Embalmed heads of the Celtic Iron Age in South of France

Salma GHEZAL[(1)], Elsa CIESIELSKI [(2)], Benjamin GIRARD[(2)], Aurélien CREUZIEUX[(2)], Peter GOSNELL[(3)], Carole MATHE[(1)], Cathy VIEILLESCAZES[(1)], Réjane ROURE[(2)]

(1) *UMR-IMBE, Université d'Avignon, CNRS 7263/IRD 237/AMU, Ingénierie de la restauration des patrimoines naturel et culturel, Faculté des Sciences, Campus Jean-Henri Fabre, 301 rue Baruch de Spinoza BP 21239, 84916 Avignon cedex 9, France.*

(2) *ASM Archéologie des Sociétés Méditerranéennes, UMR 5140, Université Paul Valéry Montpellier, CNRS, MCC, 34000, Montpellier, France. Labex Archimede « Archéologie et Histoire de la Méditerranée et de l'Egypte anciennes » (ANR-11-LABX-0032-01).*

(3) *UMR-IMBE, Université d'Avignon, ERASMUS MUNDUS MASTER IN ARCHaeological MATerials Science student.*

*Corresponding authors at: rejane.roure@univ-montp3.fr
ROURE Réjane
UMR5140-ASM, site Saint-Charles
Université Paul Valéry Montpellier 3
Route de Mende
F-34199 MONTPELLIER Cedex 5

Abstract

Ancient texts described that one of the most impressive ritual practices of the Celts during the Iron Age was to remove the heads of enemies killed in battle and to display them, once embalmed, in front of their own dwellings. An archaeological settlement excavation site in Le Cailar, in southern France, has revealed a considerable number of examples of this practice, known for many years thanks to literary sources and archaeological data: iconographic representation of heads and human bones. Weapons were also exhibited alongside the severed heads. Here we report the results of chemical investigations for the characterization of embalm biomarkers liable to be present in eleven fragments of these human cranial remains. These results may lead to answers to some of the archaeometric questions related to the subject of embalming in 3rd century BC Transalpine Gaul, mentioned by Greek authors, thus advancing the knowledge of these ritual practices as part of the wider research into the proto-historic societies of the Mediterranean coastal region.

Keywords

Iron Age, Celts, embalming, severed head, chemical analyses

1. Introduction

In the 3rd century BC, the number of wars and battles seems to increase in almost the whole of Western Europe. Indeed, hundreds of weapons have been found in sanctuaries and sacred places since they weren't display there before. In many of that places, human remains have been discovered with metal artifacts, and also fauna remains linked to sacrifice of animals (Buchsenschutz 2017; Brunaux, 2004; Barral et al., 2006). We know thanks to classical literary sources that at the end of battles, the Celts cut off their enemies' heads on the battlefield and carry them back to their settlements, hanging the decapitated heads on their

horse's neck. This very accurate picture of this practice is known through two fragments of ancient texts, written in the 1st century BC respectively by Strabo and by Diodorus of Sicily, both recording the testimony of an ancient Greek, named Poseidonios, who travelled in the south of Gaul around 100 BC (Strabo, IV, 4, 5 *in* Lasserre 1966). Other classical texts mentioned that fact (Polybius, Livy, …) and many archaeological data illustrate this practice too (Ciesielski et al. 2011; Armit 2012; Boulestin Henry Gambier 2012). At Entremont, an Iron Age settlement in Provence, many pieces of sculpture have been discovered during one of the first dig in the South of France, showing decapitated heads, with one particular sculpture representing a warrior on his horse, weapons (a sword and a spear) at his side, and a severed head suspended from the horse's neck (Arcelin 2011), just as testified literary sources. Human bones were also discovered in many settlements belonging to the Second Iron Age (Roquepertuse, Glanum, …) and other sculptures and engraved stone too, in the whole South of France (fig. 1). In some places, archaeologists found human skulls with iron nails inside them and in other places they found pillar or lintel with cavities head shaped (fig. 1).

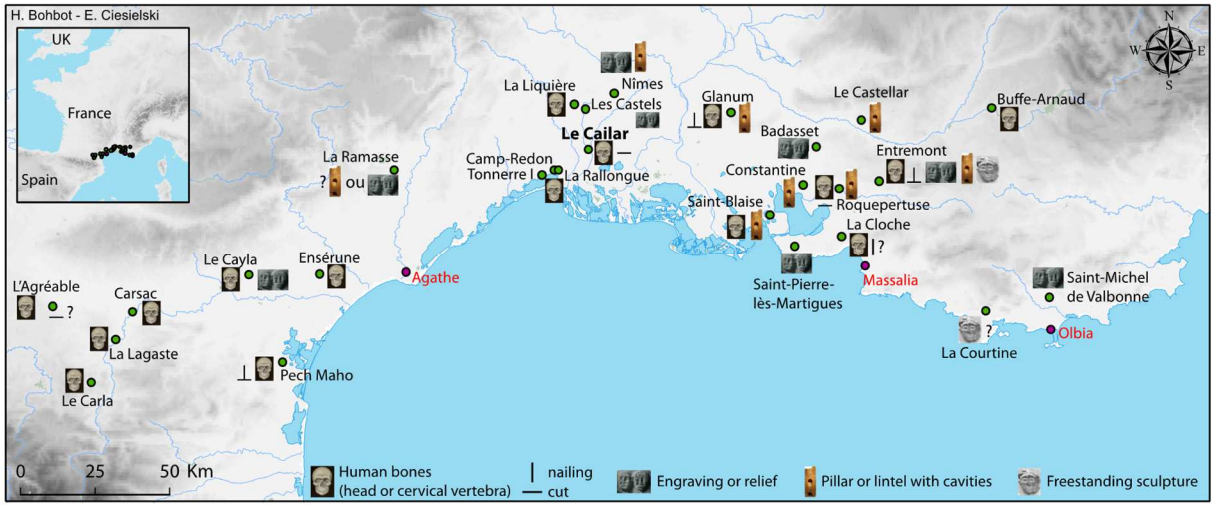


Fig 1. Map of discoveries in the South of France: human bones and sculptures. COLOR

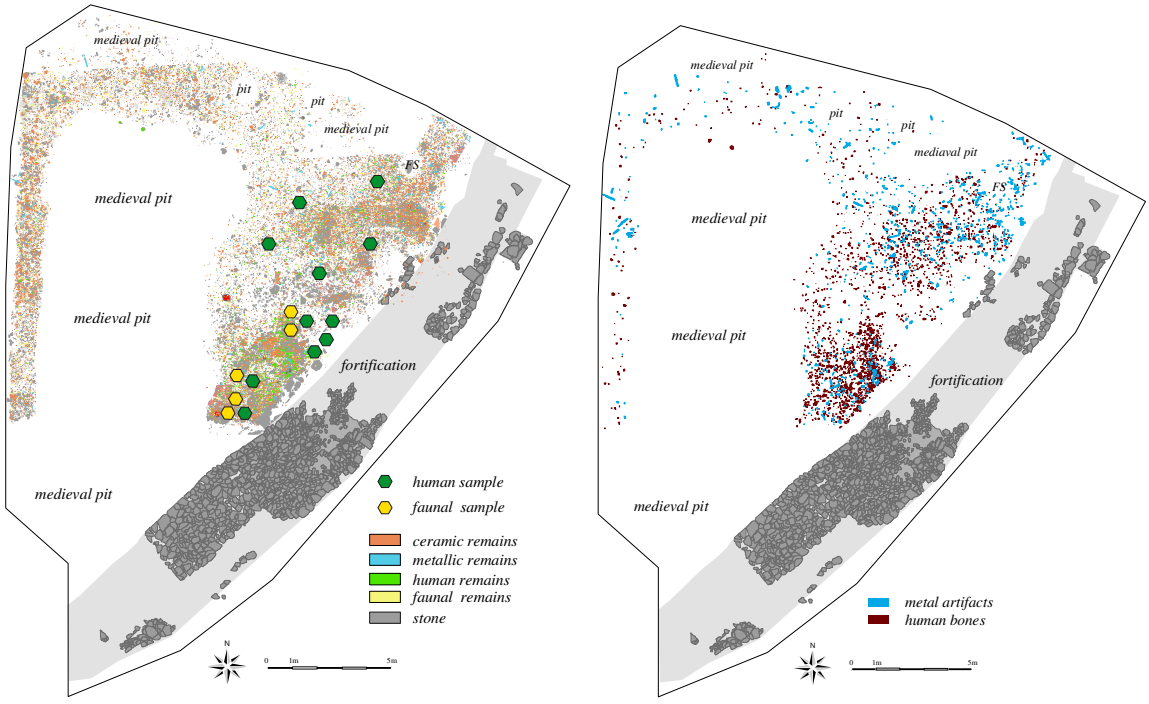
Thus, displaying severed heads was a well-known practice, but it had never been observed in recent digs in the South of France. That's why the discovery in Le Cailar is of great importance and provides us with a significant amount of new data (Roure et al. 2006), and the opportunity to make new analysis, especially chemical ones to find if the heads have been prepared as the Greeks testified it. Indeed, Strabo and Diodorus both wrote that the Celts embalmed the heads, and they indicate both with 'cedar oil'; however, it could had been a local Pinacea oil that Greeks named 'cedar' because the smell was close. That's why this paper's study aimed at verifying the presence of possible embalming remnants in archaeological cranial fragments from Le Cailar. Chemical analyses using GC-MS were so performed in order to characterise organic components liable to be present in eleven from these human cranial fragments

Embalming and other mummification phenomena are well-documented worldwide with much of both the scientific and the archaeo-historic academic literature documenting the best surviving examples of embalming in pharaonic dynasties of Egypt (Łucejko et al., 2012; Ménager et al., 2014; Nicholson and Shaw, 2000), and mummification has also been proved in Bronze Age in Britain (Parker Pearson et al. 2005). Our paper will present another example of that kind of practice.

2. Experimental section

2.1. Sample description

The Iron Age settlement of Le Cailar was situated near a wide lagoon connected to the Rhône River. It was occupied from the 6th century BC until the Roman period in Gaul (1st century AD). The fortified settlement was located on a small hill and was also a harbour for Mediterranean traders (Etruscans and Greeks at that time). Near the fortification, and probably close to one of the gates, a large public area where inhabitants exposed weapons and skulls was discovered, displayed on a large space (fig. 2). The archaeological context is very clear: there is no doubt it is an open area where the heads and weapons were displayed. We have stratigraphical and chronological evidence for each level with display of bones and metal artifacts (Roure et al. 2017). Remnants of metal (mainly weapons), potteries and animal bones, were intermingled with the human skulls. Both ceramic and metallic remains allowed a precise chronology of this embalming event to be obtained and forty Massilian coins were also found in the same place, which again confirms the chronology of the 3rd century BC (that's why there is no radiocarbon dating). Amphora and vessels from the Greek city of Massalia and vases from Italy and Spain all belong to this period and the metal artifacts are all linked to the latenian typology (La Tène B2-C1). The stratigraphy and artifacts showed that several deposits happened: the first deposits corresponded to the end of the 4th century or to the very beginning of the 3rd century BC, and that the trophy heads and weapons were displayed throughout the 3rd century BC until *circa* 200 BC, when the area was covered by



mud.

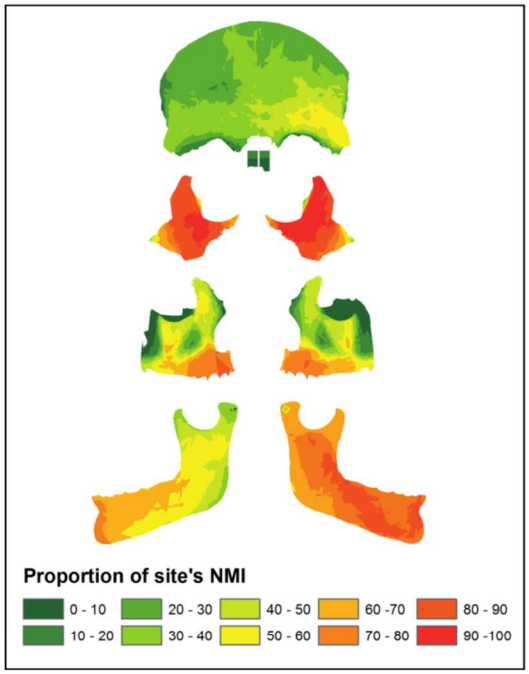
Fig 2.: Maps of Le Cailar excavation with the distribution of each kind of remains and localization of samples (on the left) and only the metal artifacts and the human bones (on the right). (all the deposit mapped together) COLOR

About 2700 fragments of human bones were recorded during ten excavation campaigns: all the fragments are part of the skull, except for six little pieces of cervical vertebrae (fig.3). Many of the skulls presents cut marks, linked not only to the act of decapitation, but also to some act connected to preparing the heads for display: removal of cervical vertebrae and aperture of the postero-inferior portion of the cranium, probably carried out in order to

remove the brain; and tongue ablation, or at least the scraping of the muscles under the mandible (Ciesielski et al. 2014).

Identified Pieces	NR
skull	1078
skull/face	2
face	286
mandible	117
teeth	973
cervical vertebrae	4
skull pieces non identified precisely	216
Total	2676

global MNI = 51



	Number of remains	Weight (g)	median weight (g)	mean weight (g)
skull	1078	12480,3	8,3	16
skull/face	2	100,3	-	-
face	286	2205,3	6,5	9
mandible	117	2997,9	20,1	28,1
cervical vertebrae	4	14,2	2,5	3,6
Total	1487	17798	37,4	56,7

Fig 3. : table of human remains and map of cranial NMI from Le Cailar (2003-2011) COLOR

The discovery of the skulls was immediately related to the above-mentioned ancient texts from Strabo and Diodorus. These texts indicated that the Gauls "embalmed the head of the most famous enemy with cedar oil" (Strabo, IV, 4, 5 *in* Lasserre 1966), and this explains why chemical analysis, as opposed to other types of analyses, was undertaken. Preliminary chemical analyses on human bones were carried out in the search for any traces of biological products which could have been used to prepare these heads for their display, even though no macroscopic or microscopic remains were visible.

Eleven cranial fragments were selected (Table 1, Fig. 4), from each of which two powder samples (100 mg to 150 mg each) were taken, first from the exterior and then from the interior surfaces. A total of twenty-two samples were analyzed. Fragments were chosen from the skulls: frontal, zygomatic, parietal, originating from different deposits. At least, our panel would be representative, even though the way the 11 pieces were chosen from among 2800 was random, because there wasn't any visible residue. All the human remains were precisely recorded with three coordinates (x, y, z) and registered by number (except the pieces discovered at the beginning of the excavation and before the installation of this protocol).

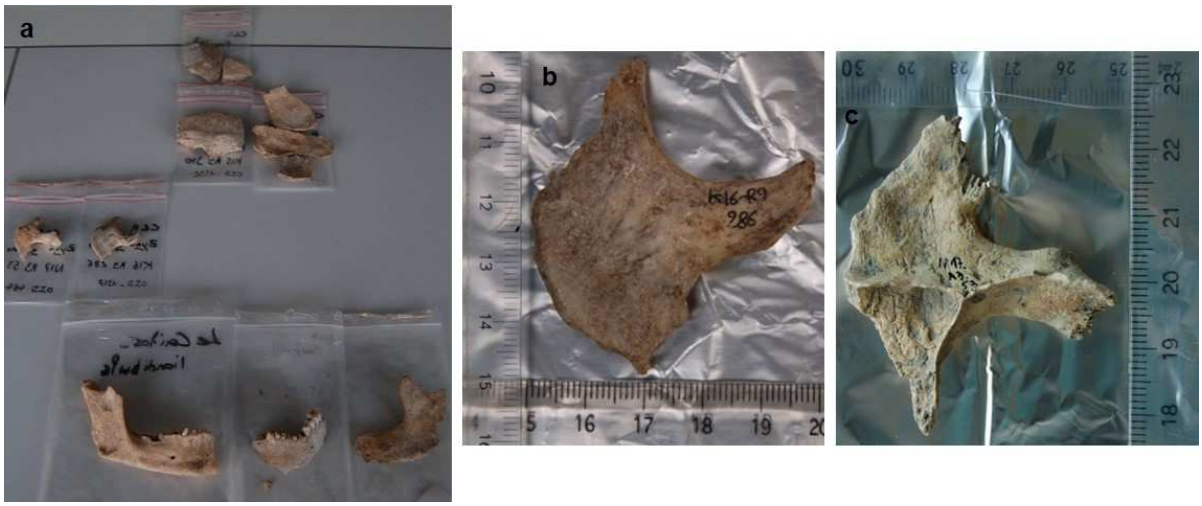


Fig 4. Pictures of a. Total assemblage b. CLR K16 R9 286 exterior surface c. CLR N17 R3 53 interior surface cranial fragments (after Ghezal and Gosnell). COLOR

Table 1. Description of cranial fragments

Alpha-numeric context number	General bone identification
CLR 07	Mandible (central section)
CLR 04 X5	Mandible (right side)
CLR 03 X103	Mandible (left side)
CLR 04 X11	Parietal
CLR 03 X29	Parietal
CLR 03 X44	Parietal
CLR M18 R6 570	Frontal
CLR N17 R0 8	Frontal
CLR K15 R5 340	Frontal
CLR K16 R9 286	Zygomatic (left)
CLR N17 R3 53	Zygomatic (left)

Five remains of fauna (Table 2), discovered strictly in the same place and at the same level as the human bones, were also analysed using identical protocols, in order to verify if the products found could come from taphonomic bias or if they could be link to a specific practice for the human heads.

Table 2. Description of faunal remains

Alpha-numeric context number
K15 R5 361
K15 R5 354
K16 R9 304
M18 R6 535 A
M18 R6 538

2.2. Materials

Dichloromethane was of GC grade and purchased from Merck (Darmstadt, Germany). High purity water (18.3 MΩ.cm) was obtained from a Milli-Q purification system (Millipore). Derivatisation was made using BSTFA (N,O-bis(trimethylsilyl) trifluoroacetamide) with 1 % TMC (trimethylchlorosilane) purchased from Sigma Aldritch.

2.3. GC-MS analyses

Each powdered cranial sample was added to 6 mL of dichloromethane and sonicated (2 min, 70% amplitude) using an ultrasonic probe (Vibra-Cell model 75186). This volume was necessary in order to obtain the immersion of the probe in the extraction solvent. The samples were centrifuged (30 min, 6000 rpm). The supernatant corresponding to the organic extract was split into two, in order to perform GC-MS analyses.

Before the GC-MS analyses, the organic extracts were derivatised by trimethylsilylation. For this purpose, the solutions were evaporated to dryness under nitrogen and mixed with 100 µL of BSTFA with 1 % TMC for 30 min at 70°C. The trimethylsilylated extracts were dried under nitrogen and dissolved in 500 µL of hexane. All the samples were filtered through a 0.45 µm PTFE filter before injection.
GC-MS analyses were carried out applying a Thermo Scientific™ Focus gas chromatographic system mounted with a Thermo Scientific Al 3000 auto-injector and coupled with a ITQ™ 700 Series GC-Ion Trap Mass Spectrometer (Thermo Fisher Scientific Inc.). GC separation was performed on a fused silica capillary column TG-5MS (Thermo Fisher Scientific, alvc), with a stationary phase (5% diphenyl-95% dimethyl-polysiloxane phase).

A volume of 1 µL for each sample was injected in splitless mode and the injector temperature was set at 250°C. Molecular components were eluted using helium at a constant flow of 1.2 mL/min. The following temperature programme was used: initial temperature 50 °C for 2 min, 50 to 220 °C at 8 °C/min, 220 to 260 °C at 2 °C/min and 260 to 330 °C at 10 °C/min.

The mass spectra were recorded in Electron Impact mode with an electron ionization voltage of 70 eV, an ionisation time of 25,000 µs and a mass range of 50 to 650 *m/z*. The ion trap and interface transfer line were respectively at 250 °C and 300 °C.

Thermo Xcalibur™ 2.2 software (Thermo Fisher Scientific Inc.) was used for instrumental control and data acquisitions.

Mass spectra peak assignment was based on a comparison with internal mass spectrum database (from commercial standards and from fresh and artificially-aged resins and oils) and NIST database (NIST MS Search 2.0).

3. Results and discussion

All of the components were identified according to their specific mass data (base and molecular ions), their retention times in comparison with standard molecules and the specialized literature (Table 3).

For an accurate interpretation of GC-MS results, the contribution of lipids from the bone cannot be disregarded. Fresh bones contain significant amounts of cholesterol and a lesser

concentration of fatty acids associated with marrow (Colonese et al., 2015; Evershed et al., 1995). Previous analyses of archaeological bones revealed only the presence of cholesterol, together with its diagenetic degradation products, especially cholest-5-en-3ß-ol-7-one (Collins et al., 2002; Colonese et al., 2015; Evershed et al., 1995; Stott et al., 1997). However, traces of saturated fatty acids (primarily $C_{14:0}$, $C_{16:0}$ and $C_{18:0}$), a lesser concentration of oleic acid ($C_{18:1}$) and a low quantity of linoleic acid ($C_{18:2}$) were recently detected in the analyses of human bones for archeological purposes (Colonese et al., 2015).

The total ion current of the lipid extract from CLR K16 R9 286 sample is presented in Figure 5 and shows that almost all lipid extracts from the analysed bones exhibited the presence of saturated fatty acids $C_{9:0}$, $C_{14:0}$, $C_{16:0}$ and $C_{18:0}$, monoacylglycerols, cholesterol and its degradation products: cholest-5-en-3β,7β-diol and cholest-5-en-3ß-ol-7-one.

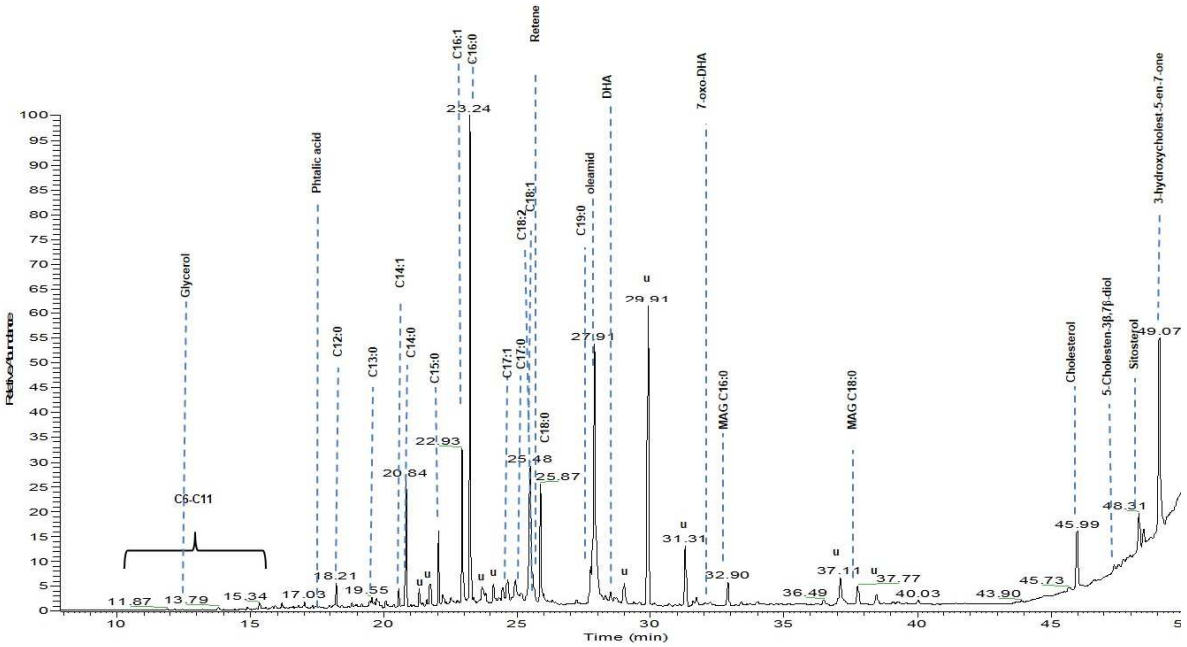


Fig 5. Total ion current gas chromatogram of internal CLR K16 R9 286 sample lipid extract.
. Except for retene, all the compounds were detected in their trimethylsilylated form.
Abbreviations: Cn:x fatty acids with n carbon atoms and x unsaturations. DHA, Dehydroabietic acid; MAG Cn:x, Monoacyl glycerol with n carbon atoms and x unsaturations; u, unknown. COLOR

The fatty acid composition of the different samples is presented in Figure 6, showing a chromatogram of the internal CLR K16 R9 286 sample with extracted signals at *m/z* 117 and the base peak of main saturated fatty acids. The chromatogram shows that almost all lipid extracts from analyzed bones exhibited the presence of saturated fatty acids $C_{9:0}$, $C_{14:0}$, $C_{16:0}$ and $C_{18:0}$. Some unsaturated fatty acids were also detected ($C_{14:1}$, $C_{16:1}$, $C_{17:1}$, $C_{18:1}$).

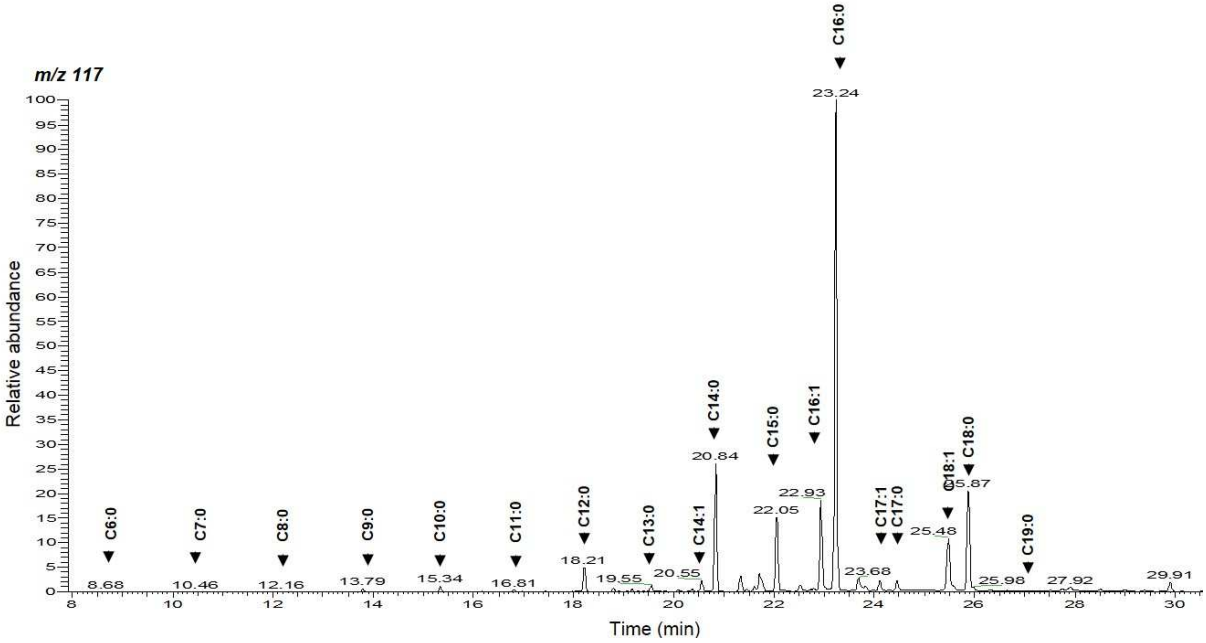


Fig 6. Gas chromatogram of internal CLR K16 R9 286 sample with extracted signals of *m/z* 117. Cn:x fatty acids with n carbon atoms and x unsaturations. All the fatty acids were detected in their trimethylsilylated form.

The high amount of saturated fatty acids, monoacylglycerol (MAG), glycerol, cholesterol and its observed degradation products, are characteristic of degraded animal fats (Mottram et al., 1999). The high ratio of palmitic acid compared with that of stearic acid, in addition to the presence of ß-sitosterol are indicative of possible plant-origin fats. However, the contribution from endogenous bone fats cannot be discounted.

Unfortunately, it is not possible to assign a precise plant origin on the basis of fatty acids alone using conventional chromatographic methods, because of the lipid thermal degradation leading to changes in saturated fatty acid proportions (Nawar, 1969).

Six of the eleven human samples (CLR 03 X44; CLR M18 R6 570; CLR N17 R0 8; CLR K15 R5 340; CLR K16 R9 286; CLR N17 R3 53) contained diterpenoic compounds, degradation products of abietic acid and biomarkers of conifer resin. The gas chromatogram of the internal K15 R5 340 sample is presented in Figure 7 with extracted signals at *m/z* 239, *m/z* 253, *m/z* 219 and *m/z* 241 base peaks respectively of dehydroabietic acid, 7-oxo-dehydroabietic acid, retene, palustric acid (26.24 min) and isopimaric acid (26.66 min).

The dehydrogenation of abietic acid leads to dehydroabietic acid. This compound was the most abundant diterpenoid detected in our samples, followed by its oxidation product, 7-oxo-dehydroabietic acid. Retene is the final product of the thermal degradation of abietane skeleton diterpenoids. The detection of such aromatic compounds in these samples is characteristic of intense heating of the resin from the tree belonging to the Pinaceae family (Marchand-Geneste and Carpy, 2003).

The traces of linear *n*-alkanes (C_{23}-C_{33}) and *n*-alkanols (C_{12}-C_{26}) which were detected are more likely caused by soil contamination. In fact, these compounds were already detected in significant amounts in the soil (Poirier et al., 2005).

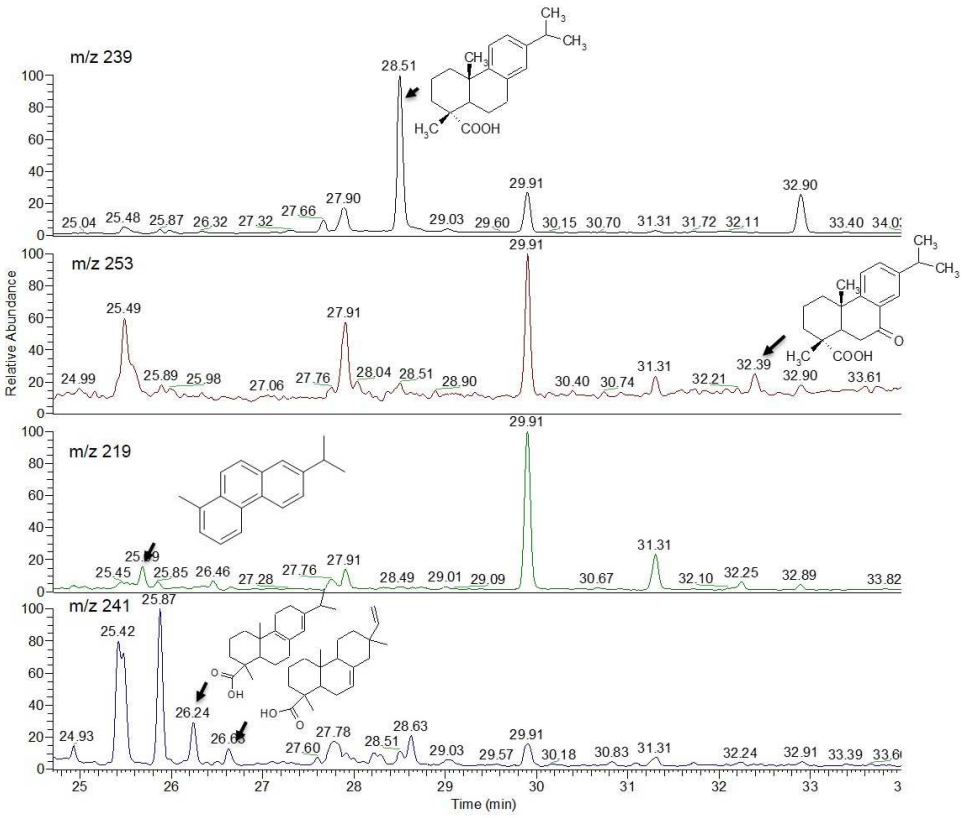


Fig 7 Gas chromatogram of internal CLR K15 R5 340 sample with extracted signals of *m/z* 239, 253, 219 and 241. Except for retene, all the terpenoid compounds were detected in their trimethylsilylated form.

Interestingly, in the lipid extracts from faunal samples only cholesterol was preserved (data not shown). Fatty acids initially present in the bones seemed degraded and terpenoid compounds were not detected. These results suggest that lipids observed in the human skull extracts originate not only from human bones, but also from vegetal or animal fats and allowed to eliminate the hypothesis of external contaminations for all the detected substances excepted linear *n*-alkanes and *n*-alkanols.

Table 3. Lipidic composition of organic extracts

	CLR 07		CLR 04 X5		CLR 03 X103		CLR 04 X11		CLR 03 X29		CLR 03 X 44		CLR M18 R6 570		CLR N17 R0 8		CLR K15 R5 340		CLR K16 R9 286		CLR N17 R3 53	
	int	ext	int	ext	int	ext	int	ext	int	ext	int	ext	int	ext	int	ext	int	ext	int	ext	int	ext
Monocarboxylic acids																						
$C_{8:0}$ and less	-	-	-	-	-	-	-	-	-	-	-	-	+	+	-	-	+	+	+	+	-	-
$C_{9:0}$	+	+	+	+	+	+	+	+	+	+	+	+	+	+	+	+	+	+	+	+	+	+
$C_{10:0}$	-	-	-	-	-	-	-	-	-	-	-	-	+	+	-	-	+	+	+	+	-	-
$C_{11:0}$	-	-	-	-	-	-	-	-	-	-	-	-	-	-	-	-	-	-	+	+	-	-
$C_{12:0}$	-	-	-	-	-	-	-	-	+	+	+	+	+	+	+	+	+	+	+	+	-	-
$C_{13:0}$	-	-	-	-	-	-	-	-	-	-	-	-	+	+	-	-	+	-	+	+	-	-
$C_{14:1\Delta 9}$	-	-	-	-	-	-	-	-	-	-	-	-	-	-	-	-	-	-	+	-	-	-
$C_{14:0}$	+	+	+	+	+	+	+	+	+	+	+	+	+	+	+	+	+	+	+	+	+	+
$C_{15:0}$	-	-	-	-	-	-	-	-	-	-	-	-	+	+	-	-	+	+	+	+	-	-
Cis $C_{16:1\ \Delta 9}$	-	-	-	-	-	-	-	-	-	-	-	-	+	+	-	-	+	+	+	+	-	-
$C_{16:0}$	+	+	+	+	+	+	+	+	+	+	+	+	+	+	-	-	+	+	+	+	+	+
$C_{17:1}$	-	-	-	-	-	-	-	-	-	-	-	-	-	-	-	-	+	-	+	-	-	-
$C_{17:0}$	-	-	-	-	-	-	-	-	-	-	-	-	-	-	-	-	+	-	+	+	-	-
Cis,*Cis* $C_{18:2\ \Delta 9,12}$	-	-	-	-	-	-	-	-	-	-	-	-	-	-	-	-	+	-	+	+	-	-
Cis $C_{18:1\ \Delta 9}$	-	-	-	-	-	-	-	-	-	-	-	-	-	-	-	-	-	-	+	+	-	-
$C_{18:0}$	+	+	+	+	+	+	+	+	+	+	+	+	+	+	+	+	+	+	+	+	+	+
$C_{19:0}$	-	-	-	-	-	-	-	-	-	-	-	-	+	+	-	-	+	+	+	+	-	-
Monoacylglycerol																						
MAG $C_{14:0}$	-	-	-	-	-	-	-	-	-	-	-	-	+	+	-	-	+	+	+	+	-	-
MAG $C_{16:0}$	+	+	+	+	+	+	+	+	+	+	+	+	+	+	+	+	+	+	+	+	+	+
MAG $C_{18:0}$	+	+	+	+	+	+	-	-	+	+	-	+	+	+	+	+	+	+	+	+	+	+
Steroidal compounds																						
Cholesterol	+	+	+	+	+	+	+	+	+	+	+	+	+	+	+	+	+	+	+	+	+	+
ß-Sitosterol	+	+	+	+	+	+	+	+	+	+	+	+	+	+	+	+	+	+	+	+	+	+
Cholest-5-en-3β,7β-diol	-	-	-	-	+	-	+	+	-	-	-	-	+	+	+	+	+	+	+	+	-	-
Cholest-5-en-3ß-ol-7-	+	+	+	+	+	+	-	-	-	-	-	-	+	+	-	-	+	+	+	+	+	+

one																						
Glycerol	-	-	+	-	+	+	-	-	+	-	+	+	-	-	+	+	-	-	-	-	+	+
n-Alkanes																						
C_{23}	-	-	-	-	-	-	-	-	-	-	+	-	-	-	-	+	-	-	-	-	-	-
C_{24}	-	-	-	-	-	-	-	-	-	-	+	-	-	-	-	-	+	+	+	+	-	-
C_{25}	-	-	+	-	-	-	+	+	-	-	+	-	-	-	-	+	+	+	+	+	-	-
C_{26}	-	-	+	-	-	-	+	+	-	-	+	-	-	-	-	+	+	+	+	+	-	-
C_{27}	+	+	+	-	-	-	+	+	-	-	+	-	-	-	-	+	+	+	+	+	-	-
C_{28}	+	+	+	-	-	-	+	+	-	-	+	-	-	-	-	+	+	+	+	+	-	-
C_{29}	+	+	+	-	-	-	+	+	-	-	+	-	+	+	-	+	+	+	+	+	-	-
C_{30}	-	-	+	-	-	-	+	+	-	-	+	-	+	+	-	+	+	+	+	+	-	-
C_{31}	+	+	+	-	-	-	+	+	-	-	+	-	+	+	-	-	+	+	+	+	-	-
C_{32}	+	+	-	-	-	-	+	+	-	-	+	-	+	+	-	+	+	+	+	+	-	-
C_{33}	+	+	-	-	-	-	-	-	-	-	+	-	+	+	-	-	-	-	-	-	-	-
n-alkan-1-ol																						
C12 OH	-	-	-	-	+	+	-	-	-	-	-	-	+	+	+	+	+	+	+	+	-	-
C14 OH	-	-	-	-	+	+	-	-	-	-	-	-	+	+	-	-	+	+	+	+	-	-
C15 OH	+	+	-	-	-	-	-	-	-	-	-	-	+	+	+	+	+	+	+	+	+	+
C16 OH	-	-	-	-	+	+	-	-	-	+	-	-	+	+	+	+	+	+	+	+	+	+
C17 OH	-	-	-	-	+	+	+	+	-	-	-	-	-	-	+	+	-	-	+	+	+	+
C18 OH	+	+	+	+	+	+	+	+	-	+	-	-	+	+	+	+	+	+	+	+	+	+
C19 OH	-	-	-	-	+	+	-	-	-	-	-	-	+	+	-	-	+	+	+	+	+	+
C20 OH	+	+	-	-	+	+	-	-	-	+	-	-	+	+	+	+	+	+	+	+	+	+
C21 OH	+	+	-	-	+	+	-	-	-	-	-	-	+	+	-	-	+	+	+	+	+	+
C22 OH	+	+	-	-	+	+	+	+	-	-	-	-	+	+	+	+	+	+	+	+	+	+
C23 OH	+	+	-	-	+	+	-	-	-	-	-	-	+	+	+	+	+	+	+	+	+	+
C24 OH	+	+	-	-	+	+	+	+	-	-	-	-	+	+	+	+	+	+	+	+	+	+
C25 OH	+	+	-	-	-	-	-	-	-	-	-	-	+	+	-	-	-	-	-	-	-	-
C26 OH	+	+	-	-	+	+	+	+	-	-	-	-	+	+	+	+	+	+	+	+	-	-
Diterpenoids																						
Palustric acid	-	-	-	-	-	-	-	-	-	-	-	-	+	+	-	-	+	+	+	+	-	-
Isopimaric acid	-	-	-	-	-	-	-	-	-	-	-	-	+	+	-	-	+	+	+	+	-	-
DHA	-	-	-	-	-	-	-	-	+	-	+	+	+	+	+	+	+	+	+	+	-	-
7-Oxo-DHA	-	-	-	-	-	-	-	-	-	-	+	-	+	-	-	-	+	tr	tr	tr	-	-
Retene	-	-	-	-	-	-	-	-	-	-	+	-	-	+	-	-	+	-	-	-	-	-

Abbreviations: Cn:x, Monocarboxylic acid with n carbon atoms and x unsaturations; MAG Cn:x, Monoacylglycerols with n carbon atoms and x unsaturations; Cn, alkane with n carbon atoms, CnOH, alkan-1-ol with n carbon atoms; DHA, Dehydroabietic acid; +, presence; -, absence; tr, traces.

Conclusion

Chemical analyses using GC-MS were performed in order to characterise organic components liable to be present in eleven human cranial fragments discovered at the Le Cailar archaeological site in the south of France.

Thanks to this study, we demonstrated that some of the severed heads exhibiting by the Celts were embalmed. We knew thanks to literary sources and to archaeology that the Celts removed the heads of their enemies slain on the battlefield and that they exhibited them in public spaces, maybe as an expression of the bravery and the strength of the community and of its warriors (Boulestin Henry Gambier 2012, Ciesielski et al. 2011).

We speak of 'mumification' because Ancient Greek texts clearly assert that Celts used to embalmed the head with cedar-oil – or a local pinacea oil that Greeks named 'cedar' – and wanted to keep those heads for a long time. Moreover, Strabo and Diodorus wrote: "They never gave back the head belonging to the most famous and brave people, even for an equal weight of gold" (Strabo, IV, 4, 5 *in* Lasserre 1966). That sentence means it was possible to recognized the severed head.

In fact, analyses highlighted the presence of saturated and unsaturated fatty acids, monoacylglycerols, sterols, alkanes, alkanols and biomarkers of conifer resins. Resins were usually heated and mixed with plant oil, which may explain the presence of retene and the high amount of fatty acids in these samples, notably palmitic and stearic acids. The use of a mixture of resin and oil is documented in antiquity, in many societies and at different periods, for their antibacterial, anti-oxidative and aromatic properties (Langenheim, 2003). Concerning the Celts, the practical results of that kind of treatment – antiseptic and avoiding smells – are maybe the first and main reason why they have done it, but this is linked to the will to preserved the head. None of the fauna remains analyzed contained that kind of biological things, so it is not something coming from the soil of this area but really a specific and voluntary practice of the Celtic people in order to embalmed the head.

The precise process of embalmment is quite difficult to know for that period: maybe the head was dived in cedar-oil or the local pinacea oil; maybe the heads were just covered with the pinacea mixture with some tool which totally disappeared with time. As noted above, biological study of the bones remains has showed many cut marks, linked to preparing the heads – probably tongue ablation and removing of the brain (Ciesielski et al. 2014). Anyway, enough oil had to be used to penetrate inside the bone, but the process could be long: oil could had penetrated slowly into the bone, while time was running, and sun and rain affecting the heads exposed outside. It is also possible that Pinaceae oil was used several times, all along the display, in order to preserve the head. In both case, that could explain why it is the parietal/frontal where the Pinaceae is found, because they were the most visible and exposed pieces.

As an important point arising from this study, it would be of great interest to determine when this specific practice actually began, in the early 3rd century BC or before, at the end of the 4th century BC. Further analyses should be carried out in order to answer this question. We will also have to question if the skulls came only from enemies or from ancestors at the same time (Ciesielski 2017), as it often happened in head hunting societies (Boulestin Henry Gambier 2012). Finally, we have to determine if the process was used for all the head or only some of them.

Acknowledgment

The authors thank all the research teams who worked on Le Cailar site (especially Michel Py and Henri Duday), as well as the institutions that contributed to the progress of the archaeological excavations and studies. This work was supported by the Labex Archimede « Archéologie et Histoire de la Méditerranée et de l'Egypte anciennes » [ANR-11-LABX-0032-01].

Conflict of interest: The authors declare that they have no conflict of interest.

References

Arcelin, P., 2011. Entremont, in Roure, R., Pernet L., (dir), *Des Rites et des Hommes. Les pratiques symboliques des Celtes, des Ibères et des Grecs en Provence, en Languedoc et en Catalogne*, Catalogue de l'exposition, Musée Archéologique Lattara, Lattes, Errance, (Archéologie de Montpellier Agglomération, 2), 63-71

Armit, I., 2012. *Headhunting and the Body in Iron Age Europe*. Cambridge University Press

Barral P., Daubigney A., Dunning C., Kaenel G., Roulière-Lambert M.-J. (éds.), 2006. *L'âge du Fer dans l'arc jurassien et ses marges. Dépôts, lieux sacrés et territorialité à l'âge du Fer. Actes du XXIX^e^ colloque international de l'AFEAF, Bienne, 5-8 mai 2005*, Besançon, Presses Universitaires de Franche-Comté, 2006 (Annales littéraires ; Série "Environnement, sociétés et archéologie »)

Boulestin B., Henry Gambier D., 2012. Crânes trophées, crânes d'ancêtres et autres pratiues autour de la tête : problèmes d'interprétation en archéologie. Actes de la table-ronde pluridisciplinaire, musée national de Préhistoire, Les Eyzies-de-Tayac (Dordogne, France), 14-16 octobre 2010, BAR International Series 2415

Brunaux J.-L., 2004. *Guerre et religion en Gaule, essai d'anthropologie celtique*, Errance

Buchsenschutz O. dir., 2017. *L'Europe celtique à l'âge du Fer, VIIe – Ier siècles*, Nouvelle Clio, L'histoire et ses problèmes

Ciesielski, E, 2017. La pratique celtique des « têtes coupées » en France méditerranéenne : l'exemple du site du Cailar (Gard) au IIIe s. av. n.-è. Approche archéothanatologique et traitements informatiques des données », PhD, Montpellier, 20 décembre 2017

Ciesielski, E., Bohbot, H., 2014. Analyses of bone modifications on human remains: a GIS approach. In Giligny F., Djindjan F., Costa L., Moscati P., Robert S. (eds.), Proceedings of the 42^nd^ annual conference on Computer Applications ans Quantitative Methods into Archaeology, CAA – 21^st^ Century Archaeology, Archaeopress, 423-429.

Ciesielski, E., Duday, H., Girard, B., Roure, R., Martin, A., Agusti, B., 2011. La pratique des têtes coupées et les dépôts d'armes en Gaule méditerranéenne et dans le nord-est de la Péninsule Ibérique, in in Roure, R., Pernet L., (dir), *Des Rites et des Hommes. Les pratiques symboliques des Celtes, des Ibères et des Grecs en Provence, en Languedoc et en Catalogne*, Catalogue de l'exposition, Musée Archéologique Lattara, Lattes, Errance, (Archéologie de Montpellier Agglomération, 2), 113-145.

Ciesielski, E., Girard, B., Roure, R., 2014. L'exposition d'armes et de têtes coupées. Pratiques rituelles des Celtes du Midi au IIIe siècle avant notre ère, Archéopages, dossier « Le sort des vaincus », 6-15.

Collins, M.J., Nielsen-Marsh, C.M., Hiller, J., Smith, C.I., Roberts, J.P., Prigodich, R.V., Wess, T.J., Csapo, J., Millard, A.R., Turner-Walker, G., 2002. The survival of organic matter in bone: a review. Archaeometry 44, 383-394. https://doi.org/10.1111/1475-4754.t01-1-00071

Colonese, A.C., Farrell, T., Lucquin, A., Firth, D., Charlton, S., Robson, H.K., Alexander, M., Craig, O.E., 2015. Archaeological bone lipids as palaeodietary markers: Lipids as dietary markers. Rapid Commun. Mass Spectrom. 29, 611-618. https://doi.org/10.1002/rcm.7144

Evershed, R.P., Turner-Walker, G., Hedges, R.E., Tuross, N., Leyden, A., 1995. Preliminary results for the analysis of lipids in ancient bone. J. Archaeol. Sci. 22, 277-290.

Font, J., Salvadó, N., Butí, S., Enrich, J., 2007. Fourier transform infrared spectroscopy as a suitable technique in the study of the materials used in waterproofing of archaeological amphorae. Anal. Chim. Acta 598, 119-127. https://doi.org/10.1016/j.aca.2007.07.021

Langenheim, J.H., 2003. Plant resins: chemistry, evolution, ecology, and ethnobotany. Timber Press Portland, OR.

Lasserre, 1966. *Strabon, Géographie*, *Tome II : Livres III-IV*, texte établi et trad. par François Lasserre, Les Belles Lettres.

Łucejko, J.J., Lluveras-Tenorio, A., Modugno, F., Ribechini, E., Colombini, M.P., 2012. An analytical approach based on X-ray diffraction, Fourier transform infrared spectroscopy and gas chromatography/mass spectrometry to characterize Egyptian embalming materials. Microchem. J. 103, 110-118. https://doi.org/10.1016/j.microc.2012.01.014

Marchand-Geneste, N., Carpy, A., 2003. Theoretical study of the thermal degradation pathways of abietane skeleton diterpenoids: aromatization to retene. J. Mol. Struct. THEOCHEM 635, 55-82. https://doi.org/10.1016/S0166-1280(03)00401-9

Ménager, M., Azémard, C., Vieillescazes, C., 2014. Study of Egyptian mummification balms by FT-IR spectroscopy and GC–MS. Microchem. J. 114, 32–41. https://doi.org/10.1016/j.microc.2013.11.018

Mottram, H.., Dudd, S., Lawrence, G.., Stott, A.., Evershed, R.., 1999. New chromatographic, mass spectrometric and stable isotope approaches to the classification of degraded animal fats preserved in archaeological pottery. J. Chromatogr. A 833, 209-221. https://doi.org/10.1016/S0021-9673(98)01041-3

Nawar, W.W., 1969. Thermal degradation of lipids. J. Agric. Food Chem. 17, 18-21. https://doi.org/10.1021/jf60161a012

Nicholson, P.T., Shaw, I., 2000. Ancient Egyptian materials and technology. Cambridge University Press.

Parker Pearson, M., Chamberlain, A.T., Collins, M.J., Craig, O.E., Marshall, P., Mulville, J., Smith, H. , Chenery, C., Cook, G., Craig, J., Evans, J., Hiller, J., Montgomery, J., Schwenniger, J.-L., Taylor, G. and T. Wess. 2005. Evidence for mummification in Bronze Age Britain. Antiquity 79: 529-46.

Poirier, N., Sohi, S.P., Gaunt, J.L., Mahieu, N., Randall, E.W., Powlson, D.S., Evershed, R.P., 2005. The chemical composition of measurable soil organic matter pools. Org. Geochem. 36, 1174-1189. https://doi.org/10.1016/j.orggeochem.2005.03.005

Roure, R., Duday, H., Gardeisen, A., Girard, B., Lenorzer, S., Marchand, G., Piques, G., Schwaller, M., 2006. Armes et têtes coupées au Cailar (Gard) : premiers éléments de réflexion sur un dépôt rituel en Gaule méditerranéenne, dans L'âge du Fer dans l'arc jurassien et ses marges (est de la France, Suisse, sud de l'Allemagne). Dépôts, lieux sacrés et territorialité à l'âge du Fer. Actes du XXIX*e* colloque international de l'AFEAF, Bienne, 5-8 mai 2005, Besançon, Presses Universitaires de Franche-Comté, 653-658

Roure, R. Creuzieux, A., Girard, B., 2017. Fonder un lieu de culte en Gaule à l'âge du Fer : l'exemple du site du Cailar (Gard). *In : Quand naissent les dieux. Fondation des sanctuaires antiques : motivations, agents, lieux*, sous la direction de S. Agusta-Boularot, S. Huber et W. Van Andringa, Ecole Française de Rome, 277-298

Stott, A.W., Evershed, R.P., Tuross, N., 1997. Proceedings of the 17th International Meeting on Organic GeochemistryCompound-specific approach to the δ13C analysis of cholesterol in fossil bones. Org. Geochem. 26, 99-103. https://doi.org/10.1016/S0146-6380(96)00132-5