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Sulphur, fats and beeswax in the Iberian rites of the sanctuary of the *oppidum* of Puente Tablas (Jaén, Spain)

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ABSTRACT

This paper discusses the results of chemical analysis with regard to a major ceramic set retrieved from the sanctuary of Puerta del Sol. The site dates back to the 5th century BC and lies in the Iberian *oppidum* of Puente Tablas in Jaén, Spain. This is the first research paper on the Iberian culture to identify and describe accurately a sanctuary rite according to data based on chemical analysis. The chemical markers identified here confirm the occurrence of native sulphur, vegetable fat, ruminant animal fat and beeswax in the vessels under study. Methodologically, lipid analysis took place in two stages: GC–MS analysis of all the vessels, and HPLC–APCI-MS of the vessels with a lipidic profile capable of specifying the occurrence and the structure of triacylglycerols.

1. Introduction

The complexity of Iberian rites becomes apparent in cult spaces such as the sanctuary Puerta del Sol of the *oppidum* of Puente Tablas, in Jaén. Unlike the basic, standard pattern, it is an exceptional Iberian sanctuary that comprehends a large number of specific properties (spatial, symbolic, material, functional, etc.). These properties result in a sanctuary that follows a complex mythology of the female deity. The female deity supports a number of rites that define the aristocratic cult of an Iberian lineage in the late 5th century BC and in the first half of the 4th century BC (Ruiz et al., 2015).

As a complex set of rites and ceremonies, the Iberian cult may manifest as a wide range of expressions. In turn, these are related with a variety of properties and deities with specific variants (Rueda, 2011). The Iberian cult expresses a number of rules and norms on religious ceremonies (of fertility, healing, death, heroification, marriage, youth, etc.), and these can be observed in a range of spatial and chronological contexts (Rueda, 2013). In this general frame, it is important to remark that ritual behaviour's manifestations and materializations can be delimited (Bell, 1992), because cultural practice reflects specific actions and, naturally, sets in a specific liturgical structure (Insoll, 2011).

Rites materialize as 'artefacts' that act as active instruments for the achievement of the rite, and as images that narrate the moment chosen for a wider recognition. From a methodological point of view, this approach to the study of rites requires the analysis of behaviour models that find a reflection in the archaeological record. These models allow approaching symbolic patterns as a conditioned expression that is connected with the liturgical structure. The latter often uses a sanctioned language that is also socially understandable. In the case of the Iberians, these processes can be tracked down via a range of materials and which demand an articulated, interdisciplinary research method (Rueda, 2013). Food offerings and/or sanctuary commensality are a clear example: they can be fully understood only by use of articulated, complementary diagnostic techniques such as chemical analysis, archaeozoological and carpological analysis, ceramic typology, iconographic studies and studies of written sources.

This paper deals with the chemical analysis of contents in a large ceramic set of the ritual area of the sanctuary Puerta del Sol. The aim is to gain a deeper knowledge of the rites performed in the sanctuary. The chemical analysis is complemented and fed back by the information provided by archaeological analysis, by the study of written sources and by the analysis of the wildlife and seeds (Montes, 2015; Rodríguez and Pradas, 2015; Ruiz et al., 2015). The method used for the chemical analysis is based on the joint use of gas chromatography–mass spectrometry (GC–MS) and high-performance liquid chromatography–atmospheric

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pressure chemical ionization-mass spectrometry (HPLC–APCI-MS) (Craig et al., 2009).

Chemical analysis of contents in ceramic vessels, specifically of lipids, has made substantial progress in the past 30 years. Use of GC–MS for research on trimethylsilylderivates (TMS) in the 90 s allowed identifying particularly relevant markers in archaeology, such as mono-, di-, triacylglycerols (TAGs) and wax esters (Evershed et al., 1990, 1992). The method prevailing until then relied on the formation of fatty acid methyl esters (FAMEs) for subsequent GC–MS analysis. Use of the latter method meant the loss of the information contained in acylglycerols and wax esters. The improvement in the methods available has thus resulted in more accurate identification of highly relevant archaeological contents such as animal and vegetable fats, beeswax and vegetal wax, drinks and resins (Colombini et al., 2005; Evershed et al., 1991, 2008; Garnier et al., 2003; Parras et al., 2011; Regert et al., 2001, 2003; Sánchez et al., 1998).

A range of variants have followed the development of the above method over the past few years. In some specific cases of analysis of ceramic materials, pyrolysis has been added as the extraction system for GC–MS (Py-GC–MS) (Garnier et al., 2003; Legnaioli et al., 2013). Still, Py-GC–MS has been used more often for other materials. Recent progress in MS and hyphenated techniques allows the identification of carbon stable isotope compositions. Using a highly sensitive device such as a gas chromatograph–combustion–isotope ratio mass spectrometer (GC–C–IRMS) and plotting the δ^{13}C values of methyl palmitate against those of methyl stearate, several animal and marine fats can be identified, and the occurrence of milk can be attested (Craig et al., 2011; Cramp et al., 2014; Evershed et al., 2008; Heron et al., 2013; Regert, 2011). The third variant, used for this paper along with GC–MS, is HPLC–APCI-MS.

Unlike former chromatographic techniques, HPLC–APCI-MS has been used recently for the analysis of lipidic contents in Archaeometry. In fact, this technique can isolate and identify intact TAGs more accurately than other techniques. Thus, it is possible to associate TAG profiles with a range of fat types by HPLC–APCI-MS (Saliu et al., 2011). First, TAGs with a high content of unsaturated fatty acids (oleic, linoleic, linolenic) are associated with vegetal fats (Fasciotti and Pereira, 2010; Kimpe et al., 2001). Second, TAGs of saturated fatty acids are associated with animals; some of them are so specific that they can be considered biomarkers of fat of specific animal species (Kimpe et al., 2002; Romanus et al., 2007; Saliu et al., 2011). Finally, the difference between ruminant animal fat and non-ruminant animal fat is based on the proportion of palmitic and stearic acids in the *sn*-2 position of TAG (P:S ratio) (Evershed et al., 2002; Regert, 2011; Saliu et al., 2011).

HPLC has a lower potential in archaeological research, even if it has led to identification of a range of contents in ceramic vessels. Thus, a substance that could be identified as olive oil was identified in lamps (Kimpe et al., 2001), and ruminant fat was identified in cooking vessels too, both in the late Roman materials of the site of Sagalassos (Turkey) (Kimpe et al., 2002). In the latter case, the TAG profiles found in the residue were compared with the TAG profiles of present-day animal fat samples used as a reference. Using the same basis of comparison, Craig et al. (2009) presented a dataset obtained from research on food residue absorbed in Early Iron Age ceramics from the site of Gordion (Turkey). HPLC–APCI-MS has also proved efficient for the identification of ruminant fat, as attested by the comparison with GC–MS and GC–C–IRMS analysis (Romanus et al., 2007). HPLC–APCI-MS analysis has also been recently considered useful for TAGs analysis in a piece of research on 18th century majolica ceramic apothecary jars and conserved in the ancient Apothecary of the Aboca Museum (Sansepolcro, Arezzo, Italy) (Saliu et al., 2011).

2. The archaeological context

The ceramic material analysed here comes from the sanctuary Puerta del Sol, in the Iberian *oppidum* of Puente Tablas (Jaén, Spain). The Iberians are known to have been the most advanced culture in the Iberian Peninsula during the Iron Age. The Iberian world developed from the 6th century BC and reached its zenith during the 5th to the 3rd century BC. Geographically, the Iberian culture spread over the south, east and north of the Iberian Peninsula, as well as over the southeast of present-day France, and represented a clearly Mediterranean culture (Ruiz and Molinos, 1998).

The *oppidum* of Puente Tablas lies 4 km north of Jaén (Andalusia, Spain) (Fig. 1). It is a triangular plateau of approximately 6 ha of surface with a pointed shape towards the northwest. It is heavily fortified with walls and towers except at the base of the triangle, which looks on River Guadalbullón and, therefore, forms a natural defence (Fig. 2). Chronologically, the settlement was occupied by the Iberians from the late 8th century BC to the late 3rd century BC (Ruiz and Molinos, 2008, 2015).

The sanctuary Puerta del Sol is next to the east gate of the *oppidum*. It is the main entrance, at the end of a 14 m corridor, and it is also where an anthropoid stone stele stands that represents the female deity (Ruiz et al., 2015). The construction has a surface of 300 m^2 and is arranged as three terraces and several rooms with a stone board and mud walls with a covering. In the more important rooms, the covering is gypsum (Figs. 3 and 4) (Parras et al., 2015; Sánchez et al., 2014).

Access is through the first terrace across a yard (Room R). This room also contains a bullskin-shaped stone altar. This is the landmark for access to the *antecella* (Room Q) and to the area dedicated to the divinity, *cella* (Room F). The latter houses the small chapel-like room (Room M) that would contain the stele for a part of the year.

The side of the yard leads to the second terrace, which is divided into several functional spaces. A quadrangular-shaped, two-storey tower lies to the east. The inside has a gypsum covering. Two *bothroi* made with remains of lamb sacrifices were at the entrance (Room J). A yard with two paved levels lies opposite (Rooms U and Z). This space has been interpreted as used for rites. Four small caves and three small altars are on the west corner. The altars are of various shapes: quadrangular, eye-shaped and ear-shaped. The caves, insofar as liminal spaces in the symbolic sense, are physically marked off from the construction space by a small stone wall. This is a *temenos* whose threshold was not allowed to be trespassed. Actually, no offerings have been recorded inside the caves. This is in sharp contrast with the record of votive offerings in Rooms U and Z, where storage vessels, small vases and a loom weight stand out. The loom weight combines a bullskin and a lotus flower in a small stamp. Attic grave offerings, namely two cups and a red-figure krater are especially remarkable. The krater depicts an initiation before the statue of a female deity, probably Aphrodite or Artemis. The fourth wall is separated by a wall. The entrance is through a trapeze-shaped room (Room V). This is a room of limited access, because this is the largest and deepest cave, and the one where the myth of the deity is born.

The third terrace is the highest one, and it is used for service. Specifically, a cistern for water supply through a channel that runs across the sanctuary has been attested here. Water is a highly symbolic element and a key functional element in this cult space.

The female deity defines the cult of this sanctuary and sanctions aristocratic practice. The stone stele depicts a lady wearing a mitre and with her arms folded on her belly and her hands holding a small circular element, perhaps intended to represent the sun. This deity is associated with equinoxes as vital landmarks of the ritual calendar. These landmarks signal their location opposite the entrance corridor and would receive the first sunbeams at dawn. Only some minutes later, the second stage of the mythical narrative of the deity takes place. The sunbeams enter the fourth cave and create a scenographic effect of a visual representation of the sacred. This religious event strengthens collective identity by way of the construction of their mythology (Hamilakis, 2011). These two stages make up the sequence of a myth based on the association between the sun and the deity. This myth describes the deity's return to her dwelling, the cave. Alternatively, it can be interpreted as her