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Organic residues analysis of Late Antique pottery from Plaça Major-Horts de Can Torras (Castellar del Vallés, Catalonia, Spain)

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Abstract

Organic residues analysis is an important tool in archaeological research because it sheds light on aspects of the daily life of the past. In particular, it allows us to obtain information on food production, consumption and trade. In this paper we present the results of residue analysis performed on ceramic vessels from the archaeological site of Plaça Major-Horts de Can Torras (Castellar del Vallés, Barcelona, Spain), a long lasting site, of which we studied the Late Antique occupation. The site is a rural settlement located in the northeast of Catalonia and it was characterized by structures made of perishable material and by the presence of silos, above which probably the living structures were placed.

Analyses were carried out on twelve cooking and storage ceramic forms that were the most representative of the Late Antique phase. In particular pots, jugs, pans and *dolia* were analyzed in order to investigate the food consumed at the site. Analyses were conducted by gas chromatography-mass spectrometry (GC-MS). The results show the cooking of animal and vegetal products in the cooking vessels (pots and pan) and the storage of wine in the *dolia*. *Pinaceae* products are particularly abundant in some vessels, which suggests the use of this substance at the site. In general, the analyses carried out provide the first case study of residue analysis of ceramic material of this period in Catalonia and allowed us to recover information both on food habits and on the use of different ceramic forms during Late Antiquity in this area.

Key words: organic residue analyses; food practices; gas chromatography-mass spectrometry; Late Antiquity; pottery.

Introduction

Pottery is one of the most common artefacts in the archaeological record since the Neolithic period. Through time and across a wide geographical area, pottery has been fundamental to fulfil multiple functions such as cooking, storage, transport and eating and drinking. For this reason, studying pottery at archaeological and ethnoarchaeological levels provides basic information for investigating both technological and cultural aspects of human societies.

In studying ceramics, the analysis of organic residues represents an important tool for archaeologists. Not only it allows us to obtain information on culinary practices of the past, but also on trade and technological aspects, such as the presence of organic coatings (Regert, 2011; 177).

Due to the fact that residues are rarely visible to the naked eye, it is necessary to employ chemical analyses to characterize the residues that are absorbed by the porous body of the ceramics and are preserved over time (Evershed, 1993; 2008; Regert, 2011). Among the techniques that can be used for the analysis of contents, one of the most widespread is Gas Chromatography coupled with Mass Spectrometry (GC-MS) (Evershed, 1993, 2008; Garnier, 2007; Pecci, 2009; Romanus et al., 2009; Giorgi et al., 2010; Regert, 2011).

In this paper we present the results of residues analysis performed by GC-MS of cooking and storage ceramic vessels recovered at the archaeological site of Plaça Major- Horts de Can Torras. The site is located in the old town of Castellar del Vallés (Vallés Occidental, Barcelona, Catalonia, Spain) (Figure 1) and was discovered during building works for the reorganization of the central square of the modern village. Archaeological excavations were undertaken between 2003 and 2008 documenting the occupation of the site from prehistory to the present.

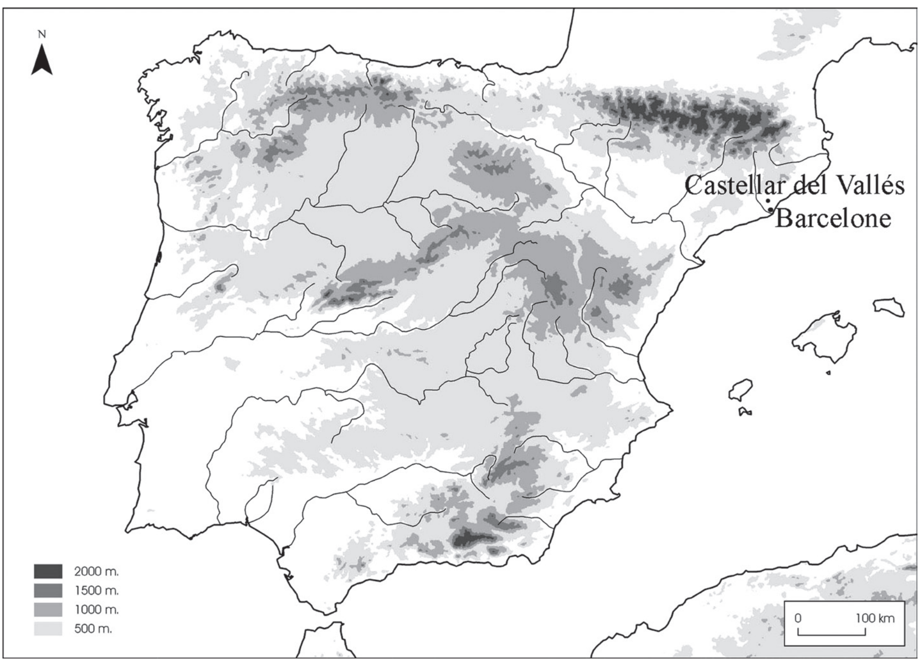

Figure 1. Location of the archaeological site.

The Late Antique site of Plaza Major-Horts de Can Torras is placed between the residential and productive areas of a former Roman Imperial villa, which had been abandoned and never re-used. The chronology of the Late Antique occupation of the site goes from the 5th to the 8th century AD (Roig, 2011). In particular, the Late Antique period can be divided in two phases. The first is dated between the late 5th and the 6th century AD, and the second between the 7th and 8th centuries AD (Figure 2). During the first phase the settlement was characterized by the presence of huts built using perishable material and it was organized in three main areas, mainly devoted to domestic, productive and storing, and funeral uses (Roig and Coll, 2010a, 2010b, 2011; Roig, 2011). During the second phase, all the previous structures were abandoned and the settlement was moved towards north-west, starting a new phase of occupation which is characterized by the presence of a conspicuous number of silos over which, probably, structures used for living

Figure 2. Plan of the Late Antique phases of the archaeological site.

and production were located (Roig and Coll, 2010a; 2010b; 2011; Roig, 2011).

The ceramic vessels selected for the present study belong to this latest phase. The ceramic forms that have been analysed are the most representative of the ceramic assemblage of the 7^{th} and 8^{th} centuries at the site. Imports gradually disappeared from the ceramic assemblages during the 6^{th} century AD. During the analyzed centuries, with the disappearance of imported materials, there is also a simplification of ceramic assemblages and cooking pottery locally produced is almost exclusive. The typological repertoire is very limited, mainly represented by cooking pots and, to a lesser extent, from vessels for liquids such as jugs and small jugs. There are also storage containers of large dimension, such as *dolia* (Roig and Coll, 2011).

Analyses were carried out on twelve cooking and storage vessels to investigate food habits at this rural site during Late Antiquity and to better understand the use of the different ceramic forms. The results were integrated with the data obtained from archaeozoological studies undertaken at the site.

Materials and methods

For this study twelve vessels were sampled and analysed (Figure 3). In particular, 5 pots (samples HCT002, HCT006, HCT008, HCT012, HCT013), 1 pan (sample HCT003), 3 jars (samples HCT004, HCT005, HCT010-11), 1 small jug (*tupí*) (sample HCT001) and 2 *dolia* (samples HCT007, HCT009) were selected (Table 1). Samples HCT010 and HCT011 were taken respectively from the upper part of the wall, near the edge, and from the lower part of the wall, near the bottom of the same jar. The objective of the double sampling was to verify if the same residues were present in the two samples.

All the ceramics analysed are unglazed. Some of them were wheel-thrown and others tournette made.

The sampled ceramics come from three different silos (number 13, 16 and 19) of the second Late Antique phase of the site, which is dated between the middle of the 7^{th} and the middle of the 8^{th} century AD (Roig and Coll, 2010a, 2010b, 2011; Roig, 2011).

Samples were taken from the lower part of the wall of the vessels, after smooth mechanical surface cleaning.

Each sample was pulverized and subjected to three extractions:

a. The total lipid extraction: 1 g of sample was extracted three times with 3 ml of $CHCl_3$/MeOH (2:1) and sonicated at 70 °C for 40 minutes. The supernatant was evaporated using a gentle stream of nitrogen (Mottram et al., 1999).
b. The hydrolysis of the total lipid extract: 3 ml of KOH in MeOH (1 M) were added to half of the total lipid extract and left overnight. 3 ml of ultrapure water were added and solution was acidified with HCl. Finally 3 ml of $CHCl_3$ were added and mixed with a vortex and the subnatant evaporated (Pecci, Cau and Garnier, 2013).
c. The extraction for the identification of wine markers: 0.5 g of sample was extracted with 3 ml of KOH in H_2O (1M) in a sonicated bath at 70 °C for 90 min. After acidification 3 ml of ethyl acetate were added to the extract and mixed with a vortex. After centrifugation the supernatant was evaporated with a gentle stream of nitrogen (Pecci et al., 2013b).

All the extracts were derivatized by adding 25 µl of N, (trimethylsilyl) trifluoroacetamide (BSTFA, SigmaeAldrich) and heated to 70 °C for 1 hour. After cooling, 75 µL of hexane were added. 1 µL of the obtained extract was injected for analysis by GC-MS.

Samples HCT001-HCT007 and HCT011-HCT014 were analyzed using a Shimadzu QP2010 chromatograph equipped with a 30 M X 25 mm X

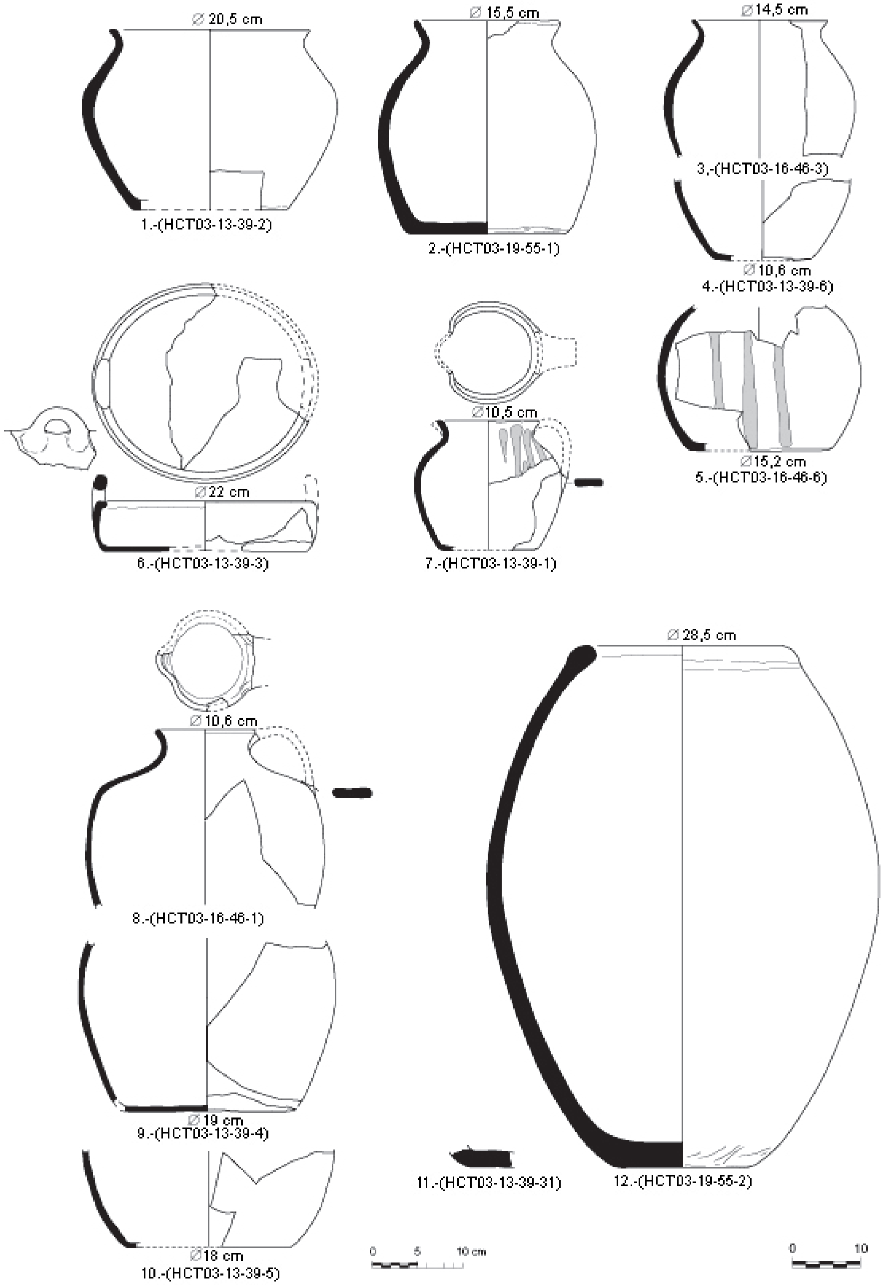

Figure 3. Analyzed ceramics: 1-5 pots (HCT002, HCT008, HCT012, HCT006, HCT013); 6 pan (HCT003); 7 small jug (HCT001); 8-10 jars (HCT010-011, HCT004, HCT005); 11-12 *dolia* (HCT007, HCT009).

Table 1: Analyzed samples.

Sample N.	Sample	Archaeological ID	Silos	Type	Part sampled	Burnt traces
HCT001	142	HCT -39-1	13	Small jug (*tupí*)	Wall	Absent
HCT002	143	HCT -39-2	13	Pot	Wall	Present
HCT003	144	HCT -39-3	13	Pan	Wall	Present
HCT004	145	HCT -39-4	13	Jar	Wall	Absent
HCT005	146	HCT-39-5	13	Jar	Wall	Present
HCT006	147	HTC-39-6	13	Pot	Bottom	Absent
HCT007	149	HTC-39-31	13	Dolium	Bottom	Absent
HCT008	150	HCT-55-1	19	Pot	Wall	Absent
HCT009	151	HCT-55-2	19	Dolium	Wall	Absent
HCT010	153	HTC-46-1A	16	Jar	Wall	Present
HCT011	154	HTC-46-1B	16	Jar	Wall	Present
HCT012	155	HTC-46-3	16	Pot	Wall	Present
HCT013	156	HTC-46-6	16	Pot	Wall	Present

0.25 μm 5% phenylmethyl silica capillary column, coupled with a Thermo Scientific ITQ900 mass spectrometer operated in the electron ionisation mode (70 eV). Samples HCT008-HCT010 were analyzed using a Thermo Scientific TraceGC ultra chromatograph equipped with a 30 M X 25 mm x 0.25 μm 5% phenylmethyl silica capillary column and coupled with a Thermo Scientific ITQ900 mass spectrometer operated in the electron ionisation mode (70 eV). The mass range was scanned in the range of 40-650 m/z. The GC oven temperature was held at 50 °C for 1 min, then increased 5 °C/min up to 300 °C and held isothermally for 10 min.

Peak assignment was made by comparison with the NIST library and the published literature.

Results and Discussion

Cooking vessels: pots and pan

Among the pots analysed, samples HCT002, HCT006, HCT012 and HCT013 are characterized by a high concentration of residues. In the total lipid extractions and their hydrolyses of samples HCT002, HCT006 and HCT012, the significant presence of cholesterol and stearic acid are related to animal origin of the fatty acids identified.

In the chromatograms of both extracts of pots HCT002 and HCT006, fatty acids C_{15} and C_{17} in their branched forms are also present. They are considered markers of ruminant animal fats (Mottram et al., 1999) indicating that at least part of the products cooked in these vessels comes from ruminants.

The identification of long chain alcohols (from C_{22} to C_{34}) and hydrocarbons suggests the presence of waxes (Evershed, 2008).

In all the extracts of the sample HCT012 also dehydroabietic acid, 7-oxodehydroabietic acid, didehydroabietic acid and 15-hydroxy-dehydroabietic acid, are present, which are considered *Pinaceae* products markers (Figure 4) (Mills and White, 1977; Colombini et al., 2005).

Also in all the extracts of the sample HCT013 (a pot that shows abundant burnt traces) compounds related to the presence of *Pinaceae* products are very abundant. In particular, the identification of dehydroabietc acid, 7-oxodehydroabietic acid, didehydroabietic acid, methyldehydroabietate, retene, suggests that the identified product is pine pitch extracted directly from wood (Colombini et al., 2005). In this pot the residues from *Pinaceae* products are so abundant that it is possible to suggest that it was used to contain this substance. There are also traces of stearic (C_{18}:0) and palmitic (C_{16}:0) acids, but these are small compared to compounds related with *Pinaceae* products.

In the total lipid extract and its hydrolysis In the total lipid extract and its hydrolysis of sample HCT008 (coming from a pot), fatty acids (that are mainly of animal origin) are very scarce, suggesting that this pot has not been used long for cooking, as confirmed by the absence of

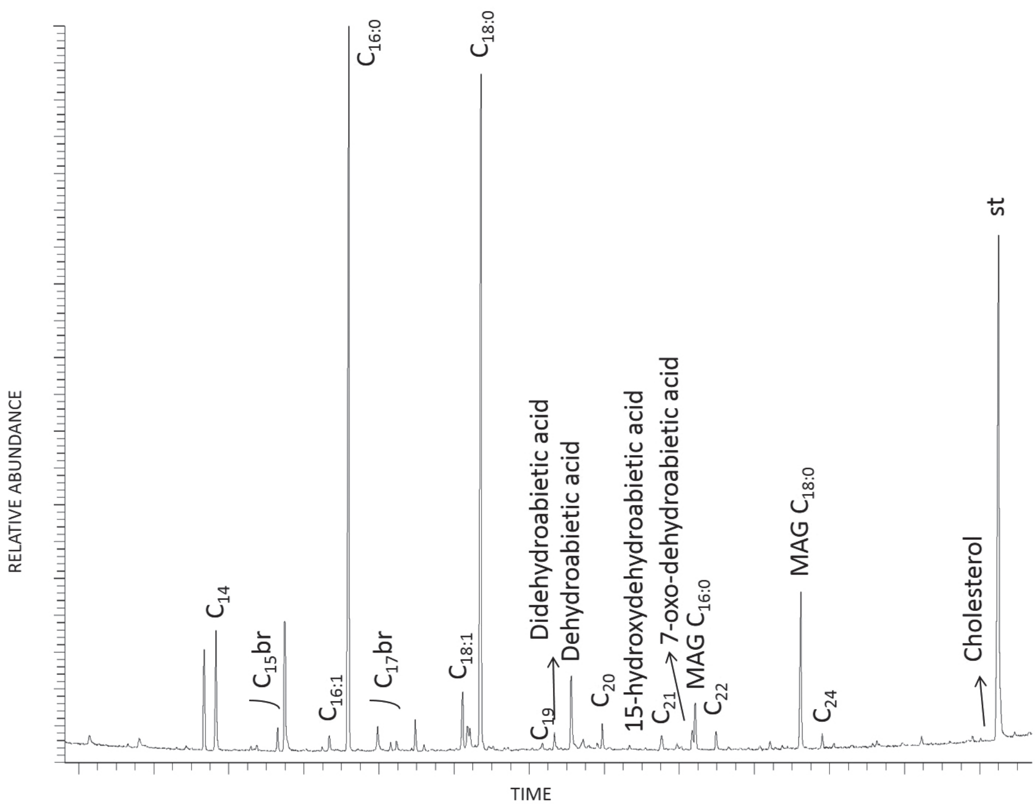

Figure 4. Chromatogram of the total lipid extract of pot HCT012.

burnt traces on the exterior part.

As for the pan (sample HCT003), the GC-MS analyses allow the identification of animal fats mainly from ruminants (abundant stearic and palmitic acids, and C_{15} and C_{17} in their branched forms) (Figure 5). Although it is only through GC-C-IRMS analyses that it is possible to identify the origin of these animal fats; the presence of relatively abundant C_{14}, along with short-chain acids, such as C_6, C_7, C_8, C_9, C_{10} and C_{12}, suggests that dairy products or caprids fats were cooked in the pot (Mc Govern et al., 1999; Kimpe et al., 2002).

Jars and little jar

Jars are usually associated to the serving of wine or water. The analysis of sample HCT004 (a jar with no burnt traces) confirms this hypothesis as in the extraction for the identification of wine markers there are traces of tartaric acid, that is considered the marker of wine (McGovern, 2003; Guash et al., 2004; Garnier, 2007; Pecci et al., 2013b). The presence of dehydroabietic acid in the sample suggests that the jar was coated with *Pinaceae* products. This coating has been identified in jugs coming from Medieval sites in Italy and was probably used to waterproof the vessels (Pecci, 2006, 2009).

As for the other jars, no traces of wine were identified. In all the extracts of sample HCT001 (a small jug or *tupi*) there is a high amount of fats of animal origin (Figure 6). The identification of C_{15} and C_{17} in their branched forms indicates that fats contained in the jar derived from

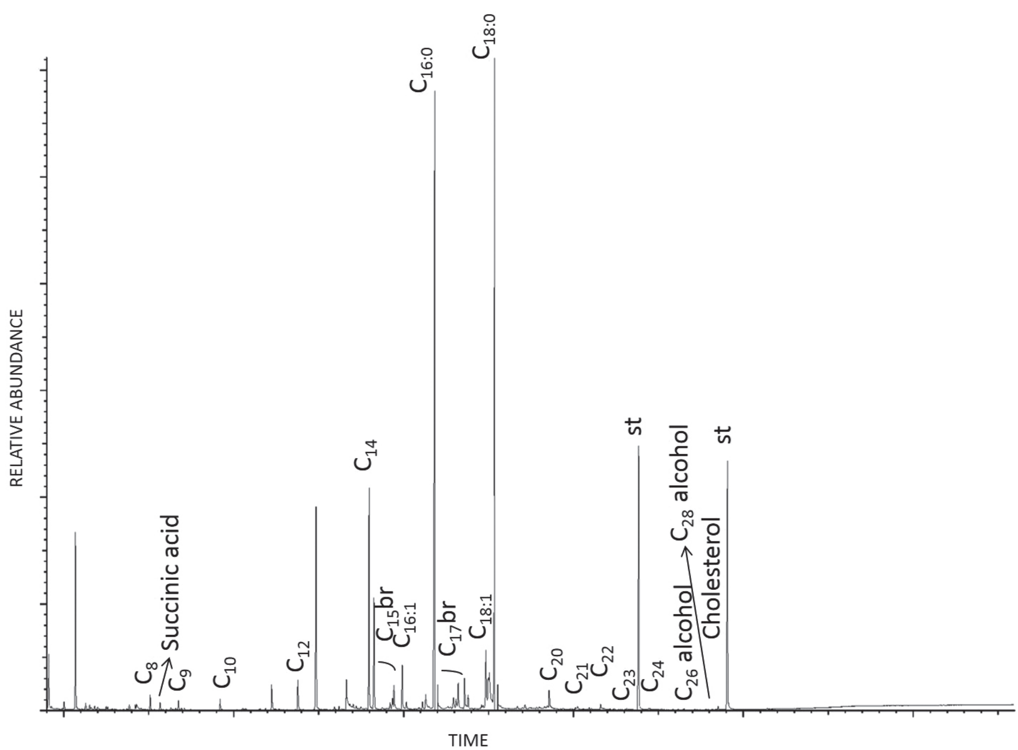

Figure 5. Chromatogram of the hydrolysis of the total lipid extract of the pan HCT003.

ruminant animals (Mottram et al., 1999). This suggests that this small jar was used not to serve water or wine, but to cook/heat broths or soups based on animal fats.

Also in the total lipid extract of sample HCT005 there are traces of ruminant animal fats (Mottram et al., 1999). However, residues are present in smaller amount than those of the previous sample.

Samples HCT010 and HCT011 were taken from the same jar. The two samples were taken respectively from the upper part of the wall, near the rim, and from the lower part of the wall, near the bottom. The objective of the double sampling was to verify if it was possible to appreciate a different enrichment of the ceramic that could give indication on the cooking method used (Charters et al., 1993). The results of the analyses of the three extracts of each sample indicate the presence of abundant dehydroabietic acid, abietic acid, 7-oxodehydroabietic acid and didehydroabietic acid. Furthermore, there are aromatized diterpenes, such as retene, and highly oxidized tricyclic diterpenes (Figure 7). The presence of these substances suggests that *Pinaceae* pitch was present in the samples (Heron and Pollard, 1988; Eerkens, 2002; Egenberg et al., 2003; Colombini et al., 2005). These residues are so abundant that it is possible to suggest that either the jar was coated with a thick layer of pitch (as it often happens e.g. for Roman *dolia*) or the jar contained pitch. No valuable differences in quantity or type of residues can be detected between the two samples taken from the same jar.

Dolia

Two *dolia* (samples HCT007 and HCT009) were analyzed. In the extraction for the identification of wine markers of sample HCT007, succinic, fumaric, glutaric and tartaric acids were identified (Figure 8). All these compounds are considered markers of the presence of wine (Garnier et al., 2003; McGovern, 2003; Guash et al., 2004; Pecci et al., 2013b). In the extraction for the identification of wine markers of the sample HCT009, tartaric acid was also identified. These data suggest that the two *dolia* were used to store wine. The presence of dehydroabietic acid in sample HCT007, and 7-oxo-dehydroabietic acid in sample HCT009 suggests the existence of *Pinaceae* products in the samples (Mills and White, 1977; Colombini et al., 2005). The identification of these compounds allows us to hypothesize that the *dolia* were waterproofed with *Pinaceae* products. This practice was common in the Roman and Late Roman periods and has been demonstrated by residue analysis (Allevato et al., 2012).

Discussion

The site of Plaça Major-Horts de Can Torras is part of the Late Antique network of settlements which gradually replace the Late Roman Imperial model of rural settlement. During this period, the exploitation of the land continued, but it is less intensive. The economy of these new settlements is basically self-sufficient and it is aimed primarily at self-consumption and the maintenance of the settlement. The main activities are primarily agricultural and pastoral, although also some craft activities were carried out. The buildings and the areas of production seem to have, in most cases, a communal character.

The residues analyses carried out on Late Antique ceramics from the site of Plaça Major-Horts de Can Torras reflect the consumption of food related to these production activities. Moreover, it allows some considerations on the use of ceramics and on food practices.

The presence of animal fats in the pan (sample HCT003) (Figure 5) confirms the hypothesis of its use for cooking, as suggested by its shape and the presence of burnt traces. In the cooking pots, mainly fats of animal origin have been

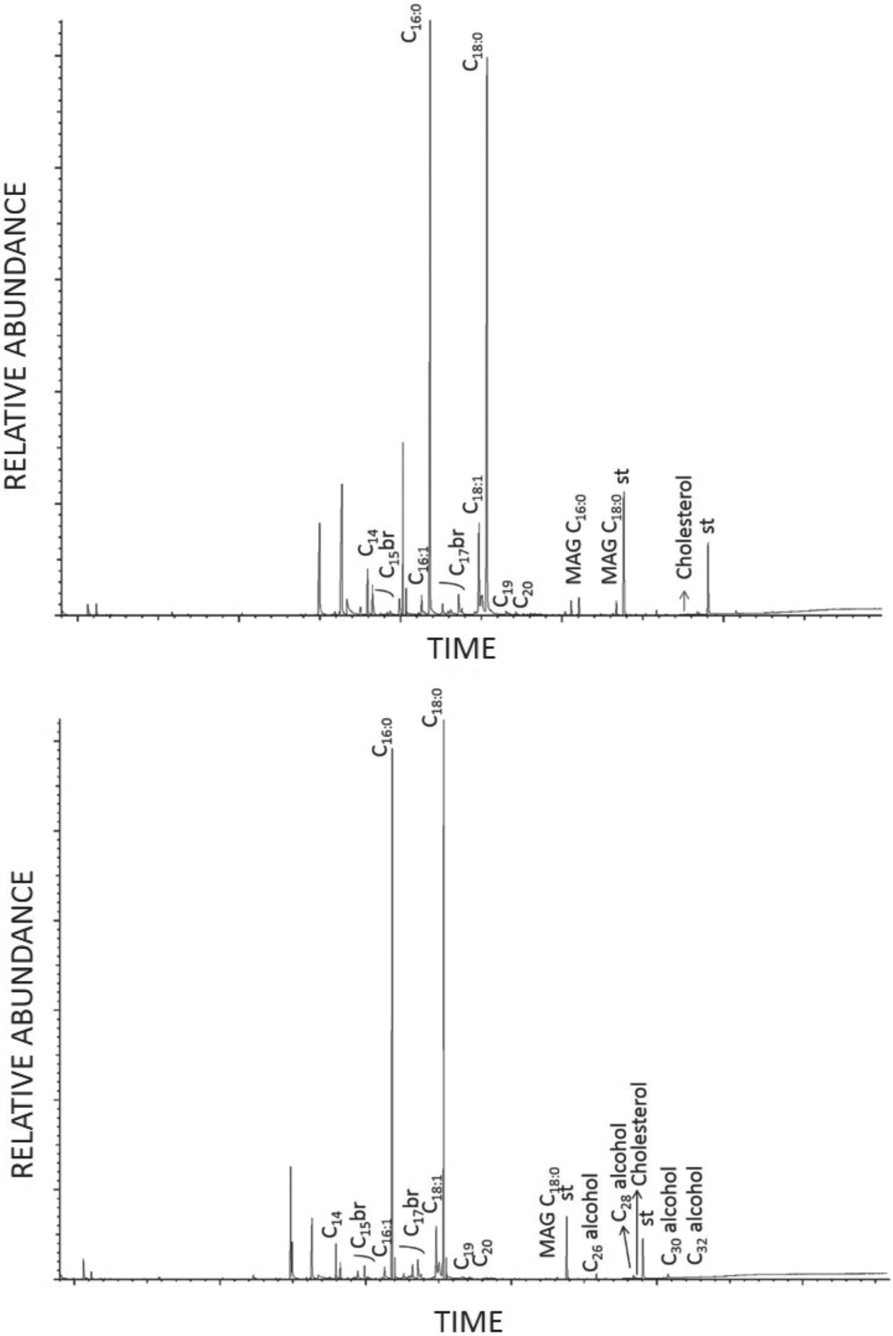

Figure 6. Chromatograms of the total lipid extract and its hydrolysis of the small jug HCT001.

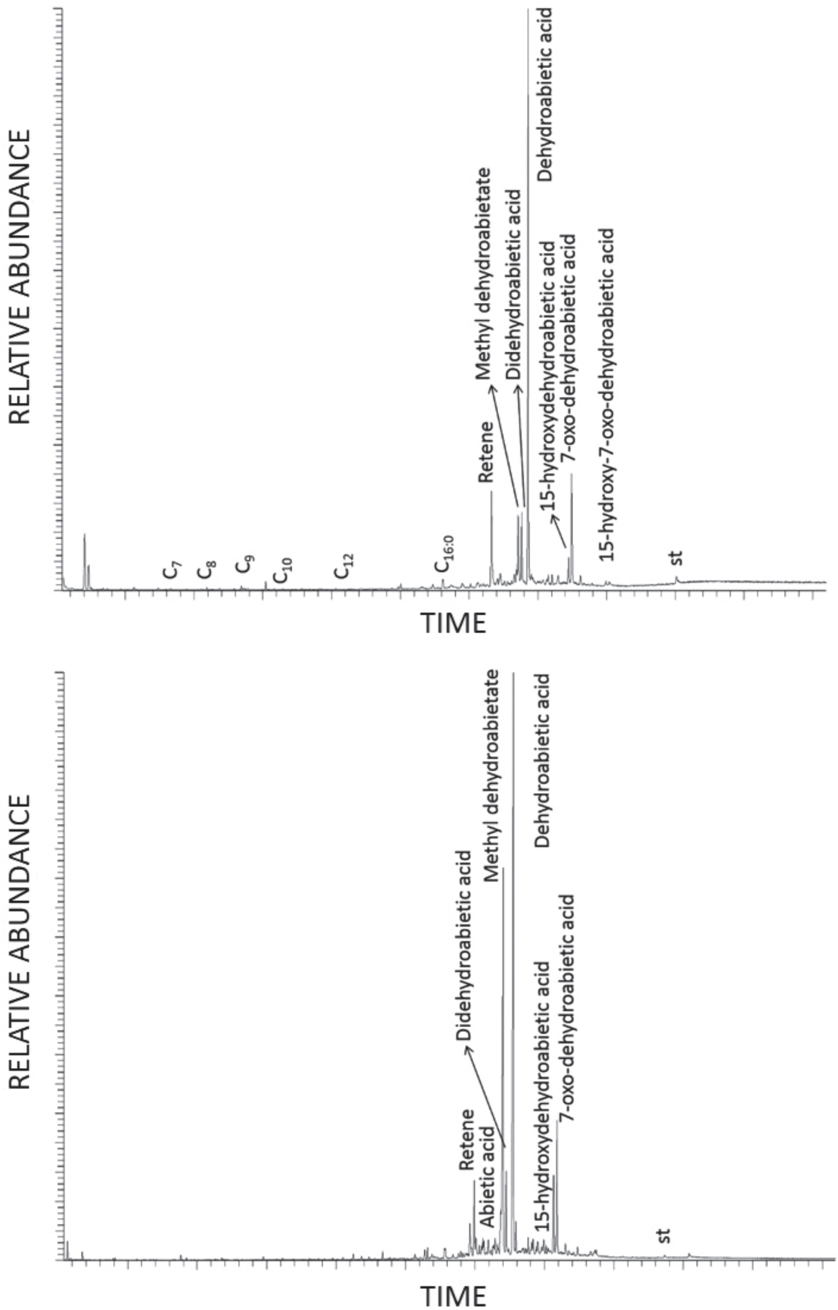

Figure 7. Chromatograms of the total lipid extract of jar HCT010 and of the hydrolysis of the total lipid extract of jar HCT011.

identified. These data, along with the presence of burnt traces, suggest that these have been used for cooking liquid or semi-liquid foods, based on animal fats, mostly from ruminants.

The cooking of ruminant animals, such as goats, sheep and cattle is confirmed by the study of animal remains found at the site (Molina, 2008). Here the predominance of ovicaprids is attested, followed by swine. The presence of animals slaughtered in the most appropriate time for consumption shows that these animals were used for food consumption, while a small number of individuals was probably used for breeding and production of milk and wool (Molina, 2008). The importance of cattle breeding during the Late Antique period appears, also, in written sources. Indeed, legislative (*Liber Iudicum or Lex Visigothorum*) and epigraphic sources (Visigothic slates) describe that rural landscape was characterized by the integration of orchards and fields, forests, pastures and fallow lands used for grazing livestock (García

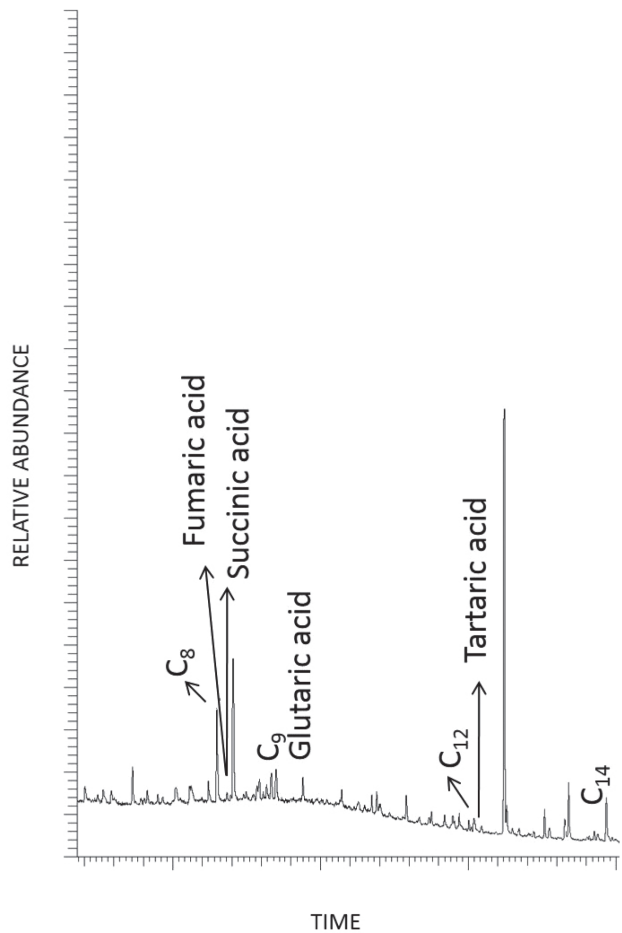

Figure 8. Particular of the chromatogram of the extraction for the identification of wine markers of the dolium HCT007.

Moreno, 1989, 193-204). These laws regulated the breeding of cattle accurately, suggesting that it was the most important economic activity. In legislative sources and slates it is observed that ovicaprids and swine were the most exploited animals for food, while cattle were used in agricultural practices.

It is also worth noting that in sample HCT013 the large amount of residues from *Pinaceae* products identified suggests that the pitch was not used to waterproof the container, but that this was the content of the vessel. It is possible to suggest that the pot was used to store the pitch and heat it before its use. The animal fats identified could be added to the pitch to soften it (Pecci, 2006). However, their presence may also be related to a previous use of the pot for cooking. Similar results -abundant *Pinaceae* products and animal fats- were found analysing two big pots found in Sant'Antimo church in Piombino, Central Italy (Salvini et al., 2008). While at Piombino, the presence of the harbour would explain the use of the pitch for caulking boats, in the case of Plaça Major-Horts de Can Torras it may have a different explanation that should be explored in the future. Samples HCT010 and HCT011 (Figure 7), belonging to a jar, show similar results to the pot HCT013, with abundant *Pinaceae* pitch residues, and might have had the same use. Also this jar shows burnt traces that suggest that the content of the jar was heated.

Regarding the other two jars analysed, the amount of residues is low. The jar HCT005 shows burnt traces. Here, the presence of a low amount of animal residues suggests that the jar -apart from being used for heating water or other products that are not identifiable through the GC-MS analyses performed- was sporadically used for heating or cooking liquid or semi-liquid products with animal fats.

It is also worth noting that the small jug (*tupí*) (sample HCT001) (Figure 6) might have been used to heat and serve liquids prepared with meat or fat of ruminants combined with vegetable fats.

As for the other jars, the jar HCT004, which does not have burnt traces, was used for wine, as mentioned above. Here the *Pinaceae* resin identified could have served as coating of the vessel and may have been added also to flavour the wine. Wine consumption in the site is attested also by the results of the samples taken from the two *dolia* where markers of this beverage have been identified (Figure 8).

The GC-MS analysis provided also information on the use of the different ceramic forms sampled. It is interesting to note that for heating, cooking and/or containing different foods not only pots, but also jars have been used. Jars are usually considered suitable for pouring or serving drinks. However, in this case, the presence of markers of animal and vegetable fats suggests that the so-called jars were used also for food preparation, together with pots. This evidence has already been identified in other parts of the Mediterranean (Buonincontri et al., 2007; Salvini et al., 2008; Pecci, 2009).

As for the dimensions of the ceramic vessels, it is possible to observe that the larger forms were used for storage, while the smaller ones, that are more manageable, served for cooking or heating the different foods consumed. The containers were sometimes waterproofed mainly with *Pinaceae* products, a practise that was observed for cooking pots in other contexts, such as Late Antique Sa Mesquida (Pecci and Cau, 2014) and Medieval Tuscany (Pecci, 2006). Also *dolia* and jars were waterproofed with resin or pitch.

For the storage and possibly heating of pitch, cooking wares were used. The identification of animal fats in the ceramics in which also *Pinaceae* products were identified, may be related to a previous content of the ceramic for cooking or heating, although the possibility that animal fats were added to these products to soften them cannot be discarded (Pecci, 2006).

Conclusions

The GC-MS analysis performed on the ceramics of the Late Antique period from Plaça Major-Horts de Can Torras has provided information about contents and use of the different ceramics analyzed. Animal fats were abundant in the cooking vessels and their consumption is confirmed by the study of animal remains found at the site (Molina, 2008) and by written sources. Also wine was part of the diet of the inhabitants of the settlement in the 7th and 8th centuries AD.

In conclusion, organic residues analyses carried out in the present study have allowed recovering information on the food habits and use of different ceramic forms during Late Antiquity in Catalonia. This, integrated with archaeozoological study seems a good approach to corroborate or refute written sources. In the future, the application of these analyses to a more extensive number of sites will allow a better understanding of the changes that have occurred in the transition between the Roman and the Medieval period, which are also reflected in food habits.

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