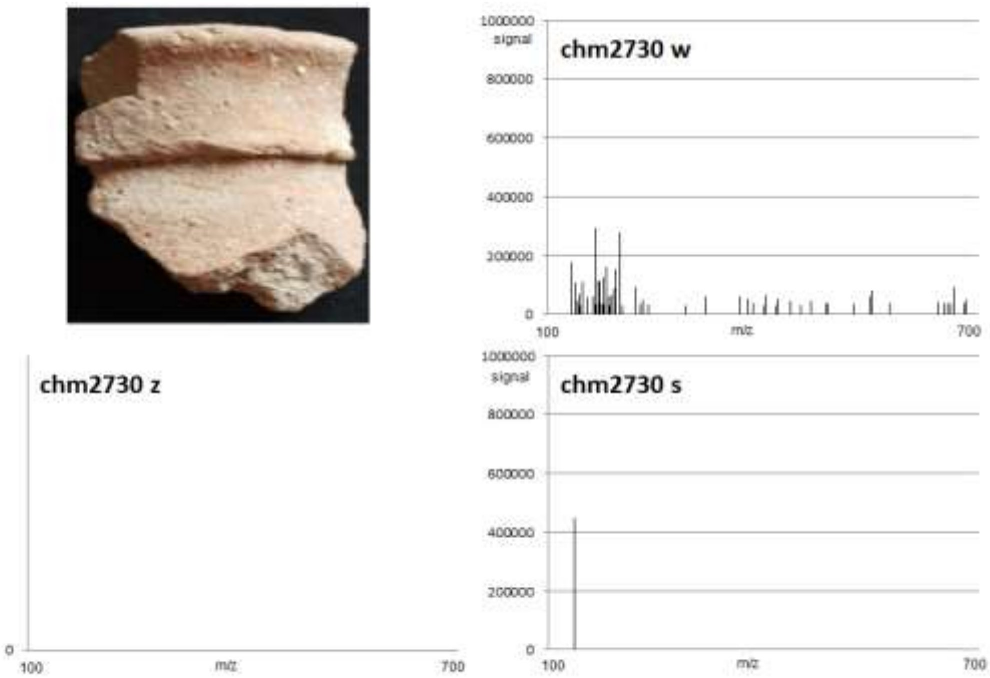

Figure 11. Chromatogram of sample 2.

<u>Sample 3 (JY2008/590)</u>

Two analytic signals were registered for this vessel's outer layer (an intense one at m/z 266) and one low intensity signal for the inner layer. No analytic signals were registered for the vessel's core.

<u>Sample 4 (JY2004/132B4)</u>

Similarly as in the case of the previous vessels, analytic signals of a relatively low intensity were registered for all the layers.

Figure 12. Chromatogram of sample 4.

Sample 5 (JY2004/134B4)

In the case of this vessel, about 30 analytic signals were registered for the outer layer; however, their intensity was relatively low. There were three signals of a quite low intensity for the mid layer, while two signals of a relatively high intensity at m/z 195 (the more intense one) and 435 were established for the inner layer.

Sample 6 (JY2004/126B4)

No analytic signals were registered for the outer vessel layer, while for the inner one only two signals of a quite low intensity. In turn, a single intense signal at m/z 214 was observed for the inner layer.

Sample 7 (JY2010/1418)

A single low intensity analytic signal was identified in the outer vessel layer. Three analytic signals were registered for the mid layer, of which the signals at m/z 282 and 283 were the most intense. Three analytic signals were also ascertained for the inner vessel layer, one of which at m/z 378 was very intense.

Sample 8 (CHM 6117) and Sample 9 (CHM 6115)

In the case of these two vessels, very few analytic signals were ascertained for all the ceramic layers, and only a single analytic signal for the mid layer.

Sample 10 (CHM 2105)

Many analytic signals of a low intensity were ascertained in the outer and inner vessel layers, with its highest value at m/z 394 for the outer layer. Only two analytic signals were identified for the core, at m/z 138 and a very intense one at m/z 136.

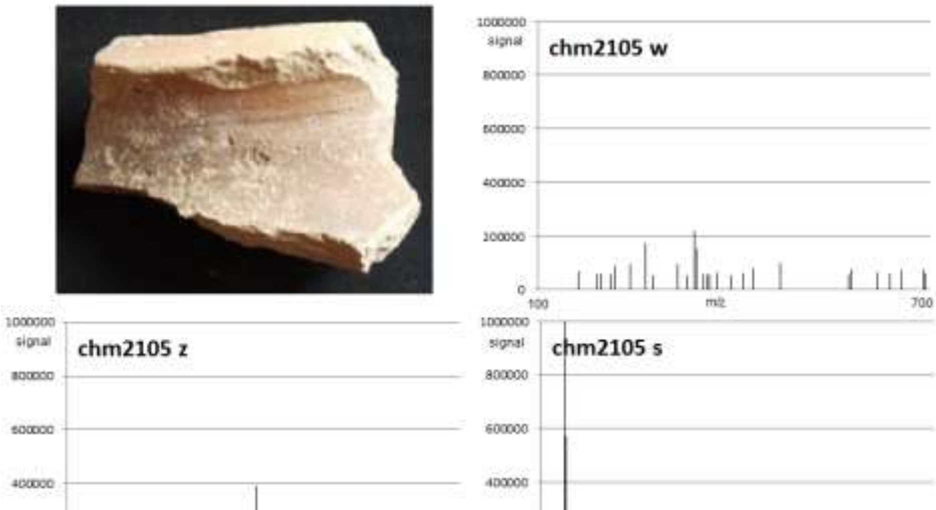

Figure 13. Chromatogram of sample 10.

Sample 11 (CHM 2058)

Organic compound residues were only ascertained on the outside of the vessel, with the analytic signal intensity at a range of between m/z *ca*. 400 and *ca*. 500; the presence of organic compounds was not ascertained in the remaining layers of the analysed pottery sherd.

Sample 12 (CHM 406)

Individual analytic signals of a low intensity were obtained for the vessel outer and mid layers. Similarly, signals of a low intensity were obtained for the inner vessel layer, with over a dozen analytic signals identified within a range of m/z 130-200. Generally, organic compound residues were ascertained for the inner parts of the vessel.

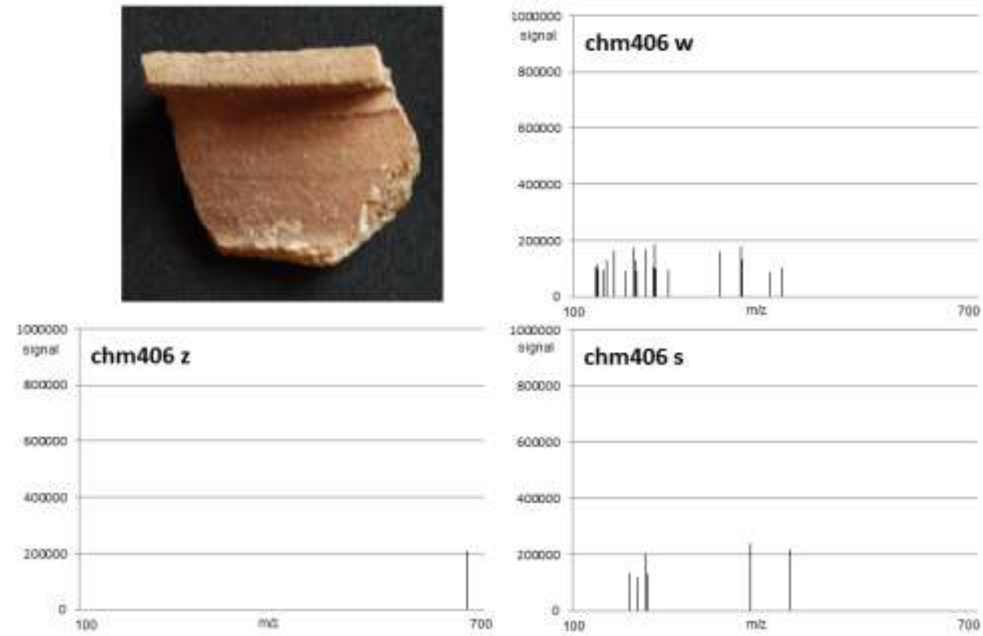

Figure 14. Chromatogram of sample 12.

Sample 13 (CHM 2711)

Analytic signals testifying to the potential presence of organic compounds were ascertained in all the layers of the analysed vessel; however, the intensity of the analytic signals was low.

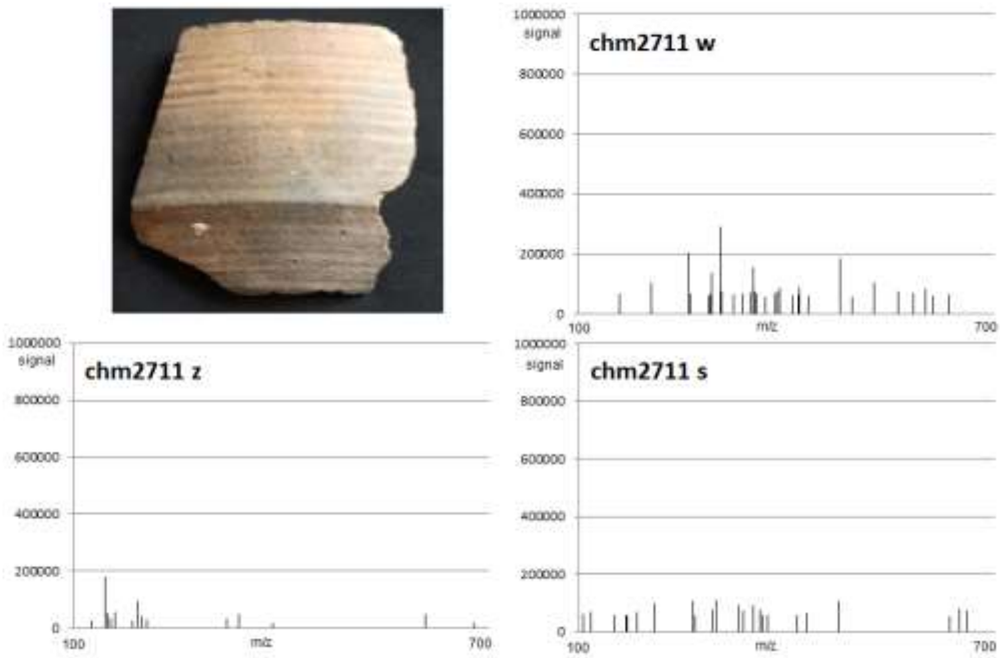

Figure 15. Chromatogram of sample 13.

Sample 14 (CHM 648)

Only two analytic signals at m/z 266 and 308 were observed in the case of the outer vessel layer; no analytic signals were registered for the remaining vessel layers. Therefore, it can be stated that the presence of organic compounds were not ascertained in the case of this vessel.

Sample 15 (CHM 2700)

The presence of analytic compounds was not ascertained either for the outer layer or for the core of the vessel. The presence of over a dozen analytic signals of a low intensity was registered for the inner layer.

Sample 16 (CHM 2067)

In the case of all of the vessel layers, the presence of over a dozen analytic signals of a low intensity was ascertained, with one strong signal at m/z 265 for the inner layer, for which additionally the lowest amount of analytic signals were confirmed. Organic compound residue was thus present in all the layers of the analysed pottery fragment.

6. CONCLUSIONS

The conducted analysis has shown the presence of organic substances in the majority of the analysed samples. Taking into account those vessel layers that might potentially come in direct (the outer layer) or indirect (the core – inner layer) contact with the food stored or prepared in the vessel, the samples can be divided into two groups:

- vessels for which organic residues were ascertained,
- vessels lacking any traces of organic compounds (Sample 11 and 14) or with faint traces of their presence (Samples 3, 6, 8, 9)

Organic compounds can be observed in all the vessel forms the samples were taken from, i.e. amphorae, pots, casseroles, storage vessels and craters. There are no dependencies between the vessel types, chronology, places of origin and the presence of organic compounds. The presence of organic compounds in the vessel sherds originating from the production zone in Jiyeh (Samples 4-6), and thus such as had not been used for the preparation and transportation of food, indicate that the registered analytic signals are not linked to food but to other organic substances that might have been present in the soil and which permeated the vessel walls as a result of post-deposit processes. The applied methodology unable to recognize more closely the nature of detected organic compounds. However, the important and undisputed input of proposed procedure is a new strategy of sampling that focuses on three zones of potsherds (inner layer, outer layer and core – mid- part). This gives a chance to track the changes in a history (microhistory) of pot usage by comparison between different sides of the same pot (e.g. discern the substances used for waterproofing the vessels and the content of pot itself). Moreover it also gives an opportunity to determine between modern contaminations and archaeological residues. Thereby, it might be an interesting and useful supplementation of other analytical procedures.

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