variation in the crystallinity index and Raman spectroscopy. The aim was to observe variations in these that resulted in deterioration. The effectiveness of cleaning has always been based on checking the suitability of these parameters.

The shirt of the Prince García at the Monastery of San Salvador de Oña arrived at the CCRBC in order to be adapted to a new preservation environment. Although no direct work was done on the piece, since it was recently restored, a study was carried out on the dyes used. Thin layer chromatography (TLC) identified the red dye kermes. Scanning electron microscopy techniques with X-ray analysis (SEM/EDAX) showed the fixer to be aluminium salt. A study comparing a dyed area of the shirt without any chromatic alteration with another discoloured area, using amplified SEM observation of the fibres and bonds, showed photosensitivity of the silk fabric caused by the dye in the part that had suffered colour loss.

The decorative strips of the funeral clothing of the Monastery of Gradefes displayed mechanical deterioration that is an intrinsic feature of the tannin-based colourant, which uses an iron salt as fixative. Acidity, which is accentuated in relatively unfavourable humidity conditions, caused the fibres to break.

The richness of the decoration of the fragment of the shroud of St. Pedro of Osma can be clearly seen in the materials used, thread from Cyprus and gold-silver alloy with a high gold content (53.91%), much higher than the gilded silver thread, as can be seen in the X-ray fluorescence elemental analysis.

Lastly, we should emphasize the crucial role of applied research in defining treatments and firmly establishing preservation conditions when undertaking projects involving material as sensitive as fabric at centres and institutions researching or holding such heritage.

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IDENTIFICATION OF FATS AND BEESWAX IN CERAMIC VESSELS OF TOMB 121 OF CASTELLÓN ALTO (GALERA, GRANADA)

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Introduction

Gas Chromatography-Mass Spectrometry (GC-MS) has been used systematically for the identification of contents of archaeological vessels since the early 90s, regardless of whether the contents had been absorbed by the porous structure of the clay matrix of the vessel (Evershed et al. 1990, 1992), or remained as solid deposits (Colombini et al. 2005, Ribechini et al. 2008). This technique meant a breakthrough in archaeology, because it allows bringing together archaeological evidence, like the shape of vessels, the setting of the finding or the written sources.

The analyses thus obtained have succeeded to identify accurately chemical markers in archeological vessels: fatty acids, sterols, acylglycerols (Evershed 2008, Colombini and Modugno 2009), wax esters (Heron et al. 1994, Regert et al. 2001) or terpenoids (Urem-Kotsou et al. 2004) revealed the occurrence of substances like vegetable oils, animal fats, milk, beeswax or coating resins (Regert 2011). In addition to the capability to identify a wide range of organic matter, GC-MS stands out for its ease of use and high sensitiveness. The latter is a major advantage, because research on contents in archaeological vessels is usually constrained by small samples and low concentrations of the compounds under study (Evershed et al. 2002).

Based on the analysis of chemical markers, this paper discusses the results obtained by GC-MS from the four ceramic vessels retrieved from tomb 121 of the Argaric settlement of Castellón Alto (Galera, Granada). The excellent state of conservation of the tomb allowed to gain access to a range of data on the burial ritual of the Argaric period. Further knowledge could be gained from the chemical data of the contents of the vessels found in the tomb as a part of the grave good.

Castellón Alto and Tomb 121

Castellón lies in the administrative term of Galera (Granada), approximately 1 km away from the town. The settlement dates back to the Argaric Culture, specifically to the mid-late Bronze period (1900-1600 b. C.) (Figure 1).

The settlement lies between two major units which are linked: one is the hill, with three natural terraces, and the neighbour eastern hillside. The bedrock of the natural terraces was artificially dug and a part of the area was flattened into several terraced plots so most of the hill could be used. The dwellings were built on the artificial terraces. A back wall was built along the rock and a front wall ran parallel to the former thus forming a quadrangle. The rooms occasionally adapted to the ground and are therefore shaped like a polygon (Rodríguez Ariza et al. 2000, Rodríguez Ariza and Guillén 2007).

Figure 1. A view of Castellón Alto in Galera (Source: Rodríguez Ariza & Guillén 2007).

Most of the rooms in the settlement house one or two tombs, so over one hundred have been recorded overall. Except for the child burials in urns, the tombs are small caves excavated in the bedrock. Most of the caves were sealed by large stone slabs or, less frequently, by wooden planks (Rodríguez Ariza 2001, Rodríguez Ariza et al. 2000, Molina et al. 2003, Rodríguez Ariza and Guillén 2007).

Tomb 121 is a small cave dug in the side of an oval-shaped terrace. It was sealed with three planks of Corsican pine (*Pinus nigra*) plastered with mud and rubble masonry. The cave was therefore safe from the outside soil and a microclimate developed which interrupted the decomposition of the organic matter, thus allowing its partial preservation (Molina et al. 2003, Rodríguez Ariza and Guillén 2007).

Inside the cave lay the remains of a man in left side prone position. The man was 1.60 m and was between 27 and 29 at the time of his death. Mummified soft tissue remains on the head and on the postcranial skeleton. It is remarkable how the hair of his head, beard and body hair in general has been preserved where skin is still present. Linen fabric and a sort of small net made with esparto rope was found around his right leg, and remains of what may be cotton can also be found (Molina et al. 2003, Rodríguez Ariza and Guillén 2007) (Figure 2).

The right front part of the tomb also housed remains of a child. Some of the bones were still joined. The body had been taken out from the original tomb and buried with the adult. Soft tissue could also be found on this body, as well as dark hair, presumably a head covering woven with wool, covered with leather and with remains of linen tissue.

Figure 2. A view of Tomb 121 (Source: Rodríguez Ariza & Guillén, 2007).

The funerary grave good consisted in four ceramic vessels, one of which was a cup, and also copper bracelets, silver rings, a copper dagger with remains of the scabbard leather, and a copper axe with the whole holm oax handle. Remains of the threads that held the axe to the handle were still present. The child wore a bronze bracelet on each arm and three beads as well as lamb bones (Molina et al. 2003, Rodríguez Ariza and Guillén 2007).

Experimental

Archaeological samples

Approximately 4 g of the bottom or of the base matter of the four vessels were sampled. The sampling area was a requirement to cause the least possible damage to vessels which were in outstanding condition.

The fragments of archaeological vessels selected for analysis were wrapped each in a piece of dark paper and stored in a freezer at least at -20 °C until their analysis. The fragments were then taken out of the freezer and a sample was collected, usually from the base. Any remains of soil were removed with an electric hand drill. The sample was then grinded to the appropriate size in an agate mortar. Two grams were taken for the analysis.

Extraction and derivatization

Extraction is in accordance with the procedure described in Evershed et al. (1990).Ten µL of tetratriacontane (internal standard) and 10 mL of the mixture chloroform/ methanol ($CHCl_3$:MeOH) (2:1 v/v) were added to 2 g of the ground ceramic fragment. Lipids were extracted with ultrasound for 15 minutes. The solution was centrifuged (3500 rpm, 5 minutes) eliminating the remaining ceramics and removing the supernatant where the lipids are solved. This process was repeated twice. The extract portions were combined into one for solvent evaporation under N_2 stream.

The dry extract was solved again in 500 µL of $CHCl_3$ and a 100 µL aliquot was removed and transferred to a smaller vial. This volume was evaporated to dryness under an N_2 stream. N,O-bis-(trimethylsilyl) trifluoroacetamide (BSTFA) with 1 % trimethylchlorosilane (TMCS) was used as derivatization agent. The derivatization reaction took place with 20 µL of this reagent at 70 °C for 30-40 minutes. When the reaction was over, the vial was cooled and the remaining derivatizing agent was evaporated under an N_2 stream. The sample was then solved again in 50 µL of cyclohexane. An amount of 1 µL of sample was injected into the chromatograph.

Gas Chromatography-Mass Spectrometry

The analyses were performed using a quadrupole type HP 5989B MS coupled to a HP 5890A serie II plus GC. Samples were introduced by on column injection into a 15 m x 0.32 mm I.D. fused silica capillary column, coated with poly(dimethylsiloxane) stationary phase with 0.1 µm film thickness. Helium was used as the carrier gas (purity 99.99%) at a flow speed of 1.0 ml/min. The GC oven temperature program was as follows: initially at 50 °C, held for 2 min; ramp to 350 °C at 10°C/min, held for 10 min.

The operating conditions were an emission current of 400 µA, an electron energy of 70 eV and an ionization source temperature with the following ramp: 0-15 minutes at 250 °C, 15-23 minutes at 300°C and 23-42 minutes at

350ºC (Parras 2008). Total ions measurements were obtained in the mass spectrometer that was scanned from m/z 50-900 at a scan rate of 2.083 s/scan. The GC-MS capillary interface was mantained at a temperature of 350 ºC.

Results

Relevant results were obtained from 2 out of the 4 samples analysed (Figure 3, Table 1). Vegetal fat, beeswax and milk fat were identified in this case.

Figure 3. A reconstruction of the vessels. Left: CA-29237, glass. Right: CA-29235 bottle.

Vessel CA-29235 (bottle). The chromatogram obtained shows two groups of compounds: one comprehends the fatty acids palmitic ($C_{16:0}$), stearic ($C_{18:0}$), oleic ($C_{18:1}$) and triacylglycerols (TAG) of oleic acid and of palmitic acid (trioleine, OOO, being the most abundant triglyceride). The amount of triacylglycerols of oleic acid is high and confirms the vegetal origin of the fat (Figure 4).

The other group of compounds belongs to the typical set of substances found in beeswax. Contemporary beeswax shows a recognizable pattern based on the occurrence of:

-Series of C_{23} to C_{33} carbon number n-alkanes displaying a unimodal distribution possessing a strong odd-over-even predominance.
-Series of C_{40}-C_{54} carbon number palmitic acid wax esters with a main constituent that contains 46 carbon atoms.
-Free fatty acids, of which lignoceric acid ($C_{24:0}$) is predominant.

In general, beeswax degradation causes a good part of the fatty acids (except for palmitic acid) to disappear, *n*-alkanes to be lost, and long chain alcohols with an even carbon number (usually from C_{24} to C_{34}, maximizing at C_{30} that are released of wax esters) to appear (Tulloch 1970, Chartes et al. 1995, Regert et al. 2001).

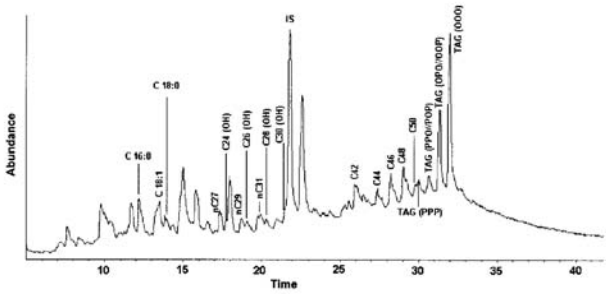

Figure 4. Sample *CA-29235.* GC-MS chromatogram of the lipid extract.

Some of these indicators, as well as others related to beeswax degradation, have been found to occur: *n*-alkanes C_{27} to C_{31} (C_{27} to a larger proportion), palmitic acid wax esters (C_{42} to C_{50}) and long chain alcohols (C_{24} to C_{30} with C_{30} as major component) resulting from hydrolysis of wax esters. Overall, these indicators confirm the occurrence of beeswax in the vessel.

The low proportion of long-chain alkanes is worthy of note. There is no evidence that the vessel was on the fire. The degradation of alkanes would thus be related either to the very dry environment of the burial site or to evaporation due to heating the beeswax before application to the inner side of the vessel (Regert et al. 2001, 2003).

Vessel CA-29237 (glass). The analysis shows peaks of fatty acids capric ($C_{10:0}$), lauric ($C_{12:0}$), myristic ($C_{14:0}$), palmitic, stearic and oleic. The former three suggest a case for milk of ruminants (Dudd and Evershed 1998) (Figure 5). Their occurrence in the milk of monogastric ruminants (humans, pigs…) is extremely low. These fatty acids are also rare in animal adipose tissues (Gunstone et al. 1994, Mirabaud et al. 2007).

A small peak occurs which has been identified as heptadecanoic acid ($C_{17:0}$), except that its proportion is comparatively smaller. Again, this fatty acid occurs in milk fat in a low proportion alongside pentadecanoic acid ($C_{15:0}$) (not found in the sample). The latter two acids may have also occurred in milk fat as ramified fatty acids *iso-* and *anteiso-* (Dudd and Evershed 1998).

The occurrence of the following group of triglycerides is also an argument in favour of a case for milk: MMP or MPM; MPP or PMP; PPP; OOP or OPO and OOO (M: myristic acid; P: palmitic acid; O: oleic acid). The occurrence of myristic acid in the triglycerides supports a case for milk fat.

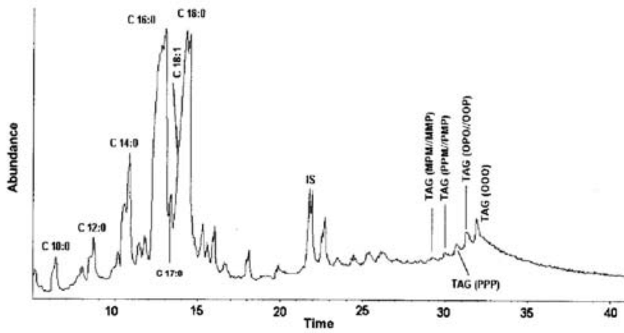

Figure 5. Sample *CA-29237.* GC-MS chromatogram of the lipid extract.

Table 1. Mass spectrometric data of the main lipidic compounds. P*: Protoned Palmitic Acid by the fission of alkyl-oxygen bond.

COMPOUNDS	MAIN PEAKS (m/z)
Fatty Acids (TMS)	
$C_{10:0}$	244 (m^+)-229(m^+-15)-145-129
$C_{12:0}$	272 (m^+)-257(m^+-15)-145-129-117
$C_{14:0}$	300 (m^+)-285(m^+-15)-145-129-117
$C_{16:0}$	328 (m^+)-313(m^+-15)-145-129-117
$C_{18:1}$	354 (m^+)-339(m^+-15)-145-129-117
$C_{18:0}$	356 (m^+)-341(m^+-15)-145-129-117
Wax Esters	
C_{42}	621$(m+1)^+$-257(P^*)-239 (P^+)
C_{44}	649$(m+1)^+$-257(P^*)-239 (P^+)
C_{46}	677$(m+1)^+$-257(P^*)
C_{48}	705$(m+1)^+$-257(P^*)
C_{50}	Detected for the retention time and the main peaks 257(P^*)-239 (P^+)
Triacylglycerols	
MMP/MPM	523($GlyMP^+$)-495($GlyMM^+$)-239(P^+)-211(M^+)-129-97
PPM/PMP	551($GlyPP^+$)-523($GlyMP^+$)-239(P^+)-211(M^+)-129-97
PPP	551($GlyPP^+$)-239(P^+)-129-97
PPO/POP	578($GlyPO^+$)-551($GlyPP^+$)-339(Gly O^+)-313(Gly P^+)-265(O^+)- 239(P^+)-129-97
OOP/OPO	604($GlyOO^+$)-578($GlyPO^+$)-339(Gly O^+)-313(Gly P^+)-265(O^+)-239(P^+)-129-98
OOO	604($GlyOO^+$)-339(Gly O^+)-265(O^+)-129-97
PPS/PSP	579($GlyPS^+$)-551($GlyPP^+$)-341($GlyS^+$)-313 ($GlyP^+$)-267(S^+)-239(P^+)-129-97
SSP/SPS	608($GlySS^+$)-579($GlyPS^+$)-341($GlyS^+$)-313($GlyP^+$)-267(S^+)-239(P^+)-129-97
SSS	608($GlySS^+$)-341($GlyS^+$)-267(S^+)-129-97

Conclusions

The results obtained from the analysis of chemical markers in the ceramic vessels of tomb 121 of Castellón Alto provide additional information on the rituals and economic activity. This information is in addition to the rest of the contextual, palynological and carpological data.

First, the highly likely occurrence of milk in vessel CA-29237 allows for putting forward some hypotheses and proposals. In general, it is assumed that the use of milk and dairy products in Europe became widespread in the 5th millennium B.C. However, the latest research by Evershed et al. (2008) based on chemical analysis proved that the milking of ruminant animals was clearly practised in the sixth and seventh millennia BC in southeastern Europe and northwestern Anatolia respectively. The chemical data of tomb support other archaeological evidence of the use of milk and dairy products in the Iberian peninsula from the 5th millennium B.C.

The presence of the infant body in the tomb is comparatively more important and also raises the question whether it is related with the presence of milk in the vessel in question. Additionally, the tomb also housed bones of a milk-producing ruminant (sheep). Use of this and other vessels before being part of the funerary dowry cannot be discarded.

Second, the occurrence of vegetal fat in the bottle (sample CA-29235) cannot be put down to a specific type of oil. Even if the state of conservation of this tomb is excellent, fatty acids go through alterations that modify their original proportion in food and therefore make it difficult to identify the type of fat accurately. In these circumstances, the palynological and carpological data available in the settlement provides further information. According to this, only wild olive trees and flax can have yielded vegetal oil. Of these, the former is rare whereas the latter is abundant (Rodríguez Ariza et al. 1996, Contreras et al. 2000).

Finally, the third element occurring in sample CA-29235 is beeswax. It can be explained in different ways according to its meaning and its timeline.

Table 2. A timeline of the use of beeswax in Eastern Andalusia-Upper Guadalquivir based on chemical markers.

Settlement	Chronology	Context
Marroquíes Bajos. Ciudad de la Justicia. Jaén	Late 3rd millennium b.C Chalcolithic	Domestic-workshop. Hut 693 (Parras 2008)
Marroquíes Bajos. Plot E.2.1, U.A.23. Jaén	Late 3rd millennium b.C Chalcolithic	Domestic (Sánchez and Cañabate 1999a)
Castellón Alto. Galera. Granada	1900-1600 b. C. Argaric Bronze	Funerary. Tomb 121
Remojadero Pescado and Huérfanos street. Jaén	IX-VIII b. C. Late Bronze	Structure built in the bedrock (Sánchez and Cañabate 1999a)
Sanctuary El Pajarillo.Huelma. Jaén	IV b. C. Iberian Culture	Storage area sanctuary (Sánchez and Cañabate 1999b)
Oppidum Puente Tablas. Jaén	IV b. C. Iberian Culture	Domestic. House 6 (Sánchez et al., 2009)

The occurrence of beeswax inside vessels since prehistory may be explained in terms of a medical use, a cosmetic, a ritual substance, protection against corrosion, paint, vessel construction, glue, coating, the occurrence of honey, or the lost wax technique (Regert et al. 2001). As the chronological and contextual frame of the materials under study allows to rule out some of the above, the most conservative approach may consider three hypotheses: as the contents of the vessel, as an inside coating, or as signalling the occurrence of honey. It cannot be said whether it cooccurred with vegetal oil and signal one and the same use where beeswax was used as internal lining, or whether the vessel may have been used for two different purposes before and after the burial.

The occurrence of beeswax completes and widens the sequence of use of this product in Eastern Andalusia thus forming a sequence where, from the point of view of chemical analysis, begins in the 3rd millennium and ends in the Iberian period (Table 2). In addition to its extensive chronological record above, beeswax occurs in a number of archaeological contexts which comprehend a varied functional spectrum: a domestic area shared with a workshop of bone figurines, only one domestic area, an inhumation tomb, the storage area of a sanctuary and, again, a room for domestic purposes.

Acknowledgements

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Conference report

FIRST MEETING OF THE NETWORK OF SCIENCE AND TECHNOLOGY FOR THE CONSERVATION OF CULTURAL HERITAGE (TECHNOHERITAGE)

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Conservation of Cultural Heritage is a multidisciplinary task involving a broad spectrum of scientific and professional institutions. To this end Spain has created a network of institutions dedicated to research and conservation of Heritage, which also involved private companies. The first meeting of the Network of Science and Technology for the Conservation of Cultural Heritage had as main objective the presentation of the different network groups and provide a forum to share experiences.

The meeting took place on 28 and 29 June in Madrid, hosted in the auditorium of IPCE, with the participation of more than a hundred scientists and professionals. The two-day program included a total of 35 communications, which have been published in an electronic book (Rogerio-Candelera and Saiz-Jimenez 2011)(Figure 1) soon to be hosted on the website Technoheritage (www.technoheritage.es).

Figure 1. Front cover of "Ciencia y Tecnología para la Conservación del Patrimonio Cultural"

The different communications included work in the areas of archaeology, architecture, physics, chemistry, biology or materials science among others, covering different issues related to the conservation of movable or immovable cultural assets, to the conservative approaches and interventions of different conservation institutions and museums, and the role of enterprises in the research/conservation system. The complete list of contributions includes: *Estructura social y territorio*, by I. Sastre et al., *Estudio de materiales y técnicas utilizados en obras de arte*, by A. Justo et al., *Materiales poliméricos y patrimonio cultural*, by M. Lazzari and A. Ledo, *Grupo de Bioingeniería y Materiales (BIO-MAT) de la Universidad Politécnica de Madrid*, by D.A. Moreno and A.M. García, *Grupo de Tecnología Mecánica y Arqueometalurgia*, by A.J. Criado et al., *ICMUV Grupo de Arqueometría*, by C. Roldán and S. Murcia-Mascarós, *Láseres y Nanotecnologías para el Patrimonio Cultural*, by M. Castillejo et al., *Rocas ornamentales: procesos fisicoquímicos*, by A.C. Íñigo et al., *Grupo de investigación en el Patrimonio Arquitectónico y Sostenibilidad (GIPAS-UAH)*, by G. Barluenga et al., *Monitorización y tratamiento de datos microclimáticos en el patrimonio cultural: IC9 Grupo de Investigación y Desarrollo Tecnológico del IVC+R*, by J. Pérez-Miralles et al., *Diagnóstico de impactos ambientales sobre el Patrimonio Cultural (Histórico, Artístico y Natural) mediante análisis in-situ, observaciones micro-espectroscópicas y modelado químico*, by J.M. Madariaga et al., *Conservación de vidrios y*